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EXTRACTION OF CELLULOSE FROM RICE HUSK, DURIAN HUSK AND PINEAPPLE PEELS USING ACID AND ALKALINE PRE-TREATMENT WITH BLEACHING PROCESS

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DECLARATION

I declare that this thesis entitled "Extraction of Cellulose from Rice Husk, Durian Husk and Pineapple Peels Using Acid and Alkaline Pre-Treatment with Bleaching Process" is the result of my own research except as cited in the references.

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LIST OF SYMBOLS

%	Percentage
°	Degrees
θ	Delta
°C	Degree Celsius
β	Beta

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LIST OF ABBREVIATIONS

AFM	Atomic force microscopy
C	Carbon
cm	Centimeter
CrI	Crystallinity index
FTIR	Fourier transform infrared spectroscopy
g	Gram
GPa	Gigapascal pressure unit
H	Hydrogen
mg	Milligram
min	Minute
ml	Milliliter
mL/min	Milliliter per minute
mm	Millimeter
MPa	Megapascal pressure unit
nm	Nanometer
O	Oxygen
OH	Hydroxyl group
rpm	Revolutions per minute
SEM	Scanning electron microscope
TEM	Transmission electron microscope
TGA	Thermalgravimetric analysis
v/v	Volume per volume
W	Watt
XRD	X-ray Diffraction

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Extraction of Cellulose from Rice Husk, Durian Husk and Pineapple Peel Using Acid and Alkaline Pre-Treatment with Bleaching Process

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ABSTRACT

Cellulose is a crucial fiber polymer that gives plants strength and high resistance to stretching, making it vital in industries such as paper, textiles, and biofuels. The material's hydrophobic properties provide excellent durability and solvent resistance, leading to prolonged performance. The aim is to extract cellulose from agricultural byproducts such as pineapple peels, rice husks, and durian husks using alkaline pretreatment and bleaching techniques to enhance purity. The extracted cellulose will undergo comprehensive analysis utilizing SEM, FTIR, XRD, and TGA. This thorough evaluation seeks to analyze cellulose samples' microstructural, chemical, and thermal properties to enhance understanding of their potential commercial applications. Extracting cellulose from rice, durian, and pineapple peels includes a practical alkaline pretreatment approach using sodium or potassium hydroxide, followed by bleaching with chlorine dioxide or hydrogen peroxide. RH 1 and PP 1 had the highest cellulose percentages at 75% and 74%, respectively, followed by DH 1 at 70%, DH 2 at 66%, and PP 2 at 66%. The samples analyzed indicated that the alkaline treatment substantially boosted the cellulose content by eliminating hemicellulose, lignin, and other impurities. During a study on cellulose sources, the pineapple peel was determined to be the most efficient compared to rice husk and durian husk. Its reduced processing time and the generation of a translucent residue revealed a superior conclusion in cellulose extraction. This study aids in identifying agricultural byproducts that can be used as cellulose sources to create biodegradable microbeads in the future.

Keywords: Hydrophobic Properties, Rice Husk, Durian Husk, Pineapple Peel, Cellulose, Agriculture Byproducts, Extraction, Biodegradable Microbeads

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Ekstraksi Selulosa dari Sekam Padi, Sekam Durian, dan Kulit Nenas Menggunakan Pra-Pemprosesan Asid dan Alkali dengan Proses Pemutihan

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ABSTRAK

Selulosa adalah polimer gentian penting yang memberikan tumbuhan kekuatan dan ketahanan yang tinggi terhadap regangan, menjadikannya penting dalam industri seperti kertas, tekstil, dan bio-bahan api. Sifat hidrofobik bahan ini memberikan daya tahan yang sangat baik dan ketahanan terhadap pelarut, yang membawa kepada prestasi yang berpanjangan. Matlamatnya adalah untuk mengekstrak selulosa daripada sisa pertanian seperti kulit nanas, sekam padi, dan kulit durian menggunakan kaedah pra-rawatan alkali dan penjerusan untuk meningkatkan kebersihan. Selulosa yang diekstrak akan menjalani analisis menyeluruh menggunakan SEM, FTIR, XRD, dan TGA. Penilaian menyeluruh ini bertujuan untuk menganalisis sifat mikrostruktur, kimia, dan terma sampel selulosa untuk meningkatkan pemahaman terhadap aplikasi komersialnya yang berpotensi. Mengekstrak selulosa dari sekam padi, durian, dan kulit nanas melibatkan pendekatan pra-rawatan alkali yang praktikal menggunakan natrium atau kalium hidroksida, diikuti dengan penjerusan dengan dioksida klorin atau hidrogen peroksida. RH 1 dan PP 1 mempunyai peratusan selulosa tertinggi pada 75% dan 74% masing-masing, diikuti oleh DH 1 pada 70%, DH 2 pada 66%, dan PP 2 pada 66%. Sampel yang dianalisis menunjukkan bahawa rawatan alkali secara ketara meningkatkan kandungan selulosa dengan menghilangkan hemiselulosa, lignin, dan kotoran lain. Semasa kajian tentang sumber selulosa, kulit nanas ditentukan sebagai yang paling cekap berbanding dengan sekam padi dan kulit durian. Masa pemprosesan yang lebih singkat dan pembentukan sisa yang transparan menunjukkan kesimpulan yang lebih unggul dalam pengekstrakan selulosa. Kajian ini membantu mengenal pasti sisa pertanian yang boleh digunakan sebagai sumber selulosa untuk mencipta mikrobead yang boleh terdegradasi pada masa hadapan.

Kata kunci: Sifat Hidrofobik, Sekam Padi, Kulit Durian, Kulit Nanas, Selulosa, Sisa Pertanian, Pengekstrakan, Mikrobead Boleh Terdegradasi

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CHAPTER 1

INTRODUCTION

1.1 Background of study

Cellulose is a crucial polymer that imparts structural stability to the plant cell wall. Brigham (2017) states cellulose is a fibrous polymer that does not dissolve in water. Crystalline cellulose exists and is held together by intramolecular hydrogen bonds. The resulting polymer is hydrophobic and possesses a tensile strength ranging within the megapascals (MPa). The tensile strength of cellulose is a critical factor, ranging from MPa, demonstrating outstanding thermochemical characteristics (Li et al., 2020)

Cellulose is an essential compound widely used in the industrial and commercial sectors to manufacture various paper and textile products. The exceptional adaptability and usefulness of cellulose make it essential for plant growth and a wide range of human activities. The structural integrity of cellulose is due to its inability to dissolve in water and most organic solvents.

The exceptional mechanical strength of cellulose arises from its inflexible and fibrous structure. The firmness and inflexibility of plant cell walls contribute to their capacity to endure growth and environmental strain. Cellulose confers plants with structural rigidity, allowing them to uphold their form and endure physical stress. Cellulose is an essential constituent of the human diet as it functions as a dietary fibre. Although humans lack the cellulase enzyme, the unique qualities of cellulose make it

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highly beneficial in various production processes. Cellulose-based items such as paper, textiles, rayon, and cellophane are produced using this substance. It is frequently employed in industries requiring an augmentation of viscosity, such as biofuels, pharmaceuticals, and food additives. Cellulose is biodegradable, meaning that living creatures may break it down into smaller components. Cellulose is an environmentally benign substance that can be recycled and biodegraded.

