ENHANCING THE POTENTIAL OF AFRICAN BAMBOO *Oxytenanthera abyssinica* VIA ASAM PULPING AS FEEDSTOCK FOR PACKAGING-GRADE PULP

By

AREEJ FATHELRAHMAN ABDALLAH HASSABELNABI

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

November 2023

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DEDICATION

This Thesis is Whole-heartedly Dedicated To:
My Lovely Big Family
My Father, Mother, and Brothers
My Beloved Small Family
My Husband and My Daughters

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

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Chairman : Professor Mohammad Jawaid, PhD
Institute : Tropical Forestry and Forest Products

Bamboo is a versatile and fast-growing plant that generates cellulose with long fibres. The cellulose can be used to produce paper with superior mechanical characteristics in comparison to paper derived from softwood. In Sudan, homesteads and small farms primarily use bamboo for constructing buildings, sheds, fences, and dividers. This study used bamboo as a raw material to alleviate the strain on foreign currency reserves caused by the importation of paper for use in packaging factories. This research objective is to investigate the potential of bamboo (*Oxytenanthera abyssinica*) culms as a feedstock for producing packaging-grade pulp using the alkaline sulfite, anthraquinone, and methanol (ASAM) pulping method, as well as to enhance the air barrier and antimicrobial properties of the pulp by incorporating nanofibrillated cellulose and chitosan. This is accomplished by analysing the fibre characteristics and chemical composition of the fibres, evaluating how ASAM pulping affects the properties of unbleached pulp and paper under different chemical conditions, evaluating how the performance of chitosan and nanofibrillated cellulose as wet-end additives in paper is affected by beating revolutions, and evaluating the antimicrobial properties of enhanced-paper sheets against food-contaminating microorganisms. The fibre characteristics were evaluated via the maceration method. The average fibre length of the samples was 1835 μm and the average fibre diameter was 16.71 μm. The chemical analysis was conducted using TAPPI standard methods. The composition comprised 50.19% cellulose and 25.67% lignin. The chips were cooked in a MK Twin Digester with a 70/30 ratio of Na_2SO_3/NaOH, 15% methanol, 0.1% anthraquinone, a 7:1 liquor-to-chips ratio, and a maximum temperature of 170 °C for 90 min. The best pulp yield was 45.24%, the kappa number was 11.04, the tensile index was 78.71 Nm/g, and the burst index was 7.4 kPa/m^2. This was done with 17% active alkali and 8,000 beating revolutions. Based on these findings, the pulp was prepared using a disintegrator at 1,000 for 45 seconds by adding chitosan at 0.5, 1, 1.5, and 2%, followed by adding nanofibrillated cellulose at 5 and 10% (oven-dried pulp) at 180,000 revolutions. the optimal mechanical properties in slurry beaten at 6,000 revolutions (5% nanofibrillated cellulose and 1.5% chitosan) with tensile and burst indices of 85.16 Nm/g and 7.69 kPa.m^2/g, respectively. Additionally, the slurry maintained its higher smoothness (1630.00 mL/min) and porosity (9.30 mL/min), achieving more advantageous barrier properties. The optical density at 600 nm served to quantify antimicrobial activity. Results demonstrated the highest antimicrobial resistance of enhanced paper against the yeast *Candida albicans* ATCC 90028 by 49.75% and gram-positive bacteria *Staphylococcus aureus* ATCC 10708 by 43.96%. In conclusion, this bamboo has fibres and chemical properties that allow it to generate a high pulp yield with a low kappa number using the ASAM pulping process, where the addition of nanofibrillated cellulose-filled voids between the fibres improved the mechanical and barrier properties of the sample, and chitosan increased resistance to certain microbes. These biopolymers reduce energy consumption by lowering the beating rate. Lastly, these enhanced papers possess properties suitable for food packaging applications.

Keywords: ASAM pulping, nanofibrillated cellulose, chitosan, antimicrobial properties, food packaging.

SDG: GOAL 8: Decent work and economic growth, GOAL 9: Industry, innovation and infrastructure, GOAL 13: Climate action.

