CHARACTERIZATION OF CORN/SUGAR PALM FIBER-REINFORCED CORN STARCH BIOPOLYMER HYBRID COMPOSITES

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By

MOHAMED IBRAHIM J IBRAHIM

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

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Chairman : Professor Ir. Mohd Sapuan bin Salit, PhD, PEng
Faculty : Engineering

Contemporary environmental concerns, such as non-biodegradable disposal materials and the growing mountain of garbage as well as the plant waste accumulation, are increasingly recognized as ecological threats. Space for landfills is limited, and additional incineration capacities require high capital investment and cause further environmental problems. All these issues forced the researchers and scientists to move toward manufacturing and developing eco-friendly engineering materials from renewable sources to replace conventional non-biodegradable materials in several applications that could preserve the green environment. Amongst these sources are corn plant and sugar palm tree, which are a vital source for many biomasses. Therefore, a series of lab experiments through a solution casting technique was carried out to prepare and characterize starch, fibers, polymers, composites, and hybrid composites in four correlated stages to achieve a hybrid composite from corn/sugar palm fiber reinforced cornstarch.

The first stage was designed to study the chemical composition, physical properties, thermal stability, and surface morphology of thermoplastic corn starch and corn hull, husk, and stalk fibers, which were extracted from different corn plant parts. The obtained samples were characterized on a powder basis. The corn husk and corn starch revealed an excellent combination of properties. Cornhusk provided the highest cellulose content (45.7%) as well as the most favorable surface morphology. Corn starch revealed acceptable amylose content (24.6 g/100g) and tolerable thermal stability with an onset melting point of 161.2 ºC. Since the cellulose and starch demonstrated an excellent correlation between the function and structure of biomolecules. Hence, both corn starch and husk have the potential for use in many applications of the biomaterial.

The second stage was accomplished to determine the effect of various concentrations of selected plasticizers in cornstarch-based films, to prepare a new biopolymer. The physical, morphological, thermal, and tensile properties of produced films were evaluated. The results showed that the thickness, moisture content, and water solubility increased with the addition of plasticizer concentration. Regardless of plasticizer sort, the tensile stress and modulus of plasticized films decreased as the plasticizer concentrations were raised beyond 25%. Likewise, the relative crystallinity decreased by increasing the plasticizer content from 0% to 25%, but it began to grow once the concentration increased above 25%. The fructose-plasticized films presented consistent and more coherent surfaces compared to sorbitol and urea counterparts. In summary, the plasticizer types and concentrations are significantly affected on the performance of the cornstarch-based film, especially for 25% fructose addition.

In the third stage, biodegradable composite films were prepared by using different concentrations of husk fiber as a reinforcing filler to the optimum biopolymer produced from the previous stage. The findings indicated that the incorporation of husk fiber, in general, enhanced the performance of the composite films. There was a noticeable reduction in the density, moisture content of the films, and soil burial assessment showed less resistance to biodegradation. The morphological images presented a consistent structure and excellent compatibility between matrix and reinforcement, which reflected on the improved tensile strength and modulus as well as the crystallinity index.

In the last stage, hybrid composites were successfully prepared by loading different concentrations of sugar palm fiber to the best composite from the previous stage. The incorporation of sugar palm fiber increased the thickness and the crystallinity index while reducing the density, moisture content, water solubility, water absorption, and water vapor permeability of the films. The tensile strength and modulus of the films were increased from 6.8 MPa to 19.05 MPa and from 61.15 MPa to 1133.47 MPa respectively for the film contains 6% sugar palm fiber, making it the most efficient reinforcing.

To sum up, corn husk/sugar palm fiber reinforced cornstarch hybrid composite films as anticipated, improved the mechanical properties, and the water barrier characteristics. Thus, they are suitable for replacing conventional non-biodegradable materials in many applications.