Rice fields are the most extensive among all cereal crops worldwide. Certain regions in Malaysia possess convenient availability of rice husk, a notable residue from agricultural activities. A significant amount of rice husks is generated as a byproduct in the rice milling process. The countries of China, India, Bangladesh, Indonesia, Vietnam, Thailand, Myanmar, the Philippines, Pakistan, and Brazil are the foremost rice producers on an international scale. It is worth noting that the top ten countries handle more than 84% of the total global rice production. The global variety of rice exceeds 50,000 distinct varieties (Team, 2023). Jackson's (1977) research demonstrated that rice husk consists of 33% cellulose, 26% hemicellulose, and 7% lignin. They used rice husk as the principal substrate, which holds significant promise for producing cellulose and nanocrystals.

The durian fruit is native to Southeast Asia. The geographic range of this species is documented in the Malay Peninsula, Indonesia, and Borneo (Subhadrabandhu et al., 1991). The durian is a tropical fruit tree that is classified under the order Malvales and the family Bombacaceae. The term "durian" is derived from the Malay word "duri," which means "thorn." The improper disposal of durian husks, especially in landfills, has led to environmental issues such as soil pollution and the transmission of diseases. Utilizing durian husk natural fiber can effectively tackle waste disposal problems and enhance

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agricultural waste utilization. The durian fruit has 40% edible flesh and 60% inedible husk. Usually, the outer covering of the durian fruit is discarded in landfills or burned, which raises environmental issues. Mohd Nordin et al. (2020) found that the durian husk consists of around 60.45% cellulose, 15.45% lignin, and 13.09% hemicellulose, similar to wood fibre.

The pineapple plant, scientifically known as *Ananas comosus (L.)* Merr (Bromeliaceae) can grow up to 1.5 meters tall in its native tropical environment. The plant is a biennial herbaceous species that produces a flavorful and substantial fruit. The fruit's pulp can vary in colour, ranging from nearly white to yellow, depending on the specific variety. The composition of pineapple peel, as outlined in a 1996 study conducted by Bardiya, Somayaji, and Khanna, which includes cellulose, hemicellulose, lignin, and pectin. The utilization of cellulose produced from pineapple peels is highly significant from both a practical and ecological standpoint.

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1.2 Problem statement

Microbeads are widely used in skincare, personal hygiene, and cosmetic goods. Microbeads are tiny, round particles composed of either polypropylene or polyethene. Most microbeads have a diameter that falls between 0.5 to 500 micrometres. As a result, these little entities can be challenging to filter out during the wastewater treatment process, which might result in their release into bodies of water such as rivers, lakes, or the ocean.

Microbeads are low-density and resistant to biodegradation, so they can accumulate in drainage systems and float on water. Introducing microparticles into aquatic habitats can induce pollution in rivers, lakes, and seas. These organisms can trick fish and birds into swallowing them by resembling fish eggs. Microbeads can absorb toxic compounds like polybrominated diphenyl ethers (PBDEs), making them dangerous. This increases their vulnerability to risk. Toxic microbeads can enter the bodies of humans and other animals when they consume fish. Polybrominated diphenyl ethers (PBDEs) have been linked to the occurrence of severe neurological, immunological, and reproductive abnormalities. The current investigation entails the extraction of cellulose from durian husk and pineapple peels to substitute plastic with microbeads.

Cellulose microbeads can alleviate the problem of microbead contamination. Cellulose is biodegradable and may be broken down by bacteria that naturally exist in the environment. This helps prevent cellulose from building up in streams and causing harm to animals. Utilizing agricultural waste for cellulose manufacturing has the potential to provide farmers with an extra income source while also addressing the problem of agricultural waste. This has the potential to benefit farmers while reducing the adverse

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environmental effects of agricultural waste. This research project can tackle the problem of microbead pollution and promote the idea of a circular economy by utilizing agricultural waste as a raw material for cellulose manufacturing.

1.3 Objectives

1. To extract cellulose from rice husk, durian husk, and pineapple peels by using alkaline pre-treatment and bleaching process.
2. To characterize the cellulose using SEM, FTIR, XRD and TGA.

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1.4 Scope of study

Utilizing waste items like rice husk, durian husk, and pineapple peels can help conserve valuable resources. Several strategies might be used instead of this one. To obtain cellulose, a chemical extraction process was utilized. Pre-treatment with alkaline solutions, as well as bleaching, are all part of this procedure. To better understand how to use each material's distinct properties, scientists study the cellulose content of rice husk, durian husk, and pineapple peels. The most cost-effective procedure involves doing an alkaline pre-treatment, and finally, bleaching.

1.5 Significance of study

The study of cellulose extraction is essential because of its wide range of applications, including its use in the production of microbeads. Cellulose microbeads are generally considered less environmentally harmful than plastic microbeads due to their lower susceptibility to decomposition. Utilizing alkali treatment and bleaching procedures shows promise for future research on extracting cellulose from durian husk and pineapple peels. This research represents a promising preliminary stage towards future inquiries.

The rationale for undertaking this research is pivotal. Firstly, it emphasizes the urgent requirement for efficient and enduring methods of extracting cellulose fibres, capable of generating diverse materials for prospective biomass renewable energy sources or manufacturing non-hazardous microbeads. Cellulose can absorb and release moisture, enabling them to maintain an optimal temperature. This attribute is particularly beneficial in the textile sector as it promotes airflow and regulates body temperature. Cellulose possess inherent biodegradability, leading to their inevitable disintegration and

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autonomous dissolution. Natural fibers are more environmentally advantageous than synthetic fibers due to their excellent biodegradability.

Cellulose is an inexhaustible resource that may be continuously created and collected since they are obtained from plants. Including cellulose in products promotes the utilization of sustainable resources while reducing dependence on non-renewable ones. Moreover, cellulose have been employed in tissue engineering and biomedicine. They function as crucial supports for the process of cell regeneration and growth.

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CHAPTER 2

LITERATURE REVIEW

2.1 Cellulose

Cellulose is a polysaccharide frequently present in plants' cell walls. The function of this component is to offer plants structural support and aid in maintaining their shape. As per the National Institutes of Health, cellulose is a crucial dietary fiber source that aids in promoting digestive health (NIH). Moreover, cellulose constitutes a substantial constituent of various paper and textile varieties, making it an indispensable material in industrial and commercial sectors. Cellulose is a vital component in the growth and development of plants, as well as in various human activities.

Cellulose is a crucial structural element within the primary cell wall of plants. Cellulose is a glucose polymer linked by β-1,4 linkage, which gives it a linear configuration (Singanusong et al., 2014). Cellulose has a chemical formula of C6H10O5, and its repeating units consist of hydroxyl groups. According to Ramamoorthy et al. (2015), the OH group in cellulose can form intermolecular and intramolecular hydrogen bonds, resulting in the hydrophilic nature of cellulose.

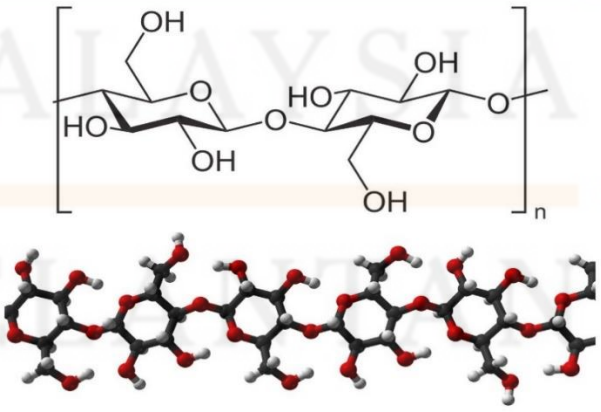

Figure 2.1: Chemical structure of cellulose

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Cellulose has garnered considerable attention for assorted reasons. This material's biodegradable nature and eco-friendliness render it an adequate substitute for synthetic materials. Renewable plant-based materials can serve as a cellulose source, promoting sustainability and waste reduction. This technology exhibits a wide range of applications across various industries, including but not limited to food, pharmaceuticals, textiles, and construction. In addition, progress in the field of nanotechnology has resulted in the creation of nanomaterials based on cellulose that possesses distinctive characteristics, thereby creating opportunities in the domains of electronics, energy storage, and biomedical engineering. In general, cellulose is a sustainable and adaptable material that aligns with increasing environmental awareness and presents opportunities for creativity.