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

MENINGKATKAN POTENSI BULUH AFRIKA *Oxytenanthera abyssinica* MELALUI PEMPULPAAN *ASAM* SEBAGAI BAHAN MENTAH UNTUK PULPA GRED PEMBUNGKUSAN

Oleh

AREEJ FATHELRAHMAN ABDALLAH HASSABELNABI

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Buluh ialah tumbuhan serba boleh dan cepat membesar yang menjana selulosa dengan gentian panjang. Selulosa boleh digunakan untuk menghasilkan kertas dengan ciri mekanikal yang unggul berbanding dengan kertas yang diperoleh daripada kayu lembut. Di Sudan, rumah dan ladang kecil terutamanya menggunakan buluh untuk membina bangunan, bangsal, pagar dan pembahagi. Kajian ini menggunakan buluh sebagai bahan mentah untuk mengurangkan tekanan rizab mata wang asing yang disebabkan oleh pengimportan kertas untuk kegunaan kilang pembungkusan. Objektif kajian ini adalah untuk menyiasat potensi batang buluh (*Oxytenanthera abyssinica*) sebagai bahan suapan untuk menghasilkan pulpa gred pembungkusan menggunakan kaedah pulping alkali sulfit, antrakuinon, dan metanol (ASAM), serta meningkatkan penghalang udara dan sifat antimikrob. daripada pulpa dengan menggabungkan selulosa nanofibrilasi dan kitosan. Ini dicapai dengan menganalisis ciri gentian dan komposisi kimia gentian, menilai bagaimana pulpa ASAM mempengaruhi sifat pulpa dan kertas yang tidak diluntur dalam keadaan kimia yang berbeza, menilai bagaimana prestasi kitosan dan selulosa nanofibrilasi sebagai bahan tambahan akhir basah dalam kertas terjejas dengan menewaskan revolusi, dan menilai sifat antimikrob pada helaian kertas yang dipertingkatkan terhadap mikroorganisma yang mencemarkan makanan. Ciri-ciri gentian dinilai melalui kaedah maserasi. Purata panjang gentian sampel ialah 1835 μm dan purata diameter gentian ialah 16.71 μm. Analisis kimia dijalankan menggunakan kaedah piawai TAPPI. Komposisi tersebut terdiri daripada 50.19% selulosa dan 25.67% lignin. Kerepek telah dimasak dalam MK Twin Digester dengan nisbah 70/30 Na_2SO_3/NaOH, 15% metanol, 0.1% antrakuinon, nisbah minuman keras-ke-cip 7:1, dan suhu maksimum 170 °C selama 90 minit. Hasil pulpa terbaik ialah 45.24%, nombor kappa ialah 11.04, indeks tegangan ialah 78.71 Nm/g, dan indeks pecah ialah 7.4 kPa/m^2. Ini dilakukan dengan 17% alkali aktif dan 8,000 putaran pukulan. Berdasarkan penemuan ini, pulpa disediakan menggunakan disintegrator pada 1,000 selama 45 saat dengan menambah kitosan pada 0.5, 1, 1.5, dan 2%, diikuti dengan menambah selulosa nanofibrilasi pada 5 dan 10% (pulpa kering ketuhar) pada 180,000 pusingan. sifat mekanikal optimum dalam buburan yang dipukul pada 6,000 pusingan (selulosa nanofibrilasi 5% dan 1.5% kitosan) dengan indeks tegangan dan pecah masing-masing 85.16 Nm/g dan 7.69 kPa.m^2/g. Selain itu, buburan mengekalkan kelicinan yang lebih tinggi (1630.00 mL/min) dan keliangan (9.30 mL/min), mencapai sifat penghalang yang lebih berfaedah. Ketumpatan optik pada 600 nm berfungsi untuk mengukur aktiviti antimikrob. Keputusan menunjukkan rintangan antimikrob tertinggi bagi kertas dipertingkatkan terhadap yis *Candida albicans* ATCC 90028 sebanyak 49.75% dan bakteria gram positif *Staphylococcus aureus* ATCC 10708 sebanyak 43.96%. Kesimpulannya, buluh ini mempunyai gentian dan sifat kimia yang membolehkannya menghasilkan hasil pulpa yang tinggi dengan nombor kappa yang rendah menggunakan proses pulping ASAM, di mana penambahan lompang berisi selulosa nanofibrilasi di antara gentian meningkatkan sifat mekanikal dan penghalang sampel, dan kitosan meningkatkan ketahanan terhadap mikrob tertentu. Biopolimer ini mengurangkan penggunaan tenaga dengan menurunkan kadar denyutan. Akhir sekali, kertas yang dipertingkatkan ini mempunyai sifat yang sesuai untuk aplikasi pembungkusan makanan.