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

PENCIRIAN SERAT JAGUNG/ENAU DIPERKUKUHKAN BERSAMA KANJI JAGUNG BIOPOLYMER HIBRID KOMPOSIT

Oleh

MOHAMED IBRAHIM J IBRAHIM

Disember 2019

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Kebimbangan terhadap alam sekitar, seperti bahan pelupusan tidak biodegredasi dan pembuangan sampah sarap yang semakin meningkat telah memberi ancaman kepada ekologi. Ruang pelupusan sampah adalah terhad dan kapasiti kawasan pembakaran tambahan memerlukan pelaburan modal yang tinggi, telah menyebabkan masalah persekitaran yang berleluasa. Semua isu ini telah memaksa para penyelidik dan saintis untuk bergerak ke arah pembangunan bahan-bahan mesra alam daripada sumber yang boleh diperbaharui untuk menggantikan bahan konvensional yang tidak boleh dibiodegredasi. Diantara sumber-sumber ini adalah tumbuhan jagung dan pokok enau yang merupakan sumber yang penting bagi kebanyakkan biomas. Oleh itu, satu siri eksperimen makmal melalui teknik pengacuan cairan telah dijalankan untuk menyediakan sampel. Seterusnya pencirian kanji, serat, polimer, komposit dan komposit hibrid dalam empat peringkat turut dijalankan untuk mencapai komposit hibrid daripada serat jagung/enau diperkukuhkan kanji jagung.

Peringkat pertama direka untuk mengkaji komposisi kimia, sifat fizikal, kestabilan termal, dan morfologi permukaan termoplastik kanji jagung, sekam jagung, dan tangkai jagung, yang diekstrak daripada pelbagai bahagian pokok jagung. Sampel yang diperoleh telah diasingkan dalm bentuk serbuk. Sekam jagung dan kanji jagung menunjukkan kombinasi ciri-ciri yang terbaik. Sekam jagung mengandungi kandungan selulosa yang tertinggi (45.7%) serta permukaan morfologi yang memuaskan. Kanji jagung menunjukkan kandungan amilosa yang baik (24.6g/100g) dan kestabilan terma yang mampu boleh diterima dengan titik lebur permulaan sebanyak 161.2 ºC. Fungsi dan struktur biomolekul diantara selulosa dan kanji menunjukkan hubungan yang sangat baik. Oleh itu, kedua-dua kanji dan sekam jagung mempunyai potensi untuk digunakan dalam banyak aplikasi biomaterial.

Tahap kedua telah dicapai bagi menyelidik kesan pelbagai ukuran kepekatan plasticizer terpilih dalam filem berasaskan kanji jagung dan juga untuk menyediakan biopolimer baru. Ciri-ciri fizikal, morfologi, haba, dan ketegangan filem yang dihasilkan telah dinilai. Keputusan menunjukkan bahawa ketebalan, kandungan kelembapan, dan kelarutan air meningkat dengan penambahan kuantiti plasticizer. Berbeza dengan jenis plasticizer, tekanan tegangan dan modulus filem plastik semakin berkurang apabila kandungan plasticizer meningkat melebihi 25%. Begitu juga, kekristalan relatif mulai menurun dengan meningkatkan kandungan plasticizer dari 0% hingga 25%, namun ianya mula meningkat apabila kandungan plasticizer meningkat melebihi dari 25%. Filem fructose-plasticized menemukan permukaan yang konsisten dan lebih koheren berbanding dengan tindak balas sorbitol dan bahagian urea. Akhirnya, jenis plasticizer dan kandungan kepekatan akan terjejas dengan ketara terhadap prestasi filem berasaskan cornstarch, terutamanya untuk penambahan fruktosa 25%.