According to Karataş and Arslan (2016), cellulose is the predominant structural constituent of the plant cell wall, accounting for approximately one-third of the total plant biomass present on the planet. The white powdery material is obtained by processing dried lignocellulosic masses using delignification and bleaching treatments. The extracted cellulose is a biopolymer composed of repeating β-D-anhydroglucopyranose monomeric units that are linearly linked by covalent (β-1,4) acetal linkages between the C1 and C4 of adjacent units. This is the chemical composition of cellulose. The macromolecule exhibits chemical activity due to three hydroxyl (-OH) groups on each monomer. The formation of inter- and intramolecular hydrogen bonds is observed in glycosidic chains in natural settings, as noted by Aravamudhan, Ramos, Nada, and Kumbar (2014). The distribution of highly packed, ordered arrangements and loosely arranged, unordered glycosidic chains within the polymeric matrix of cellulose results in its inherent crystallinity and amorphousness. The phenomenon of varying crystalline patterns, called 'allomorphism' in technical parlance, may exhibit differences across different sources. Allomorphism may

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undergo alterations during extraction or due to various physical and chemical treatments. The factors mentioned above impact the substance's characteristics (Kumar et al., 2018).

Considerable efforts have been devoted to the valorization of cellulose, which is the most easily extractable biomaterial. The product in question exhibits significant demand within the paper industry. According to Nsor-Atindana et al. (2017), the utilization of this substance has been limited to the textile and pharmaceutical sectors, as well as the production of regenerated fibers, films, packaging materials, and food ingredients. Cellulose consumption is unavoidable in any dietary practice, as plant-based constituents constitute a significant portion of most diets, wherein the cellulose is inherently present.

2.2 Sources of Cellulose

2.2.1 Rice Husk

Rice (Oryza sativa, L.) has the distinction of being the world's largest cereal crop. Rice husk is a substantial agricultural waste widely available in some Malaysian states. The rice milling business generates a substantial volume of rice husks while milling paddy from the fields. The top ten rice-producing countries, such as China, India, and Bangladesh, contribute over 84% of global production, with more than 50,000 distinct rice varieties worldwide (Team, 2023). This situation directly impacts the ecosystem and offers a considerable environmental danger, causing damage to the soil and the surrounding area where it was discharged. Rice husk contains 33% cellulose, 26% hemicellulose, and 7% lignin, according to Jackson (1977). As a result, using rice husk as a significant source for generating cellulose and nanocrystals appears viable. Derivatization of cellulose from rice husk for culinary applications could be a sustainable

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method of valorizing rice husk. Rice husk CMC has been created, and its potential use in filtering membranes and formulation additives has been suggested (Biswas, Kim, Selling, & Cheng, 2014).

The presence of cellulose is particularly beneficial for the extraction of cellulose. Furthermore, the fibrous structure of the rice husk, which comprises long, entangled strands, affects the extraction process and fiber quality. Pre-treatment is frequently required when extracting cellulose fiber from rice shells. Acidic and alkaline pre-treatment procedures are often used to break down the lignocellulosic structure and enhance cellulose fiber separation. However, remember that rice husk includes much silica, which might make extraction difficult. Silica particles can clog extraction and purification procedures, requiring extra steps to remove them.

One of the key benefits of using rice husk to extract cellulose is that it is a byproduct of the rice milling process. Because of its widespread use, it is a low-cost and easily accessible substance. Using rice husk in the cellulose extraction process improves sustainability by reusing agricultural waste and reducing environmental effects. Researchers can improve pre-treatment processes, evaluate the composition, and address silica concentration difficulties by defining rice husk for cellulose extraction. The extraction of rice husk cellulose is expected to become more efficient and of higher quality due to these activities, allowing for a broader range of applications.

Overall, research into rice husk as a source of cellulose extraction shows promise for long-term resource usage. By using its high cellulose content, understanding its physical features, and addressing pre-treatment requirements and issues, researchers may realize the potential of rice husk as a profitable and ecologically acceptable cellulose

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source.

2.2.2 Durian

The durian fruit is native to Southeast Asia. The geographic range of this species is documented in the Malay Peninsula, Indonesia, and Borneo (Subhadrabandhu et al., 1991). The durian is a tropical fruit tree that is classified under the order Malvales and the family Bombacaceae. The term "durian" is derived from the Malay word "duri" which means "thorn." The improper disposal of durian husks, especially in landfills, has led to environmental issues such as soil pollution and the transmission of diseases. Using durian husk natural fiber can effectively tackle waste disposal problems and enhance agricultural waste utilization. The durian fruit is composed of 40% flesh and 60% husk. Usually, the outer layer of the durian fruit is discarded in landfills or burned, which raises environmental issues. Mohd Nordin et al. (2020) found that the durian husk consists of about 60.45% cellulose, 15.45% lignin, and 13.09% hemicellulose, like wood fiber.

2.2.3 Pineapple Peel

Ananas comosus (L.) Merr (Bromeliaceae) is the botanical name for the pineapple plant, which can attain a maximum height of 1.5 meters in its indigenous tropical environment. This perennial plant flowers biennially and produces edible fruit. Depending on the type, the fruit's flesh can vary in colour, ranging from almost white to yellow. Bardiya, Somayaji, and Khanna (1996) identified cellulose, hemicellulose, lignin, and pectin as the main constituents of pineapple peel. Aside from being commonly consumed as fresh fruit or juice, pineapple is a versatile component that may be used in various cuisines.

Dai and Huang (2017a, b), Kaur et al. (2016), and Krishni and Hameed (2014)

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have reported that bromelain may be obtained from pineapple, making it a valuable component in the food processing industry. Approximately 35% of the total weight of a pineapple consists of its skin following the processing stage. Given its versatility and eco-friendliness, pineapple peel cellulose necessitates modification and utilization for various purposes. Hu et al. (2010) observed that the extensive disposal of pineapple peels could be economically burdensome and environmentally troublesome. Because pineapple peel can be obtained without incurring additional expenses for waste management, it is evident that it is a desirable source of biomass for industrial applications. The pineapple peel contains cellulose, which is advantageous. Additionally, the peel possesses numerous other uses.

2.3 Extraction method on cellulose

2.3.1 Conventional method

The conventional method for isolating cellulose from rice husk, durian husk, and pineapple peel involves an alkaline pretreatment and subsequent bleaching procedure, which is methodical. At first, the raw materials are gathered and carefully processed, which includes thorough cleaning and drying to remove any impurities and moisture. Afterwards, an alkaline pretreatment is utilized, commonly employing sodium hydroxide (NaOH) or potassium hydroxide (KOH) to degrade lignin and hemicellulose constituents, hence allowing the extraction of cellulose fibers, after the pretreatment, which aims to separate the cellulose fibers, the pulping phase, whether mechanical or chemical, occurs. To achieve purity and whiteness, a bleaching procedure is conducted utilizing substances such as chlorine dioxide (ClO2), hydrogen peroxide (H2O2), or oxygen (O2). The leftover chemicals and contaminants are effectively eliminated through meticulous

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washing and screening methods, followed by the drying of cellulose fibers to decrease their moisture content. Ultimately, these processed cellulose are utilized in sectors such as papermaking and textiles or as reinforcement in composite materials. Customizing the process parameters according to the unique plant material and desired fibre qualities is crucial. Additionally, incorporating environmental considerations is frequently done to improve sustainability.