Kata Kunci: Pengpulpaan *ASAM*, selulosa nanofibrilasi, kitosan, sifat antimikrob, pembungkusan makanan.

SDG: MATLAMAT 8: Pekerjaan baik dan kemajuan ekonomi, MATLAMAT 9: Industri, inovasi dan infrastruktur, MATLAMAT 13: Tindakan iklim.

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LIST OF ABBREVIATIONS

AA	Active alkali
AB	Alcohol Benzene
AC	Ash Content
ANOVA	Analysis of variance
AQ	Anthraquinone
ASAM	Alkaline sulfite anthraquinone & methanol
B	Brightness
B. I	Burst Index
BNC	Bacterial nanocellulose.
ºC	Centigrade
CELL	Cellulose
CH	Chitosan
CH_3OH	Methanol
CNC	Cellulose NanoCrystals
CWC	Cold Water Content
CWT	Cell Wall Thickness
DMRT	Duncan Multiple Range Test
DPX	The (D) is for distyrene, a type of the plastic polystyrene. The (P) represents a plasticiseran additives designed to reduce the viscosity of a material. The (X) is for xylene.
EDATA	Ethylene Diamine Tetraacetic Acid
FD	Fibre Diameter
F. E	Folding Endurance
FL	Fibre Length
FLD	Fibre Lumen Diameter
FR	Flexibility Ratio

FRA	Forest Resources Assessment
g	gram
HOL	Holocellulose
HWC	Hot Water Content
ISO	International Organization for Standardization
LIGN	Lignin
MCC	Microcrystalline Cellulose
MeOH	Methanol
Min	Minute
mL	Millimetre
NaOH	Sodium hydroxide
Na_2SO_3	Sodium Sulfite
Na_2O	Sodium Oxide
NFC	Nanofibrillated cellulose
Nm (Kg)	Net mass Kilogram
O	Opacity
o.d	oven dry weight
OP	O_2 permeability
OPEFB	Oil Palm Empty Fruit Bunch
P	Primary
RR	Runkel Ratio
SAS	Statistical Analysis Software
S1	Secondary Cell Wall (first layer)
SR	Slenderness Ratio
TEM	Transmission electron microscopy
Temp	Temperature
Ten. I	Tensile Index

TGA	Thermogravimetric Analysis
T. I	Tear Index
WVP	Water vapor permeability
XRD	X-ray diffraction Analysis

CHAPTER 1

INTRODUCTION

1.1 General Background

In 2020, the worldwide output of paper and paperboard amounted to 401 million metric tonnes, indicating a slight decline of approximately 1% compared to the preceding year (Statista, 2022). The increase resulted in significant deforestation, with a rate of 420 million tonnes of paper and cardboard being produced globally each year (Anon, 2022). Nevertheless, the projected increase in paper demand is expected to double from 2005 to 2030. In order to mitigate the depletion of rainforests, numerous efforts have been undertaken to identify alternate sources of fibre for paper production, notably those derived from non-woody plants (Bidin et al., 2015). Non-woody plants fibrous materials such as wheat straw, rice straw, cotton stalks, canola stalks, sugarcane bagasse, switchgrass, and reed are valuable sources of fibre for pulp manufacture in nations facing a scarcity of wood resources (Gülsoy and Şimşir, 2018). Using non-woody fibres for pulp and paper manufacture offers various advantages, such as a shorter growing and harvest period compared to the longer cycles required for wood production, which facilitates the cooking process for the former (Sharma et al., 2023; Tye et al., 2016), optimal irrigation and fertilisation demands are moderate (Obi Reddy et al., 2014; Onakpoma et al., 2019), the reduced lignin content facilitates the chemical and mechanical processes involved in non-woody pulping (Kamoga et al., 2013), non-woody fibres necessitate a reduced amount of chemical substances, lower energy consumption, and less time expenditure during the pulping process, all at a comparatively affordable cost (Areej et al., 2023).

Non-woody materials have lower activation energies due to their lower lignin concentration, making them generally more easily delignified compared to wood (Sarkar et al., 2021). The non-woody fibres yield pulp that is easily processed, resulting in raw materials that are highly suitable for producing specialty grades of paper. Additionally, the bleached pulp obtained from these fibres is of superior quality (Mannai et al., 2018). According to the Food and Agriculture Organisation of the United Nations (2022) report, there are 43 nations involved in the manufacture of pulp using nonwood fibres. China and India have recently emerged as the leading producers, with China producing over 5 million metric tonnes and India producing over 3 million metric tonnes (Sapuan et al., 2023). Recently, there has been an increased focus on using annual grass for the manufacturing of pulp, paper, paperboard, and cellulose derivatives. Cellulose fibres derived from nonwood raw materials, including bamboo, bagasse, jute, cereal straw, flax, esparto grass, reeds, and sisal, are increasingly being used in emerging countries (Maghchiche, 2023). The percentage of bamboo pulp experienced a significant increase, rising from 2.7% in 2000 to 21.4% in 2015 (Liu et al., 2018).