Pada peringkat ketiga, filem komposit biodegredasi disediakan dengan menggunakan kandungan kepekatan serat sekam yang berbeza untuk pengukuhan biopolimer secara optimum yang dihasilkan dari peringkat yang sebelumnya. Penemuan menunjukkan bahawa penggabungan gentian sekam, secara amnya, meningkatkan prestasi filem komposit. Terdapat pengurangan yang ketara dalam ketumpatan, kandungan lembapan filem, dan penilaian pengebumian tanah menunjukkan rintangan yang berkurangan terhadap biodegradasi. Imej morfologi membentangkan struktur yang konsisten dan keserasian yang sangat baik antara matriks dan penguat, yang mencerminkan peningkatan kekuatan tegangan dan modulus serta indeks kritalisasi yang lebih baik.

Pada peringkat yang terakhir, komposit hibrid telah berjaya disediakan dengan memuatkan kandungan serat enau yang berbeza ke komposit yang terbaik berbanding eksperimen terdahulu. Penggabungan serat enau meningkatkan ketebalan dan indeks kristaliniti. Disamping itu, ia juga sambil mengurangkan kepadatan, kandungan kelembapan, kelarutan air, penyerapan air dan kebolehterapan wap air dari filem. Kekuatan tegangan dan modulus filem meningkat dari 6.8 MPa ke 19.05 MPa dan dari 61.15 MPa menjadi 1133.47 MPa bagi filem ini mengandungi 6% serat enau, menjadikannya cara pengukuhan yang paling berkesan.

Kesimpulannya, serat enau/jagung diperkukuhkan bersama kanji jagung hibrid komposit film meningkatkan sifat mekanikal dan sifat menghalang air. Oleh itu, bahan ini sesuai untuk menggantikan bahan konvensional tanpa biodegredasi dalam pelbagai jenis aplikasi.

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

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LIST OF ABBREVIATIONS AND SYMBOLS

A_a	Amorphous area
A_c	Crystallinity area
CHF	Cornhusk fiber
C_i	Crystallinity index
CS	Corn starch
CSF	Cornstalk fiber
DTG	Derivative thermogravimetric
Dn	Number mean diameter
Dv	Number mean volume
FTIR	Fourier transform infrared
EB	Elongation at break
$M_{initial}$	Initial mass
M_{final}	Final mass
KBr	Potassium Bromide
MC	Moisture content
PLA	Polylactic acid
SEM	Scanning electron microscope
SPF	Sugar palm fiber
T_c	Conclusion temperature
T_o	Onset Temperature
T_p	Peak gelatinization temperature
TGA	Thermal-gravimetric analysis
TPS	Thermoplastic starch
TS	Tensile strength

WA	Water absorption
WC	Water content
WHC	Water holding capacity
$w_{initial}$	Initial weight
w_{final}	Final weight
WS	Water solubility
w/w	Weight to weight
WVP	Water vapor permeability
XRD	X-ray diffraction
d_i	Particle (i) diameter
G''	Loss modulus
G'	Storage modulus
TG'	Storage modulus temperature
tan ϱ	Loss factor
ΔH	Enthalpy of gelatinization
ρ	Density
θ	Diffraction angle