2.4 Chemical Extraction Method

2.4.1 Alkaline Treatment

Alkali treatment frequently modifies natural-fiber composites. It effectively removes many undesirable compounds, including hemicellulose, lignin, oils, and wax, from the surface of the cell wall or fibers. This method is widely adopted because of its high efficiency and user-friendly nature. The alkali treatment process leads to the depolymerization of the original cellulose structure and the emergence of smaller crystallites. This treatment also causes the defibrillation of cellulose microfibrils on the surface (Abraham et al., 2011). Lignin removal facilitates molecular rearrangement and enhances cellulose crystallinity. The final products' mechanical properties have been enhanced due to these interlocking and bonding occurrences.

By strengthening the hydroxyl groups, sodium hydroxide (NaOH) as a solvent enhances the link between the fiber and the matrix. NaOH also affects the geometry of the fibers, making the surface rougher (Pickering et al., 2016). The initial stage of the study determined that alkalization enhances the crystalline structure of amorphous cellulose by eliminating the carbonyl group and disrupting the hydrogen bonds within the network structure of cellulose's hydroxyl group. Sodium hydroxide (NaOH) alkali

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treatment successfully removed carboxyl groups (which act as fatty acid tracers) and created a high cellulose content. In addition, the bonding between the fibers at the interface is greatly strengthened, improving the composites' mechanical properties, particularly in tensile and flexural strength (Khan et al., 2013). Johar et al. (2012) revealed that Alkali treatment efficiently decreases hemicellulose concentration. The hemicellulose concentration reduced from 33 wt% to 12 wt% following the treatment.

2.4.2 Acid Treatment

The acid pretreatment of cellulose fiber is a crucial stage in the extraction of top-notch cellulose, which is indispensable for various applications in material science, biotechnology, and the production of sustainable materials. This method entails subjecting untreated lignocellulosic materials to targeted acid treatment to eliminate lignin and hemicellulose, increasing cellulose concentration. The effectiveness of this approach in improving the quality and use of cellulose fibers is extensively documented.

Recent breakthroughs and research in this field demonstrate the efficacy of acid pretreatment in improving cellulose fiber for many applications. An example is a study by Nguyen et al. (2022) titled "Enhancing Cellulose Fiber Quality by Acid Pretreatment," published in the Journal of Sustainable Material Science. The study shows that acid pretreatment greatly enhances cellulose fibers' purity and structural integrity. This research highlights the significant contribution of the technology in creating fibers that have improved mechanical properties and heightened reactivity, expanding their suitability for a broader array of applications.

2.4.3 Bleaching

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Utilizing bleaching agents is crucial in the bleaching procedure, which seeks to eliminate pigmentation from substances. This is the subsequent stage in the cellulose pretreatment procedure. This technique is highly acclaimed for its capacity to efficiently remove natural dyes from fabrics without causing any damage to the fibers. Bleaching uses hydrogen peroxide, which causes oxidation, to eliminate colourants from their underlying materials permanently.

The choice of raw materials can significantly impact the overall efficacy of the bleaching process. Therefore, it is essential to consider aspects such as bleaching duration and solution concentration. The complete elimination of cementing components from the fiber necessitates using the bleaching method (Abraham et al., 2011).

According to Rayung et al. (2014), hydrogen peroxide can modify the colour of fibers. The breakdown of lignin and cellulose occurs when hydroxyl ions (OH-) react with light-absorbing chromophoric groups due to the presence of hydroxyl ions (OH-) generated by the dissociation of hydrogen peroxide in an alkaline setting. The approach modified the chemical composition of the fiber and removed lignin by disrupting the aromatic C=C bond of lignin (Khan et al., 2013). Separating fiber bundles into individual fibers following bleaching, as Cherian et al. (2010) discovered, results in the degradation of lignin. The hydroxyl, carboxyl, and carbonyl groups formed during degradation are beneficial in various stages of the cellulose purification process.

2.5 Cellulose Characteristics

2.5.1 Scanning Electron Microscopy (SEM) – Morphology Analysis

In the scanning electron microscope (SEM), signals are produced by focusing a

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beam of high-energy electrons onto the solid surface of the specimen. The call will disclose the sample's chemical composition, morphology, and crystallographic arrangement. Scanning electron microscopy (SEM) involves directing an electron beam onto the surface of an object and subsequently detecting the resulting electrons to generate a pixelated image. The image column's apex is where the electron beam is generated and focused. The emission of secondary electrons, Auger electrons, X-ray photons, backscattered electrons, and cathode luminescence is initiated by the interaction between the initial electron beam from the column and the surface.

SEM is used to evaluate the pretreatment results and monitor any structural changes in the sample. Scanning electron microscopy (SEM) can be used to visualize and eliminate impurities such as oils and wax present on the surface of a sample. The bundles contain visible fibrils (Cherian et al., 2010). Scanning electron microscopy (SEM) has numerous advantages, including obtaining detailed 3D and topographical images and collecting data using variousors. The assay is rapid, requiring less than five minutes for completion and minimal sample preparation.

The present scanning electron microscope (SEM) can generate digital data due to its state-of-the-art technology. In addition, SEM offers a deeper depth of field and better resolution. Scanning electron microscopy (SEM) has effectively been employed to detect the degradation of lignin, pectin, and hemicellulose after treatment. The typical fiber diameters were measured using SEM (Alemdar & Sain, 2008). Utilizing scanning electron microscopy (SEM) at different magnification levels can provide further insights into the characteristics of the surfaces of the sample (Manilal & Sony, 2011).

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2.5.2 Fourier Transform Infrared (FTIR) – Functional Group Determination

Fourier-transform infrared spectroscopy (FTIR) can acquire the absorption or emission spectra of a solid, liquid, or gas. An FTIR spectrometer collects high-resolution spectral data simultaneously across a wide spectral range. This is a substantial enhancement compared to the outcomes achieved using a dispersive spectrometer, which is limited to detecting intensity across a narrow range of wavelengths. The Fourier transform is a mathematical process that converts raw data into the natural spectrum, known as Fourier-transform infrared spectroscopy.

The FTIR technique evaluated the functional groups in plant components such as lignin, hemicellulose, and cellulose. The decreased intensity peaks or absence of specific peaks prove that the functional groups have changed after various treatments. The untreated fibers peak at 3443 cm-1, caused by the high absorption of intermolecular-linked hydroxyl groups. The peak at 1509 cm-1 is associated with the aromatic asymmetric stretching of lignin, whereas the peaks at 1435 cm-1 are related to the C-O-C links in lignin (Aprilia et al., 2015). The absence of the peak at 1435 cm-1 and 1509 cm-1 provides evidence for eliminating lignin (Saurabh et al., 2016). The lack of a peak at 1732 cm-1 indicates that the hemicellulose has been successfully eliminated.

A peak at 1025 cm-1 indicates a higher cellulose content and is ascribed to the C-O stretching vibration in cellulose. Additionally, the peak at 828 cm-1 corresponds to the features of the β-glycosidic bond (Saurabh et al., 2016). The signal observed at 2913 cm-1 corresponds to cellulose's aliphatic saturated C-H stretching vibration. The bands seen within the 1370-1390 cm-1 range are associated with cellulose's C-H deformation, as mentioned by Pelissari et al. in 2014.