The growing ecological consciousness of advanced societies has heightened the need for more environmentally-friendly manufacturing methods for goods like paper and cardboard. Conventional chemical and semi-chemical pulping methods generate substantial quantities of a noxious waste known as “black liquor,” which is particularly detrimental to the environment, especially when sulphur compounds are employed. To address this issue, the industrial and scientific sectors are increasingly focusing on utilising alternative raw materials to replace conventional options like annual plants or agricultural and forestry residues. Additionally, they are working towards developing new pulping methods that rely on less polluting and more readily recoverable reagents, such as organic solvents (Rodríguez et al., 2018). The organosolv method offers the benefit of utilising compounds that are less detrimental to human health and the environment in comparison to conventional methods (such as kraft and sulfite) that result in significant pollution issues (Sadiku and Yusuph 2023). The process employs organic solvents, such as low-boiling solvents (e.g., methanol, ethanol, and acetone), as delignifying agents. These solvents can be readily recovered through distillation. The methods do not produce sulphur compounds (Wutisatwongkul et al., 2016).

The Alkaline sulfite anthraquinone methanol pulping process is a modified form of the alkaline sulfite process that incorporates anthraquinone and methanol. This modification aims to improve the rate of delignification and the selectivity of pulping (Klinaberg, 2013). As a result, ASAM pulping completely lacks the offensive smell that methyl mercaptan emits during kraft pulping. Additionally, the effortless and eco-friendly bleaching process of the resulting pulp is a notable benefit. Therefore, this pulping method is a strategy for generating pulp without causing pollution. It achieves a high rate of removing lignin with very low kappa numbers, resulting in a bright pulp. Additionally, it yields a large amount of pulp and makes the bleaching process easier (Jahan et al., 2021; Paridah et al., 2018). Numerous studies have investigated the utilisation of ASAM in the pulping process of hardwood ; *Trema orientalis* (Jahan et al., 2007), *Eucalyptus globulus* Labill (Gominho et al., 2014), *Albizia lebbeck* (Elzaki et al., 2012a), softwood; Pine (Patt et al., 1991a; Borgards et al., 1993), Red Pine (Paik et al., 1994), poplar and mossy oak (Winkler and Patt, 1988), and non-wood biomass; Kenaf (Khristova et al., 2002), corn stalk (Jahan et al., 2006), bamboo (Khristova et al., 2006; Moradbak et al., 2016 a,b) and cotton stalk (Osman Khider et al., 2012).

1.2 Problem Statement

Sudan is a vast African nation, encompassing an expansive territory of 1,882,000 square kilometres (Ismail, 2020). Sudan is a country rich in forests, with a diverse array of tree species. It is home to over 3156 species, spanning 1137 genera and 170 families (Bekele, 2021). Furthermore, the bamboo forest area in Sudan has decreased from 40,000 hectares in 1990 to 30,000 hectares in 2020 (FRA, 2020). The current uses of this bamboo are mostly limited to the construction of house buildings, huts, and sheds (known as Rakoba in native Sudanese). Additionally, homesteads and small farms use it as fences, decorative units, and dividers. The food packaging sector in Sudan has shown consistent expansion in recent years, necessitating study in the broader subject of packaging and specifically in the area of food packaging. Sudan possesses six packaging facilities; however, it relies on imports of paper from Saudi Arabia, Singapore, and Sweden for its raw materials (Khider, 2013). Moreover, the imports of packaging paper throughout the period 2010–2020 (Figure 1.1) exhibit variability in both quantities and values, with fluctuations occurring from year to year. The quantities showed an upward trend from 2013 to 2019, then a decline in 2020. However, the negative effects of the COVID-19 pandemic on Sudan led to an increase in value, counterbalancing the decline.

