CHARACTERIZATION AND PROPERTIES OF MISWAK (*Salvadora persica* L.) FIBER-REINFORCED POLYLACTIC ACID BIOCOMPOSITES

By

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

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

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Chair : Professor Ts. Khalina Abdan, PhD
Institute : Tropical Forestry and Forest Products

The characterization and properties prepared from miswak fiber (MF)/polylactic acid (PLA) using internal mixer and compression molding was investigated. The chemical, physical, and thermal properties of untreated and alkali-treated fiber biocomposite were all examined. The aspect ratio and fiber density were found to be exceptionally low. While alkaline treatment exposes a clean and clear fiber surface, untreated fiber's shape indicates the existence of residual material. The TGA statistics demonstrate the thermal resilience of fiber because its quality only starts to decline at 88°C. Contrarily, alkaline treatment raises fiber thermal stability to 96°C. Miswak fiber content significantly affected the mechanical and physical properties of MF/PLA composites as higher loading increased the density, water absorption and thickness swelling. The 10wt% fiber loading (M1) in PLA showed highest tensile strength (33.7 MPa) and highest modulus (2.9 GPa), respectively. In all composites, M1 composite have better crystallization and melting temperatures at 95°C and 139°C as well as better thermal stability when the maximum degradation occurs at 341°C. DMA noticed that the addition of miswak fiber increased E' curve and slightly shifted Tan δ to a lower temperature. As compared to PLA matrix, the biodegradability of the composites was superior as deterioration was observed after 100 days in soil burial test. As for alkali treated composites properties, the FTIR shows constant shift as the alkali concentration increases up to 3%. Tensile modulus results show mechanical performance increase from 1% to 3% with 30 wt.% fiber loading and SEM micrograph supported the tensile results. The alkali treated fiber composites exhibit better thermal stability as TGA curve showed slightly to high temperature as compared to untreated fiber composites and DSC curve showed improved in crystallization temperature for alkali treated fiber composites recorded at highest 116°C for 1% alkali concentration. The DMA found that adding alkali treated fiber in PLA affected the level of storage modulus curve and Tan δ peak magnitude analysis results also showed the 1% alkali-treated fiber had better adherence to the MF/PLA matrix. In conclusion, composites with 10wt% of miswak fibre loading in PLA showed better in overall properties.

Moreover, 1% alkali treated fiber/PLA composites showed increase thermal stability among the other alkali concentration with 10wt.% fiber loading. Hence the M1 composites would be very suitable for light density and good mechanical and thermal properties area for variety of potential application.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

PENCIRIAN DAN SIFAT MISWAK (*Salvadora persica* L.) GENTIAN-DIPERKUKUHKAN ASID POLIKLAKTIK BIOKOMPOSIT

Oleh

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Pencirian dan sifat yang disediakan daripada gentian miswak (MF)/asid polilaktik (PLA) menggunakan pengadun dalaman dan pengacuan mampatan telah disiasat. Sifat kimia, fizikal dan haba bagi gentian yang tidak dirawat dan dirawat alkali biokomposit semuanya telah diperiksa. Nisbah aspek dan ketumpatan gentian didapati sangat rendah. Walaupun rawatan alkali mendedahkan permukaan gentian yang bersih dan jelas, bentuk gentian yang tidak dirawat menunjukkan kewujudan bahan sisa. Statistik TGA menunjukkan ketahanan haba gentian kerana kualitinya hanya mula merosot pada 88°C.Sebaliknya, rawatan alkali meningkatkan kestabilan haba gentian kepada 96°C. Kandungan gentian siwak memberi kesan ketara kepada sifat mekanikal dan fizikal komposit MF/PLA kerana pemuatan yang lebih tinggi meningkatkan ketumpatan, penyerapan air dan bengkak ketebalan. Pemuatan gentian 10wt% (M1) dalam PLA masing-masing menunjukkan kekuatan tegangan tertinggi (33.7 MPa) dan modulus tertinggi (2.9 GPa). Antara komposit lain, komposit M1 mempunyai penghabluran dan suhu lebur yang lebih baik pada 95°C dan 139°C serta kestabilan haba yang lebih baik apabila degradasi maksimum berlaku pada 341°C. DMA mendapati bahawa penambahan gentian miswak meningkatkan lengkung E' dan sedikit mengalihkan Tan δ ke suhu yang lebih rendah. Berbanding dengan matriks PLA, kebolehbiodegradan komposit adalah lebih baik kerana kemerosotan diperhatikan selepas 100 hari dalam ujian pengebumian tanah. Bagi sifat komposit yang dirawat alkali, FTIR menunjukkan anjakan berterusan apabila kepekatan alkali meningkat sehingga 3%. Keputusan modulus tegangan menunjukkan peningkatan prestasi mekanikal daripada 1% kepada 3% dengan beban gentian 30 wt.% dan mikrograf SEM menyokong keputusan tegangan. Komposit gentian dirawat alkali menunjukkan kestabilan terma yang lebih baik kerana keluk TGA menunjukkan sedikit kepada suhu tinggi berbanding dengan komposit gentian yang tidak dirawat dan keluk DSC menunjukkan peningkatan dalam suhu penghabluran untuk komposit gentian dirawat alkali yang direkodkan pada 116°C tertinggi untuk kepekatan alkali 1%. DMA mendapati bahawa penambahan gentian dirawat alkali dalam