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2.5.3 X-Ray Diffraction (XRD) - Crystallinity

X-ray diffraction analysis (XRD) is a widely used and ecologically friendly technology that accurately identifies a material's crystal structure, chemical composition, and physical characteristics. This method is based on the phenomenon of constructive interference, in which monochromatic X-rays interact with a crystalline substance. X-rays are generated when charged particles slow down, producing electromagnetic radiation with a shorter wavelength. X-ray diffraction (XRD) involves the interaction between nanomaterial samples and incident X-rays, leading to diffracted X-ray generation. Subsequently, the X-rays that have undergone diffraction are recognized, subjected to processing, and quantified. The resulting diffraction pattern, depicted as a graph illustrating the strength of diffracted rays at different angles, is unique for each material phase due to differences in chemistry and atomic arrangement.

X-ray diffraction (XRD) is crucial for assessing the crystallographic arrangement of cellulose. The study conducted by Yao et al. (2014) provides a comprehensive analysis of cellulose composition at the atomic level using the measurement of X-ray scattering. Furthermore, X-ray diffraction (XRD) can be employed to evaluate the degree of purity of a substance. Cellulose's thermal and mechanical properties are significantly influenced by its crystallinity, with a greater degree of crystalline orientation linked to improved mechanical characteristics (Dungani et al., 2014). Cellulose's crystalline structure is influenced by intramolecular and intermolecular hydrogen bonding, especially in the absence of hemicellulose (Li et al., 2015). The crystallinity index (CrI) indicates the amount of secondary molecular bonds and the level of compaction inside the crystalline areas (Andrade-Mahecha et al., 2015). A larger CrI signifies an augmented level of crystallinity, which impacts the ability to compress and absorb water (Gopinathan et al.,

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2017).

2.5.4 Thermal stability

One practical and cost-effective method for determining the chemical makeup of lignocellulosic biomass is thermogravimetric analysis (TGA). Compared to other methods, it offers a more functional and straightforward approach. If you want to know how much α-cellulose and hemicellulose are in anything, thermogravimetric analysis (TGA) is better than wet chemical methods. We utilized thermogravimetric analysis (TGA) to examine the biochemical makeup (namely lignin, cellulose, and hemicellulose) of pre-treated Avicel, beechwood, alkaline lignin, switchgrass, and maize stover. TGA's high-throughput nature shines when evaluating polymers like lignin, mainly when used online. Degradation and changes in polymer analytes due to the rise in lagging temperature harm this technique.

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CHAPTER 3

MATERIALS AND METHODS

3.1 Apparatus and Materials

The materials used were rice husk, durian husk, pineapple peels, sodium hydroxide (99%), sodium chlorite, acetic acid, sodium hydroxide, sodium hypochlorite (NaOCl, 4% W/V), oven, blender, round bottom flask (500mL), heating mantle, beaker (100, 500 and 1000 mL), Erlenmeyer flask, measuring cylinder (5 and 50 mL) and zipper bags.

3.2 Sample Preparation

The rice husk obtained from Sekinchan, Selangor. The presence of durian husk and pineapple peels will be detected in Kelantan. 2 kilograms of rice husk, 15 pieces of durian husk, and 2 pieces of pineapple peels were employed, each in their appropriate quantities. The samples were carefully washed with distilled water and thereafter subjected to a drying process in an oven for a duration of 24 hours at a temperature of 80°C. Subsequently, the material was pulverized into a fine powder and subsequently kept in a zippered container for future utilization.

3.3 Chemical Extraction

3.3.1 Delignification

A 250 mL beaker was used in a laboratory to blend 10 grams of rice husk powder with 100 mL of a 12% sodium hydroxide (NaOH) solution. The solution is heated to 80°C using a magnetic stirrer and maintained at this temperature for three hours. The solution

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was then filtered to separate the solid remains. The leftover component was extensively washed with distilled water until the pH level remained constant. It is critical to note that the same precise techniques will be used for both durian husk and pineapple peel, ensuring consistency and precision throughout the experimental process.

3.3.2 Alkaline Pre-treatment

This scientific process involves immersing rice husk, durian husk, and pineapple peel in a mixture and agitating them constantly for 6 hours. After the agitation step, the combination, including all three ingredients, will thoroughly filtrate to remove any remaining husks and peels. Afterwards, the gathered remnants, including rice husk, durian husk, and pineapple peel, will undergo an extended drying procedure to guarantee thorough dehydration.

Additionally, the fibrous peels of durian husk and pineapple peel, together with rice husk, will undergo treatment using a sodium hydroxide (NaOH) solution at a concentration of 0.1 M. This treatment will require constant agitation for 4 hours, ensuring comprehensive and uniform processing. After the treatment, every specimen will go through a thorough rinsing process using distilled water. This process will be carried out painstakingly until a neutral pH is reached, effectively removing any remaining traces of NaOH.

3.3.3 Acid Pre-treatment

This scientific methodology entails submerging rice husk, durian husk, and pineapple peel in a specific mixture and maintaining constant agitation for a duration of 6 hours. Following this phase of agitation, the mixture containing the three components

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is thoroughly filtered to remove any residual husks and peels. Subsequently, the extracted remnants, comprising rice husk, durian husk, and pineapple peel, are subjected to an extensive drying process to ensure complete dehydration.

Moreover, the fibrous content of the durian husk and pineapple peel, along with the rice husk, is treated with an acetic acid solution at a concentration of 0.1 M. This treatment involves continuous agitation over a period of 4 hours to achieve a uniform and comprehensive effect. Post-treatment, each sample is meticulously rinsed using distilled water. This rinsing process is conducted diligently until a neutral pH level is attained, ensuring the complete removal of any residual acetic acid. Our research protocol is designed to prioritize precision and reliability, adhering to a structured and systematic approach.

3.3.4 Bleaching Process

Building upon the previously outlined approach utilizing rice husk, durian husk, and pineapple peel, the bleaching process was commenced following the alkali pre-treatment step. The method was done by injecting a buffer solution of acetic acid, aqueous chlorite (1.7 wt%), and distilled water. The materials, such as rice husk, durian husk, and pineapple peel, were treated with bleaching conditions by refluxing them in a temperature ranging from 100 to 130℃ for 4 hours.

After the thermal treatment, the mixture was cooled to ambient temperature and underwent a meticulous filtration process using abundant distilled water. It is important to emphasize that the bleaching process was repeated four times for all materials examined in the study. Implementing this methodical technique guarantees that

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the bleaching process of rice husk, durian husk, and pineapple peel is carried out uniformly and regulated, enhancing the dependability and precision of the research results.

3.4 Characterization of The Extracted Cellulose

3.4.1 Functional group analysis

The chemical structure of chemical treated cellulose were analyzed by FTIR in the range of 400-4000 cm^{-1} with the resolution of 4 cm^{-1}.

3.4.2 Morphology analysis

The morphology of chemical treated cellulose were analyzed by SEM. The cellulose was put on a tape on aluminum for analysis.

3.4.3 Crystallinity Determination

Crystallinity of samples were determined by X-ray Diffraction (XRD) diffractometer. The scattered radiations were detected in the range 2θ range of 10° to 90° at speed of 0.04°/min (Gopinathan et al., 2017). The degree of crystallinity was determined by crystallinity index (CrI) using Equation 3.2, where $I002$ is the maximum intensity for the crystalline portion in samples (i.e., cellulose)at about 2θ= 22 º and Iam is the intensity attributed to the amorphous portion of samples(i.e., hemicellulose and lignin) at 2θ= 18 º.

$$CrI = [(I_{002} - I_{am}) / I_{002}] \times 100$$

3.4.4 Thermal analysis

Thermal stability of celluloses was determined by Thermalgravimetric analysis

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(TGA) measurements. Thermal analysis was carried out for chemical treated cellulose. Approximately 7 mgof sample was transferred into the alumina crucible with a pinhole and followed by heating from room temperature to 550 ºC at a heating rate of 10 K min^{-1}. All measurements were performed under a nitrogen atmosphere with a gas flow rate of 40 mL/minute.