CHAPTER 1

INTRODUCTION

1.1 Background

The accumulation of agricultural residues, together with petroleum-based plastic wastes, have contributed dramatically to increase environmental pollution to the point where it has caused problems for natural life and human health as well. The invention of plastics brought about a revolution in materials production in various sectors such as the medical, automotive, electronics, packaging, among others (Sharuddin, Abnisa, Daud, & Arou, 2016). It is characterized by durability, heat resistance, and suitability for mass production. The global production of synthetic plastics reached 140 million tons annually, an increase of 2% per year (Shimao, 2001); This indicates that the percentage of plastic trash that ended up in the landfill is very high and occupies a large area. Since plastics are clearly valuable and necessary in our daily lives, some material engineers are trying to develop safer and more environmentally friendly plastics. Some innovators are developing bioplastics, made from plant crops rather than fossil fuels, to create more environmentally friendly materials than conventional synthetic plastics. Others are attempting to fabricate truly biodegradable plastics. Some researchers are looking for ways to make recycling more effective and hope to master the process of converting plastics back into fossil fuels from which they are originated. All these scientists realize that plastics are not perfect but a necessary and crucial part of our present and future (Pfaendner, 2006). In order to mitigate the issue of non-biodegradable plastics and biomass waste disposal, the production of environmentally friendly materials to compensate for long-lasting plastics is inevitable (Edhirej, Sapuan, Jawaid, & Zahari, 2017d). The development of eco-friendly materials from natural renewable sources has reduced dependence on conventional plastics, which in turn contributes to solving the complications of environmental pollution. In recent times, there has been growing interest in using raw materials and agricultural by-products in achieving biodegradable plastics, such as from cassava, potato tubers, sugar palm, and corn. Despite their multi characteristics such as availability, biodegradation, affordability, and recycling, it is known that bioplastics developed from natural sources have certain disadvantages, especially in terms of mechanical performance and water sensitivity compared to fossil sources plastics (Averous & Boquillon, 2004). Therefore, it is necessary to maintain and increase research efforts in this area, taking into account the use of local raw materials obtained from the region such as corn plant, which are studied in research projects, through which the methodology aims to produce biodegradable plastics can be reproduced on an industrial scale, taking into account the specific functional requirements of various applications.

Corn (maize) is a cereal plant belonging to the grass family and is extensively used as human food, livestock feed, a source of biofuels as well as a raw material in manufacturing sectors. It was first cultivated in Mexico by local peoples about 10,000 years ago, currently, it is widely cultivated in Latin America, Asia, tropical Africa, and North America and it is the most important crop in developing countries, with an approximate production of 1.4 billion tons in 2014, equivalent to 30% of world's grains production (R. Singh, Ram, & Srivastava, 2016). Furthermore, corn is the main source of commercial starch available, each corn granule (kernel) consists of more than 70% starch type alpha-linked glucose, and the rest is minor ingredients such as crude fats, crude proteins, ash, and minerals (McAloon, Taylor, Yee, Ibsen, & Wooley, 2000a). Genetically modified corn starch is widely used as an enhanced matrix for composites, due to its attractive characteristics such as natural availability, biodegradability, and affordability. Corn starch applications extend to health check instruments, electrical appliances, packaging, furniture, and alternative to plastic parts of automobiles (Guimarães, Wypych, Saul, Ramos, & Satyanarayana, 2010). The value of harvested corn plant could be improved by obtaining lignocellulosic fibers from the corn stover (leaves, stems, hulls, and cop). Corn stover typically contains 15% husk, 35% cobs, and 50% stalk; the majority of the stover is discarded as residues despite their high potential for use as biomass (Sokhansanj, Turhollow, Cushman, & Cundiff, 2002).

In modern biomaterials science, natural lignocellulosic fibers (NLF) are known to be unique reinforcing fillers for polymers and composites. Compared to synthetic fillers, the NLF characterized by many advantages such wide variety and availability, renewability nature, low density, biodegradability, cost-effective, lower energy consumption as well as high specific strength and recyclability (Bodirlau, Teaca, & Spiridon, 2013; Gilfillan, Nguyen, Sopade, & Doherty, 2012). Moreover, it provides high sound attenuation and relatively easy processing and handling due to its good flexibility and anti-rust nature that allows high reinforcing quantities (Al-Oqla & Sapuan, 2014). Natural plant fibers can be obtained from the processing of agricultural residues. These residues include process residues and field residues; Process residues are obtained following the crop being processed into valuable resources, includes seeds, husks, bagasse, molasses, and roots. While field residues indicated to the wastes left in the cultivation field after harvesting, this type includes leaves, stalks, seeds, stems, and pods. Both types provide additional value for natural materials (Richards, Wafter, & Muck, 1984).