PLA menjejaskan tahap keluk modulus penyimpanan dan keputusan analisis magnitud puncak Tan δ juga menunjukkan gentian dirawat alkali 1% mempunyai pematuhan yang lebih baik kepada matriks MF/PLA.Kesimpulannya, komposit di mana 10wt% pemuatan gentian miswak dalam PLA menunjukkan sifat keseluruhan yang lebih baik di kalangan komposit lain. Selain itu, komposit PLA bertetulang gentian dirawat alkali 1% menunjukkan peningkatan kestabilan haba antara kepekatan alkali lain dengan pemuatan gentian 10wt%. Oleh itu, komposit M1 akan sangat sesuai untuk ketumpatan cahaya dan kawasan sifat mekanikal dan terma yang baik untuk pelbagai aplikasi yang berpotensi.

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To Al-Quran, the greatest source of knowledge

Bring me sheets of iron"-until when he had levelled [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 its molten copper."(Al-Kahf: Verse 96)

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I hereby confirm that:

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LIST OF EQUATION

LIST OF ABBREVIATION

α	Alpha
µm	Micrometer
ASTM	American Society for Testing and Materials
DTG	Derivative Thermo-gravimetry
FTIR	Fourier Transform Infrared Spectroscopy
g	Gram
HDPE	High Density Polyethylene
IR	Infrared
LDPE	Low Density Polyethylene
MC	Moisture Content
MF	Miswak Fiber
NaOH	Sodium Hydroxide
OH	Hydroxyl Group
PBS	Polybutylene Succinate
PCL	Polycaprolactone
PE	Polyethylene
PHA	Polyhaydroxyalkanoates
PLA	Polylactic Acid
PMMA	Poly methyl methacrylate
PP	Polypropylene
PS	Polystyrene
PU	Polyurethane
SEM	Scanning Electron Microscopy
Tc	Crystallization Temperature

Tg	Glass Transition Temperature
TGA	Thermal-gravimetric Analysis
Tm	Melting Temperature
TPE	Thermoplastic elastomers
TS	Thickness Swelling
WA	Water Absorption
wt.%	Weight percentage
β	Beta

LIST OF UNITS

°C	Degree Celsius
%	Percentage
°	Degree
°C/min	Degree Celsius per minute
cm^{-1}	per centimetre
g/cm3	gram per cubic centimetre
GPa	Gigapascal
gsm	gram square meter
J/m	Joule per meter
Kg/m3	Kilogram per cubic meter
kJ	kilo Joule
kN	kilo Newtons
min	minute
mm	millimetre
MPa	Megapascal
ms-1	meter per second
rpm	rotation per minute
μ	micro

CHAPTER 1

INTRODUCTION

1.1 Background

Recently, there has been a rise in interest in the development of biodegradable plastic to replace the non-biodegradable polymers used in most applications. Utilizing biodegradable plastics would reduce pollution and waste disposal concerns while also being environmentally friendly. Synthetic and natural polymers can be used to make biodegradable plastics. Synthetic polymers are made from non-renewable petroleum resources, whereas natural polymers are generated in massive quantities and come from renewable resources.

Given its biodegradability, renewability, and high strength to weight ratio, polylactic acid (PLA) is one of the most extensively developed and commercially available polymers. Numerous studies have employed PLA extensively as the polymer matrix for composite materials for a wide range of purposes. Additionally, PLA was frequently employed in biomedical applications due to its biocompatibility, biodegradability, and non-toxic characteristics. It was also used for food packaging, tissue engineering, household engineering, and drug delivery applications.