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CHAPTER 4

RESULTS AND DISCUSSION

4.1 Physical appearance of Cellulose

4.1.1 Rice Husk (RH 1)

The rice husk turned an intense brown colour when immersed in sodium hydroxide. The alkali treatment with 20% sodium hypochlorite transformed it into a golden-brown colour, as seen in Figure 4.1. Khawas and Deka (2016) state that sodium hypochlorite dissolves and hydrolyzes pectin, starch, and hemicellulose.

Before	After
	

Figure 4.1: The rice husk changed colour from intense brown to dark goldish brown because of alkali treatment.

4.1.2 Durian Husk 1 (DH 1)

The durian husk 1 turned greenish red colour when immersed in sodium carbonate. The alkali treatment with 20% acetic acid transformed it into a transparent with goldish cellulose residue, as seen in Figure 4.2.

Before	After
	

Figure 4.2: The DH 1 changed colour greenish red to transparent because of acid treatment.

4.1.3 Durian Husk 2 (DH 2)

The rice husks turned an intense black reddish colour when immersed in sodium hypochlorite. The alkali treatment with 20% sodium hypochlorite transformed it into a transparent goldish residue, as seen in Figure 4.3.

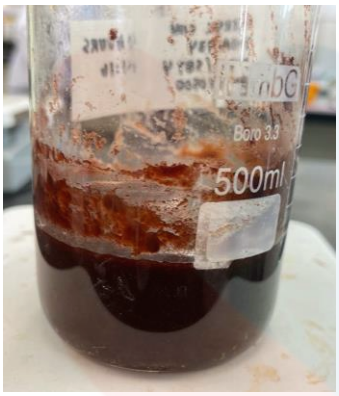
Before	After
	

Figure 4.3: The DH 2 changed colour black reddish to light yellowish gold because of alkali treatment.

4.1.4 Pineapple Peel 1 (PP 1)

The pineapple peel turned greenish colour when immersed in sodium hydroxide. The alkali treatment with 20% sodium hypochlorite transformed it into a transparent white residue as seen in Figure 4.4

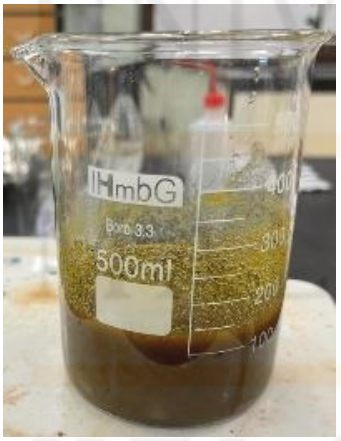
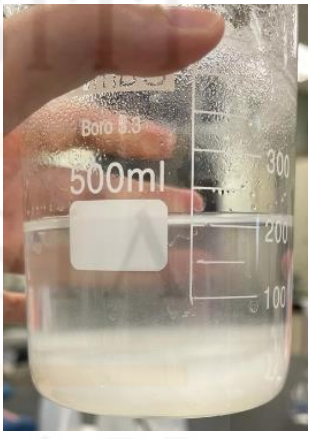
Before	After
	

Figure 4.4: The PP 1 changed colour greenish to clear transparent because of alkali treatment.

4.1.5 Pineapple Peel 2 (PP 2)

The pineapple peel 2 turned greenish colour when immersed in sodium hydroxide. The acid treatment with 20% acetic acid transformed it into a transparent white residue as seen in Figure 4.5.

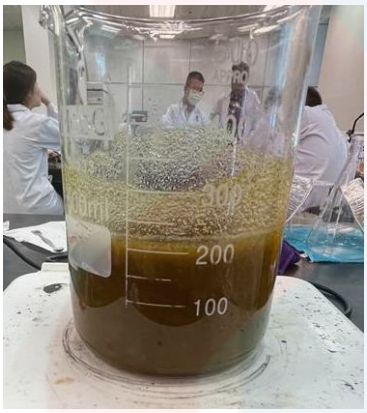
Before	After
	

Figure 4.5: The PP 2 changed colour greenish to cloudy transparent because of acid treatment.

4.2 Chemical composition of Rice Husk, Durian Peel and Pineapple Peel

4.2.1 Rice Husk

Rice is not just a staple food serving more than half of the global population, but it's also a cornerstone in agricultural practices across all six continents: Asia, Africa, Australia, Europe, North America, and South America. The annual global rice yield is a staggering 600 million metric tons. This remarkable production, as noted by Riaz et al. (2017), comes with a significant byproduct - rice husk. Annually, the rice industry generates about 120 million tons of solid waste, of which rice husk constitutes an impressive 20%. This husk is not mere waste; it is a complex composition of cellulose (50%), lignin (25-30%), and silica (15-20%), with a moisture content that typically

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ranges between 10% to 15%, as detailed by Korotkova et al. (2016).

The fascinating composition of rice husk varies depending on environmental conditions. Cellulose, a polysaccharide made up of β-linked D-glucose molecules, can vary from several hundred to thousands in number. Lignin, on the other hand, is a complex polymer of aromatic alcohols, specifically monolignols. It plays an integral role in the makeup of secondary cell walls in plants and certain algae types. The presence of diverse functional groups such as carbonyl, amide, amino, phenolic, carboxyl, hydroxyl, ester, and sulfhydryl in lignin allows it to form various complexes.

In the world of biomass, lignocellulose stands out with its dry weight comprising 56-72% of fermentable carbohydrates, including cellulose and hemicellulose. The process of alkaline processing, which predominantly uses sodium hydroxide (NaOH), calcium hydroxide (Ca(OH)2), ammonia solution (NH3·H2O), and sodium carbonate (Na2CO3), proves especially advantageous for agricultural waste and other biomasses with low lignin content. This method simplifies the hydrolysis of O-methyl ester groups into galactic acid groups and facilitates ion exchange with metal ions.

The outcomes of this alkaline processing on rice husks are multifaceted. It leads to the extraction of lignin, partial degradation of hemicelluloses, alteration in the crystalline structure of cellulose, and an expansion in cellulose. Moreover, this process fosters the development of a more porous structure, reduced cellulose polymerization, and an increase in interior surface area, making it a topic of great interest in both agricultural and industrial sectors.

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4.2.2 Durian Husk

One potential substitute for wood-based cellulose pulp is the utilization of durian husk leftovers. An economical and environmentally friendly approach to maximizing the value of durian fruit is using its peel to make cellulose. As an agricultural waste, durian husks have great potential as a fiber resource. How easy it is to get to be a factor here - From 2015 to 2017, the production of durian fruit in Sumatera Utara increased, according to Statistics Indonesia, from 65,529 tons to 74,811 tons. This amount will rise consistently over the next twenty years to meet market demand. Presently, the annual number is close to 90,000 tons. Garbage output is proportional to the growth in production statistics. Durian waste has two parts: the husk, which makes up 70-85% of the garbage, and the seed, which makes up 20-25% of the junk. The studies above show that durian has a lot of organic waste (Silviana & Subagio, 2019; Penjumras et al., 2014; Foo & Hameed, 2011). The chemical composition of durian reveals that it consists of cellulose (60.45%), hemicellulose (13.9%), and lignin (15.45%) (Rachtanapun et al., 2012).