Thermoplastic starches (TPS) are natural polymers recognized as one of the promising biomaterials in the field of biomass production due to their attractive properties that are combining the affordability, availability, and performance (Abdillahi, Chabrat, Rouilly, & Rigal, 2013). Therefore, they have been used extensively as a supporting matrix for the production of bioplastics. However, TPS-based materials revealed certain drawbacks due to its high hydrophilic characters such as brittleness, water propensity, and inadequate strength (Averous & Boquillon, 2004). Thus, the incorporation of enhancing substances like plasticizer is required to alleviate such drawbacks. The primary function of the plasticizers is reducing the strong attraction of hydrogen bonds within the starch network and facilitate the mobility of the polymer particles; this, in turn, improves the flexibility and stiffness of starch-based plasticized materials (Sanyang, Sapuan, Jawaid, Ishak, & Sahari, 2015). Examples of using plasticizers as enhancing agents within the starch-based biopolymers have been stated elsewhere in this thesis. The results indicated that the achieved TPS plastic polymers still had insufficient dimensional stability and proved more brittleness as they lost water or were exposed to high humidity. These shortcomings are severely restricted to their wide application.

Due to the high correlation between the starch polymer and cellulose fiber, many material researchers have moved towards enhancing the performance of TPS-based materials by incorporating natural cellulosic fiber as reinforcing fillers to form a biocompatible composite. Significant improvement was observed in the final product, particularly in terms of mechanical characteristics and water barrier properties. For instance, Edhirej et al., (2017b) produced biocomposites films by filling the cassava TPS matrix with cassava bagasse. Rabe et al., (2019) investigated the influence of coconut fiber on corn TPS biocomposites. Hassan et al., (2019) reinforced potato TPS by PLA. Gazonato et al., (2019) studied the thermomechanical properties of the cornstarch-based film filled with coffee ground waste. Although acceptable properties have been achieved, the results suggest upgrading the tensile properties and water barrier characteristics in order to further improve in the performance and extend the usability.

In an attempt to settle such deficiencies, incorporating two or more different fibers into a single matrix might be led to the development of hybrid biocomposites with better characteristics. The behavior of the hybrid material is a weighted sum of an individual constituent in which there is a more appropriate balance between inherent advantages and disadvantages, also, using more than a single fiber type, the advantages of one type of fiber can substitute what the other lacks (Edhirej et al., 2017a). In general, the characteristics of the hybrid composite depend mainly on the fiber content, the particle size distribution of the individual fiber, the bonding of the fibers to the matrix, the arrangement of both fibers as well as the compatibility of the failure strain of the fibers used (Sreekala, George, Kumaran, & Thomas, 2002). As a consequence, hybridization with a fiber characterized by high-water resistance such as sugar palm fiber is expected to provide better results. The sugar palm tree is a member of the Palmy family (Siregar, 2005). It mostly planted in tropical regions that cover southeast Asia and north Australia. Also, it is a multi-purpose tree besides being a potential source of starch and natural fiber (Ishak et al., 2013).

Sugar palm fiber (SPF) is a natural lignocellulosic fiber characterized by high resistance to seawater, high tensile strength, low degradation rate, and durability (Ilyas, Sapuan, Ishak, & Zainudin, 2017). The preparation of SPF requires no effort, as it does not involve any secondary processing or treatment such as mechanical decorticating or water ratting (Edhirej, Sapuan, Jawaid, & Zahari, 2017a). In the field of composite materials, many studies have been published about the utilization of sugar palm fiber as a reinforcing agent with the polymer matrix. The results indicated that sugar palm fibers have the potential to be used in many applications of composite materials, especially those requiring high water resistance.

This study will focus on the manufacturing of a biohybrid composite material by using agricultural residues (biomass) of corn and sugar palm trees; these hybrid biocomposites will be used as an alternative to synthetic plastic composites. Therefore, the objectives of this research are to extract starch and potential fibers from corn plant parts such as stalk and husk and then develop a biopolymer by adding different plasticizers then combining corn starch and corn fiber to produce biocomposites. Finally, eco-friendly and degradable hybrid materials will be produced using corn starch as matrix and corn/sugar palm fiber as reinforcement. Characterization processes will be accompanied by preparation procedures in terms of mechanical performance, thermostability, physical, and morphological properties.