There have been several attempts made to lower the cost of manufacturing these biodegradable polymers, including mixing them with relatively inexpensive filler to create cost-effective bio composite. Lignocellulosic fiber is one of the fillers that has frequently been used to enhance these biodegradable polymers. It can alter the properties of the biodegradable polymers and create an affordable biocomposite. Natural fibers have attracted considerable interest in a variety of uses, including packaging, furniture, building, and automotive (Shahzad et al., 2022).Additionally, it was determined that the lignocellulos fibers might be used to create engineered materials like composites and panels (Nair et al., 2022; Shahzad et al., 2022).

S. persica is frequently used as a toothbrush, and the roots, stems, leaves, and fruits are eaten as food in arid climates. The woods were also used to make firewood or charcoal, and the flowers were a rich source of nectar for honey bees. However, the most significant of all the plant's parts was its root, bark, stem, leaves, seeds, flowers, and fruits, which were used to make medicine for a variety of maladies and disorders, including digestive and circulatory(Farag et al., 2021; Mekhemar et al., 2021). Therefore, it is with this in mind that this abundant supply can be turned into a highly valuable product. Previously considered to be low-value items, wood, stems, and roots are now being used as potential reinforcement components in a range of thermoplastic composites(Chaaben, Taktak, Elleuch, et al., 2020; Savaş, 2018).

Numerous research from the last few years have documented the use of natural fiber as a reinforcing material in the manufacture of polymer composites(Darie-Nita et al., 2022; Jagadeesh et al., 2021; Kandemir et al., 2020; Mokhothu et al., 2022; Nair et al., 2022). This is mostly because natural fibers are flexible during processing, light weight, durable, affordable, eco-friendly, and renewable. Many of these fibers increase the polymer composite's characteristics while providing good mechanical performance. Although a great deal of research on various natural fibers and their composites has been published, this study aims to start the endeavours to incorporate a new natural fiber, namely miswak, as reinforcement in the creation of new composite materials for a variety of possible applications.

1.2 Problem Statement

Natural fiber reinforced polymer composites have gained popularity recently because of their wide range of qualities that make them appropriate for a variety of prospective applications. Natural fibers are gaining popularity due to their low cost, biodegradable, recyclable, nonabrasive, flammable, lightweight, and nontoxic qualities. But the processing of raw materials and the creation of composite structures, which is still difficult today, require more fundamental understanding. The quality of natural fibers is influenced by numerous characterizations and property profiles.

Natural fibers such as kenaf, pineapple leaf fiber, banana fiber, and bamboo fiber have all been given varied characterizations and profiling properties, whilst miswak (*salvadora persica*) fiber research is still in its early stages. Most scientists studying miswak (*salvadora persica*) only concentrate on its biological, pharmacological, and periodontal characteristics, with the primary application being dental-based items. In this work, a distinct approach is employed to use miswak fiber (MF) to construct composites, and knowing how variable fiber loading affects composite properties helps to provide insight into the potential of these composites for further study.

Additionally, the surface properties of natural fibers could be improved by choosing the right chemical and surface treatments. Because of its efficiency and affordability, alkaline treatment (NaOH) is the most used chemical treatment available. These fiber treatments increase the adhesive properties of the final polymer composite, decrease the proportion of water intake, and improve overall performance. Natural fibers' physical, thermal, and mechanical characteristics are studied to determine whether they are suitable for use as discontinuous reinforcing materials while also creating a database for material selection.

PLA polymer has appealing physical and mechanical qualities such as good clarity, barrier properties, superior modulus, and strength performances during competitive materials and processing costs of biopolymers. However, the price of PLA's raw materials is relatively higher than that of traditional synthetic polymers, which has reduced industrialists' inclination to use it. It is more difficult to employ PLA as the matrix of a composite since it is susceptible to thermal distortion temperature, brittle, and has low hydrolysis resistance and flexibility. As PLA has several inherent disadvantages, including a high cost and brittleness, attempts have been made to address these by melting PLA with natural fibers to lower the cost.

1.3 Hypothesis

Miswak fiber, derived from *S. persica* roots and combined with PLA pallets, may be utilized as fiber reinforcement in natural fiber composites with greater sustainable benefits, such as renewable resources and a smaller carbon footprint. When combining miswak fiber with PLA, however, its performance degrades as the weight percentage of miswak fiber increases. By testing and obtaining their behaviors and properties, there should be a good and efficient mixing quantity of blending weight percentage ratio between them. The idea is that it may be utilized in a specific application, such as a toothbrush handle.