4.2.3 Pineapple Peel

Pineapples, native to tropical America, have thrived in Hawaii, where they held a dominant global market in 1947. Hawaii dominated the global market for canned pineapple production, accounting for an impressive 85% of the 12 1/4 million cases produced that year. It emerged as the main centre for pineapple farming and export.. The Food and Agriculture Organization's (FAO) report, as reported by Hadidi et al. in 2020, highlights the significant worldwide influence of this fruit. The research reveals a

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remarkable production of almost 26 million metric tons in 2019 alone. Pineapples have a wide range of uses beyond being eaten fresh. Wu and Shiau (2015) and Braga et al. (2020) have observed that they are essential in many goods, such as juices, jams, pulp, and syrup.

Aruna's 2019 research provides an intriguing revelation regarding the sector, indicating that around 20% of pineapple fruit is expressly designated for juice manufacturing. Nevertheless, this procedure has its disadvantages. Dai and Huang (2017) and Kumar et al. (2021) have highlighted that approximately 35% of the fruit's overall weight is abandoned after it undergoes processing. As Ketnawa et al. (2012) noted, this creates a substantial issue in managing trash, as only a small portion is utilized for animal feed or fertilizer. According to Gnanasekaran et al. (2021), a significant portion of this garbage is frequently disposed of by incineration, which leads to environmental problems such as the release of smoke and haze, causing air pollution.

Given these considerations, the pineapple peel, which contains a lot of cellulose, stands out as a promising possibility. Banerjee et al. (2018) emphasized the diversity in the makeup of cellulose, hemicellulose, lignin, and ash in pineapple peels, which is affected by factors such as season, variety, maturity, and growing region. The research sample comprised 24.1% cellulose, 29.3% hemicellulose, 6.3% lignin, and 5.0% ash. This composition has prompted numerous investigations into novel uses of these natural fibers, which consist primarily of carbohydrate polymers such as cellulose and hemicellulose and aromatic polymers like lignin and ashes. The prospective applications are extensive and diverse, encompassing the manufacturing of bioethanol, the synthesis of hydrogels, uses in plasticization, adsorption processes, the generation of nitrocellulose, and additional utilization as a plasticizer.

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Component (%)	Current Study	(Ban-Koffi and Han 1990)	(Casabar et al.2020)	(Pardo et. al 2014)	(Rani and Nand 2004)
Cellulose	24.15±1.64	14.00	20.90	40.50	11.20
Hemicellulose	29.39±2.13	20.20	31.80	26.60	7.00
Lignin	6.35±0.28	1.50	10.40	10.10	11.20
Ash	5.05±0.10	0.60	9.90	1.50	3.80

Table 4.1: Chemical composition of fresh pineapple peel

4.3 Characterization of Cellulose from RH, DH and PP

4.3.1 Fourier Transform Infrared (FTIR) – Functional Group Determination

The prepared sample were analyzed by FTIR at wavenumber region of 400-4000 cm^{-1} to determine the chemical structure. The aim of using FTIR in this study is to determine the lignin and hemicellulose were removed by chemical to obtain cellulose. The extracted cellulose present vibration bands of components corresponding to lignin, hemicellulose, and cellulose. The vibration is summarized in Table 4.3. For all sample, a broad absorption of band at 3000-3650cm^{-1} region that attributed to –OH groups which reflected the hydrophilicity of rice husk, durian husk and pineapple peel. The peak at 3328.69 cm^{-1}, 3335.85 cm^{-1}, 3334.08 cm^{-1}, 3330.95 cm^{-1} and 3331.72 cm^{-1} were observed in Figure 4.3 for these five samples which were RH1, DH1, DH 2, PP 1, and PP 2.

Two significant absorbance areas are commonly found in the spectra: one ranging from 1100.00 cm^{-1} to 1200 cm^{-1} and the other from 2850 to 2950 cm^{-1}. Consistently, the same trend is observed across all samples. Specifically, there are no marked changes in the presence or absence of bands. However, there is a noticeable enhancement in the

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intensity of the polysaccharide group (3000-3500 cm^{-1}). The primary spectral peaks were observed at 3330, 2900, 1730, 1640, 1512, 1200–1300, and 894 cm^{-1}.

The bands at 3300 cm−1 and 2900 cm-1 represent the hydroxyl groups (inter, intra, and accessible OH) (Dai and Fan 2011; Ornaghi et al. 2014) and the stretching vibration of methyl and methylene.

The chemical structure of the prepared samples was thoroughly examined in this research, utilizing Fourier-transform infrared spectroscopy (FTIR) in the wavenumber range of 400-4000 cm^{-1}. The main aim of utilizing FTIR in this study was to determine the successful removal of lignin and hemicellulose through chemical treatments to isolate cellulose. The cellulose samples obtained exhibited different vibration spectra corresponding to the significant constituents: lignin, hemicellulose, and cellulose. The vibration properties are thoroughly consolidated in Table 4.3.

An important finding in all the samples, including rice husk, durian husk, and pineapple peel, is the wide absorption range seen in the 3000-3650 cm^{-1} region. The presence of -OH groups in this band indicates the hydrophilic nature of these compounds. The highest values, specifically at 3328.69 cm^{-1}, 3335.85 cm^{-1}, 3334.08 cm^{-1}, 3330.95 cm^{-1}, and 3331.72 cm^{-1}, were observed for the five distinct samples identified as RH1, DH1, DH2, PP1, and PP2, as shown in Figure 4.3.

The FTIR spectra exhibited two prominent regions of absorption in these materials. The first range extends from 1100.00 cm^{-1} to 1200 cm^{-1}, whereas the second covers 2850 to 2950 cm^{-1}. The observed pattern remained constant in all samples, showing no significant alterations in the presence or absence of bands.

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The bands seen at 3300 cm^{-1} and 2900 cm^{-1} hold great significance. The hydroxyl groups, including intermolecular, intramolecular, and accessible OH groups (as mentioned in Dai and Fan, 2011 and Ornaghi et al., 2014), are represented. The stretching vibration of methyl and methylene groups is also accounted for. The detailed examination of the FTIR spectra provides a deep understanding of the chemical composition of these biomass samples, which opens possibilities for their future utilization in other domains.

Group frequency wavenumber, cm^{-1}	**Origin**	**Assignment**
800-950	C-H	C-H deformation vibration in cellulose
1000-1100	C-O	C-O stretching vibration in cellulose
1100-1200	C-O-C	C-O-C asymmetrical stretching in cellulose
1250-1290	C-O	C-O stretching of acetyl in hemicellulose
1370-1390	C-H	C-H symmetric deformation cellulose
1420-1440	CH_2	CH_2 asymmetric stretching in lignin
1500-1700	C=C	Aromatic C=C bonding in lignin
1635-1640	O-H	O-H of water absorbed from cellulose
1718	C=O	C=O stretching of carboxylic acid
1744	C=O	C=O stretching of carboxyl ester
2850-2950	C-H	C-H stretching in cellulose-rich materials
3000-3650	-OH	Free and hydrogen bonded OH stretching

(Sources: Obi Reddy *et al*., 2012)

Table 4.2: The vibration bands of components of lignin, hemicellulose, and cellulose.