1.2 Problem statements

Annually, millions of tons of residues remain as a by-product of agriculture crops such as corn, wheat, barley, rice, cassava, sugarcane, etc. Agricultural residues are materials left on cultivated land after the crop has been harvested, varying greatly in properties and decomposition rates (Lal, 2005). Globally, it is estimated that between 2003 and 2013, the production of agricultural residues increased by 33 %, reaching 5 billion tons in 2013. The Asian continent is the largest producer of crop residues, 47 % of the total, followed by America (29 %) Europe (16 %), Africa (6 %), and Oceania (2 %) (Cherubin et al., 2018). These residues caused substantial environmental issues, such as increased CO2 and other greenhouse gas emissions, soil degradation, biodiversity loss, and water degradation due to excessive nutrient leaching (Foley et al., 2011). Corn plant is one of the major sources of agriculture residues in the form of stover; the global production of corn residues reached 1016.7 million tons in 2013. The production of corn residues in Asia reaching 304.3 million tons, this accounts for approximately 30 % of the total global production (Cherubin et al., 2018). Corn stover typically consists of 50% stalk (stem), 35% leaves and cobs, and 15% husk. Most of the stover is disposed of as waste, while it is likely to be detected as natural fiber (Sokhansanj et al., 2002). These residues are generally left to compost in the fields or are incinerated. The incineration of agricultural wastes continually generates a large amount of greenhouse gases like carbon dioxide, methane, nitrous oxide, and ozone. Such gasses have a negative impact on health and contribute to global warming and global pollution as well.

In response to community demand to dispose of agricultural and polymeric wastes that have environmental problems, finding value-added uses of these undervalued field crop residues together with bio-based polymers may help maintain a carbon dioxide balance and has the potential of reducing problems associated with emissions produced during the manufacturing of petroleum-based composites and from field waste incineration. Therefore, the preparation and development of environmental materials derived from sustainable resources are considered an appropriate solution for waste management and alternative to petroleum-based materials. Such bio-resources are having a positive impact on air, land, water, and characterized by renewability, availability, biodegradability, and cost-effective. Natural cellulose fibers and polymers typically extracted from plant residues have great potential to meet the requirements of environmental complications. A systematic approach on how to select the best biopolymer and natural fiber, along with the best conceptual design, that helps to reduce the environmental impact of the entire product life cycle. Hence, it is necessary to maintain and increase the research efforts in the field of composite biomaterials, taking into account the use of local raw materials sourced in the region such as corn plant and sugar palm tree, which are being studied in research projects, through which it is intended that the methodology production of biodegradable plastics is reproducible on an industrial scale, considering the specific functional requirements for various applications.

1.3 Research objectives

The research objectives of this study are: -

1. To characterize starch and potential fibers from corn plant parts (hull, husk, and stalk) in order to prepare and develop a new biodegradable and environmentally friendly composite.
2. To investigate the physical, mechanical, thermal, and morphological properties of the cornstarch-base films as affected by different plasticizers.
3. To investigate and characterize the potential of using multiscale corn husk fiber as reinforcing filler in cornstarch-based biocomposites.
4. To determine and characterize the effect of sugar palm fiber loading on corn/sugar palm fiber reinforced cornstarch hybrid composites.

1.4 Significance of study

1) Development of a new biodegradable hybrid composite characterized by low in cost production, renewable, biodegradable, and environmentally friendly for use in a variety of industrial applications as an alternative to non-biodegradable petroleum plastics.
2) Substituting petroleum-based polymers with TPS-based polymers may reduce the growth rate of petroleum polymers, thereby reducing the health effects resulted from it and reducing dependence on petroleum.
3) The utilization of corn plant and sugar palm residues, which are highly abundant and inexpensive that may contribute to mitigate the problem of wastes and responds to the community's demand for agricultural and polymeric waste disposal, which also improves the economic growth through the transfer from waste to wealth.
4) Besides, this research provides a cognitive contribution to product design specifications, material selection analysis, conceptual design development, and conceptual design selection.