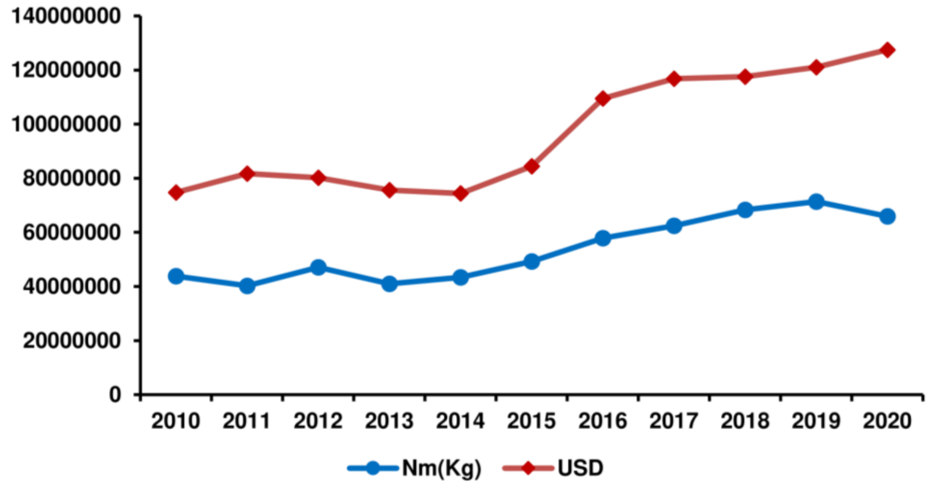

Figure 1.1 : The Imports of Packaging Paper during the Period 2010–2020 in Sudan
(Source: SCA, 2020)

Sudan, similar to the majority of developing countries, relies heavily on imports to meet its demands for pulp and paper. Assessing locally accessible raw materials is necessary to determine their suitability for packaging paper production. This involves analysing their compatibility for the pulping process in order to fulfil the growing demands of both cultural and industrial sectors, promote economic development, and reduce the need to spend foreign currency. Moreover, the escalating cost of packaging paper in relation to the burden of foreign exchange serves as a significant incentive to foster the domestic packaging paper industry by utilising rapidly growing raw materials with elongated fibres, such as bamboo. Furthermore, bamboo is an exceptionally flexible and rapidly growing plant, making it a valuable and abundant renewable resource on earth. The growth of this species requires a shorter maturation period of 3 to 4 years before it can be harvested and used, in comparison to softwood or hardwood sources of fibre. In the future, bamboo will emerge as the preeminent alternative to wood. Consequently, bamboo has become the primary subject of research in recent years (Hakeem et al., 2015). Moreover, bamboo is extensively used as a fibrous primary material for the production of paper, owing to its elongated fibres. The resulting pulp produces paper with excellent mechanical strength, in contrast to paper made from pulp derived from softwood. Due to this factor, bamboo has garnered growing interest as a distinct raw resource, and its chemical pulp is also used as reinforcement pulp in certain countries (Bhardwaj, 2019). Besides that, bamboo has rapid growth, high toughness, as well as antibacterial and anti-odour properties (Haile et al., 2021).

Several research investigating the antibacterial characteristics of bamboo have revealed lignin as the main source of the antibacterial component (Ramful et al., 2022; Wang et al., 2019a). Therefore, employing bamboo pulp without bleaching is helpful for producing unbleached paper, which is beneficial for applications in food packaging. However, food packaging materials must adhere to particular criteria. The primary purpose of food packaging is to extend food's shelf life and ensure its quality and safety. The barrier properties have a crucial role in minimising the interchange of gases and water vapour between the food and its surrounding environment, thereby reducing the rates of chemical, physical, and microbiological alterations. Water vapour permeability (WVP) and oxygen permeability (OP) are crucial characteristics for materials designed for food preservation (Azeredo et al., 2017). Therefore, in this study, will pulp chips from bamboo (*Oxytenanthera abyssinica*) using an alkaline sulfite anthraquinone and methanol process. In addition, nanofibrillated cellulose (NFC) and chitosan will be employed to enhance the mechanical strength, barrier qualities, and antibacterial characteristics of the bamboo paper. Nanofibrillated cellulose (NFC) is an excellent nanomaterial that possesses distinctive physical and mechanical characteristics, including high tensile strength, low density, oxygen barrier qualities, transparency, chemical modifiability, biodegradability, and biocompatibility. However, chitosan possesses distinctive physical and mechemical properties, including a larger surface area, resulting in a higher surface charge density and more cationic sites. Additionally, chitosan exhibits a greater affinity for bacterial cells due to its capability to tightly adhere to their surface, leading to membrane disruption and cell death (Hassan et al., 2016).