1.4 Research Questions

1) What are the characteristics of miswak fiber?
2) What is the performance and behavior of miswak fiber-reinforced PLA?
3) How does fiber treatment affect the performance of miswak fiber-reinforced PLA?

1.5 Objectives

The aim of this research is:
To evaluate behaviors and performance of miswak as fiber reinforcement in PLA matrix.

The objectives of the research are as follows:

1) To characterize the physical, chemical, and thermal properties of miswak fiber
2) To determine the effect of different miswak fiber loading on physical, mechanical, chemical, thermal, and degradable properties of miswak fiber reinforced PLA bio composites.
3) To determine the effect of different alkaline concentration on the tensile, FTIR and thermal properties of miswak fiber reinforced PLA bio composites.

1.6 Scope and Limitations

1) In this study, miswak sticks were supplied by Al-Khair Natural Product, Karachi, Pakistan. There are a distinct set of properties that distinguish them from other natural fibers. Hence, the properties of MF will be different from other natural fibers.
2) Miswak sticks have intense smell and fragrance, which may prevent certain users from using them. As a result, it is advised to completely dry the miswak stick. Its persistently strong odor signals that it is fresh, damp, and susceptible to fungal attack if not dried completely.
3) Given the ease of processing and fiber limitations, the maximum fiber loading was fixed at 30 wt. %. The fiber must be dried, crushed, and ground; therefore, it takes a while to obtain even 1 kg of MF. As it takes a long time to acquire 1 kg of MF, the recovery of the fiber is low. As a result, the ability to produce numerous formulations is restricted by the minimal quantity of fiber collected for a prolonged period.
4) As the fiber has a poor recovery rate and must be collected in such small quantities to perform other types of fiber treatment, this study primarily focuses on the alkali treatment of fiber.
5) Polylactic acid was made by Nature Works and supplied by Polycomposite Sdn. Bhd., a local company. It has a unique set of features that set it apart from other biopolymer matrixes.
6) All bio composite boards were produced using an internal mixer and the compression molding technique. The internal mixer's mixing is prone to human errors as the material must be manually inserted into the equipment. Also, compression molding is susceptible to technical errors because the heat on the plate is not always evenly distributed. Therefore, this inaccuracy will be present in the sample result.

1.7 Potential Application

Since its primary applications are in dental and oral hygiene, miswak was chosen for this study's reinforcing fiber in PLA. The wealth of benefits miswak offer to the teeth, gums, and mouths in terms of antibacterial and antioxidant characteristics has been demonstrated by numerous research. As a result, when miswak is incorporated into PLA matrix, the idea is that these newly developed composite materials may be used in ways that are comparable to how a toothbrush is used. Moreover, this composite material can be a coating of the miswak stick, which would shield it from dangers outside, is another potential use. The miswak stick's coating would also make it more appealing to use as a toothbrush in our daily lives. In relation to biomedical applications, PLA matrix was frequently used. Consequently, combining miswak fiber with PLA matrix could offer a variety of benefits, including in biomedical and dentistry applications.

1.8 Thesis Outline

The introduction is presented in the first chapter of the thesis. The background of natural fibers and the fundamentals of natural fibers are presented in the introduction section. The possibility of natural fibers to be used as an alternative in industries is then examined in general. The study's goals and scope are given in the first chapter.

The second chapter contains literature reviews that have been taken from journals, books, and websites that are relevant to the study's objectives. Natural fibers and reinforced fibers are studied. The performance of natural fibers as an option is reviewed in detail. The findings and rationale of the study, methodology, and theoretical background are provided through evaluations of past studies on using natural fibers as an alternative.

The research methodology is presented in the third chapter. The procedures for preparing miswak fiber are explained in this chapter. In addition, the testing procedures and tools utilized for the experiment of miswak fiber and also miswak and PLA-reinforced composites are detailed. The technique for selecting miswak fiber is also described in depth. Finally, the experimental design utilized for planning the experimental runs and analyzing the findings is described.

The results and discussion section of the thesis is covered in the fourth chapter. The first parts are devoted to the miswak fiber characterization and PLA property evaluation. The remaining sections concentrate on the outcomes of several trials. A comparison of the current outcome with results reported in the literature by other researchers is also presented.

Finally, the fifth chapter discusses the research's conclusion, future work recommendations, and importance to the local community, manufacturers, and future researchers.

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