1.5 Scope and limitation of study

In the current research, cornstarch (CS) and corn hull fiber were derived from fresh corn ear, while corn husk and stalk fibers were extracted from the leaves and stems of corn plants, respectively. The obtained biomasses in powder form were characterized in terms of physical, thermal, structural, morphological properties, and chemical composition as well. The influence of various plasticizers kind and concentration on physical, thermal, tensile, and structural properties of cornstarch based-film were evaluated. The criteria for selection were mainly based on the best physical and tensile properties. Hence, the CS-plasticized film with optimum characteristics was reinforced with corn husk fiber CHF (the best fiber) at different loadings (0, 2, 4, 6, and 8%). The optimal fibrous loading ratio of the obtained composite films was then selected based on the physical and tensile properties supported by morphological, structural, and thermal properties. After that, the selected composite film was hybridized by sugar palm fiber (SPF) at different concentrations (2, 4, 6, and 8% w/w dry starch). The specimens achieved were tested for their tensile and thermostability properties along with other analyses such as XRD, FTIR, and SEM. Finally, the CS-CH/SPF hybrid composites were submitted to the water barrier assay and biodegradation test to investigate its environmental impact.

It should be noted that all film samples were prepared using the solution casting technique into an aqueous medium containing distilled water, and all concentrations of the used substances were calculated based on the weight of corn starch (5g). Furthermore, there was not any treatment or modifying the chemical structure of the materials used in the current work.

1.6 Structure of thesis

The outline of the thesis is following the alternative thesis format of Universiti Putra Malaysia based on publications, in which each research chapter (4-7) represents a separate study it is own included: 'Introduction,' 'Materials and methods,' 'Results and discussion,' and 'Conclusion.' The details of the thesis structure are presented below.

Chapter 1

The problem statement and research objectives are clearly described in this chapter. Furthermore, the importance and contribution of the research, as well as the scope and limitation of the study, were also illustrated in this chapter.

Chapter 2

A comprehensive review of the literature on the critical areas related to the subject of this thesis is presented in this chapter.

Chapter 3

This chapter of the methodology includes every activity related to this research, from the beginning of material preparation to material processing, testing procedures, and data collection and analysis.

Chapter 4

This chapter presents the first article entitled "Extraction, Chemical Composition, and Characterization of Potential Lignocellulosic Biomasses and Polymers from Corn Plant Parts." In this article, the physical, morphological, structural, and thermal properties of a cornstarch and corn hull, husk, and stalk fibers were evaluated.

Chapter 5

This chapter presents the second article entitled "Physical, Thermal, Morphological, and Tensile Properties of Cornstarch-Based Films as Affected by Different Plasticizers." In this article, the influence of different plasticizer types (fructose, sorbitol, and urea) at concentrations (25, 40, and 55%) on the properties of the corn-based film was investigated.

Chapter 6

This chapter presents the third article entitled "Potential of Using Multiscale Corn Husk Fiber as Reinforcing Filler in Cornstarch-Based Biocomposites" This article focused on producing and characterizing of biocomposite films based on cornstarch matrix and cornhusk fiber as reinforcement filler at different loadings.

Chapter 7

This chapter presents the fourth article entitled "Processing and Characterization of Corn/Sugar Palm Fiber Reinforced Corn Starch Biopolymer Hybrid Composites." This article studied the effect of various loading of sugar palm fiber (2%, 4%, 6%, and 8%) on the physical, thermal, structural, tensile, and water barrier properties of thermoplastic cornstarch-based hybrid composite contains 25% of fructose plasticizer and 8% of cornhusk fiber.

Chapter 8

This chapter provides general conclusions from various research articles, as well as relevant suggestions and recommendations for future research.

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