1.3 Objectives

The main aim of this study was to assess the potential of Sudanese bamboo culms (*Oxytenanthera abyssinica*) as feedstock in the production of packaging-grade pulp through the use of the alkaline sulfite anthraquinone and methanol (ASAM) pulping method. Additionally, the study aimed to enhance the air barrier and antimicrobial properties of the paper by incorporating nanofibrillated cellulose (NFC) and chitosan. The study was divided into four main sections: evaluation of the fibre morphology and chemical composition of *Oxytenanthera abyssinica* bamboo; optimisation of the ASAM pulping process and assessment of its properties; evaluation of the ASAM unbleached pulp; and enhancement of paper properties through the addition of NFC and CH, followed by the evaluation of the paper's antimicrobial properties. The specific objectives were to:

1. Identify the fibre morphology and chemical composition of bamboo fibre.
2. Assess the impact of ASAM pulping under varying chemical conditions on the properties of unbleached pulp and paper.
3. Determine the influence of beating revolutions on the performance of nanofibrillated cellulose and chitosan as wet-end additives in paper.
4. Appraise the antimicrobial properties of the modified paper sheet against food-polluting microorganisms.

1.4 Scope of the Study

The bamboo plant was selected for this research because its fast growth and the presence of long fibres, which allow it to produce paper with unique qualities suitable for the production of packaging sheets. This study covers the fiber characteristics and chemical analysis of bamboo culms, ASAM pulping of bamboo culms, evaluation of the performance of nanofibrillated cellulose and chitosan additives as wet-end agents on the physical and mechanical properties of papers, and evaluation of the microbial resistance of these enhanced papers. This study involved evaluating the fibre and chemical characteristics of bamboo culms due to their significant impact on the quality and production of paper. Subsequently, the bamboo chips underwent cooking utilising the ASAM pulping method, a cooking method that was first documented in 1986. This process stands out for its environmental consciousness and sustainability, as it doesn't release any harmful substances into the air. Additionally, it effectively eliminates a significant amount of lignin, resulting in pulp that has a high percentage, high viscosity, and a low kappa value. Afterwards, produce paper at various beating rates, specifically 2,000, 4,000, 6,000, and 8,000 revolutions.

The samples exhibiting superior pulp quality and a low kappa number were chosen. Then, the addition of ecologically friendly and biodegradable biopolymers, including nanofibrillated cellulose and chitosan, during the papermaking process will enhance its characteristics. These materials are recognised for their green nature, biodegradability, non-toxicity, and eco-friendliness. The nanofiber functions by filling the spaces between the fibres, thus augmenting the paper's capacity and bolstering its strength. Chitosan functions as an antimicrobial agent. These biopolymers were loaded as wet-end using beating forces of 4,000 and 6,000 revolutions to determine the optimal sample with optimal physical, mechanical, thermal, and barrier properties. This sample was then tested against food-contaminating microbes. The enhanced-papers were evaluated for their antibacterial effectiveness against gram-positive bacteria (*Staphylococcus aureus* ATCC 43300), gram-negative bacteria (*Escherichia coli* ATCC 25922 and *Salmonella choleraesuis* ATCC 10708), and the fungus *Candida albicans* ATCC 90028 via optical density at 600 nm.

This study did have some limitations, including limited financial resources and time limitations, as it was planned to conduct a diffusion disc test to evaluate microbial resistance and confirm the findings with an optical density test. I actually started the evaluation of the enhanced-papers by employing the diffusion test method. It gave positive results with the appearance of inhibition zone. However, due to financial limitations, I was unable to complete the entire set of samples and instead relied solely on the optical density method.

1.5 Significance of the Study

This study is anticipated to result in the utilisation of local raw resources, specifically bamboo, which is abundant in wide areas of up to 30 hectares, for paper manufacturing. The aim is to manufacture bamboo papers with excellent physical and mechanical characteristics, which will alleviate the foreign exchange issues associated with importing papers from abroad. Moreover, minimising the use of plastic containers composed of non-biodegradable petroleum components is crucial to mitigating the energy crisis, global warming, and various other environmental issues. In addition, the production of eco-friendly papers suitable for food packaging applications is expected by employing a pulping process and incorporating non-toxic and biodegradable biopolymers. This is in contrast to industrial polymers, which have a detrimental impact on the environment due to their non-biodegradable nature.

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