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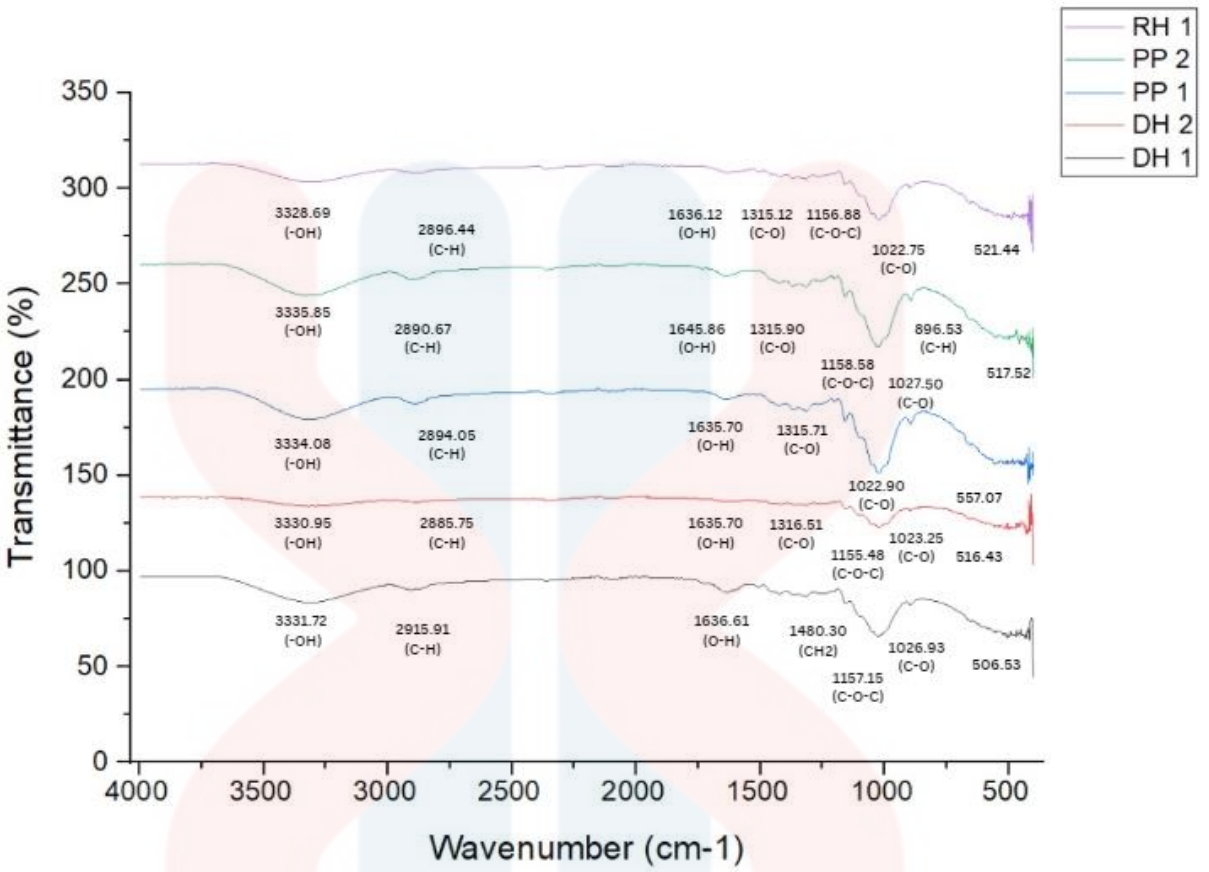

Figure 4.6: FTIR spectra of Extracted Cellulose from Risk Husk, Durian Husk, and Pineapple Peel.

Based on Figure 4.6, it is evident that sodium hydroxide effectively eliminates lignin from the sample, as there are no peaks observed around 1420-1440 cm^{-1}, which are characteristic of CH_2 asymmetric stretching in lignin and waxes, except DH1. The aromatic rings and carboxyl groups present in the polyphenolic structure of lignin are eliminated (Tibolla et al., 2014).

The isolated cellulose was deemed impure due to the presence of a peak in the range of 1500 cm^{-1} to 1700 cm^{-1}, which indicates aromatic C=C bonding in lignin. Furthermore, the absence of a signal in the 1250-1290 cm^{-1} area suggests that the sample does not include hemicellulose. The data shows that hemicellulose is eliminated by a solution of 20% sodium hydroxide.

The peak observed between 2850 cm^{-1} and 2950 cm^{-1} corresponds to the stretching vibration of aliphatic saturated C-H bonds in cellulose. The bands seen in the 1370-1390 cm^{-1} range are associated with cellulose's C-H deformation, as stated by Pelissari et al. in 2014. In addition, the cellulose isolated from RH, DH, and PP exhibits a peak within the range of 1635 cm^{-1} to 1640 cm^{-1}, ascribed to the presence of O-H bonds from water absorbed by cellulose.

Sample	**Compound (%)**			
	Cellophane	**Index No.**	**Cellulose**	**Index No**
RH 1	72.45	40	64.97	4
DH 1	76.32	40	71.59	4
DH 2	72.24	40	64.12	4
PP 1	74.97	40	69.43	4
PP 2	75.27	40	69.09	4

Table 4.3: The compound percentage of cellophane and cellulose in every sample.

In Fourier-Transform Infrared (FTIR) spectroscopy, "compound" refers to a chemical substance created by the specific bonding of atoms from different elements. These substances possess discrete molecular structures, leading to characteristic energy levels. FTIR analysis utilizes the interactions between infrared light and energy, resulting in absorption at specific wavelengths that serve as a unique molecule identifier. The spectroscopic approach is highly beneficial for chemical identification since it may effectively uncover a substance's particular types of bonds and functional groups.

Cellulose and its derivative, cellophane, have an essential link. Cellulose, a

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complex chemical compound made up of interconnected glucose molecules, provides structural integrity to the cell walls of plants. On the other hand, cellophane is highly valued for its clearness and ability to withstand different substances. The remarkable transparency of cellophane enables the transmission of light, making it an excellent choice for packaging due to its low permeability. The production methods for these materials vary, as cellulose is obtained from plants, whereas cellophane is synthesized using a solvent-based technique.

Cellulose has a wide range of applications across several industries, but cellophane is mainly used for packaging due to its distinctive combination of transparency and barrier characteristics. The differentiation in materials makes cellophane a favored option for products such as confectionery wrappers and cigarette packaging.

Each sample shows a very constant pattern in the percentage composition of cellophane and cellulose, according to the Fourier-Transform Infrared (FTIR) examination. The results of the FTIR analysis show that the cellophane percentage present in the samples varies between 72.45% and 76.32%. On the other hand, the cellulose percentage in these samples ranges from 64.97% to 71.59%, which is a little lower. (Refer to Table 4.3)

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4.3.2 Scanning Electron Microscopy (SEM) – Morphology Analysis

Scanning Electron Microscopy (SEM) is a crucial instrument in materials science and other scientific fields, providing a detailed examination of solid specimens. This technique utilizes a concentrated beam of high-energy electrons directed at the surface of the specimen. The complex interaction between these electrons and the sample's surface reveals valuable insights into its chemical composition, morphological characteristics, and crystallographic arrangement. The strength of SEM resides in its capacity to generate high-resolution images by detecting and collecting the emitted electrons from the sample, enabling a thorough investigation and characterization of the item being studied.

This method is essential for conducting pre-treatment evaluations and tracking alterations in the sample structure. The utility of SEM is further enhanced by its capability to detect and assist in removing surface impurities such as oils and waxes. Cherian et al. (2010) thoroughly documented the presence of visible fibrils in the observed specimens.

The SEM's versatility is demonstrated in examining cellulose, as evidenced by the intricate SEM images in Figures 4.7 to 4.11. The photos, especially in the study conducted by Mandal & Chakrabarty (2011), highlight the complex structure of cellulose that have been isolated. The micrographs of the durian peel that has not been treated reveal several non-cellulosic elements, such as pectin, lignin, and hemicellulose, distributed on the surface. These elements contribute to a greater diameter compared to cellulose. The conversion of these constituents becomes apparent after removing lignin and treatment with alkali.

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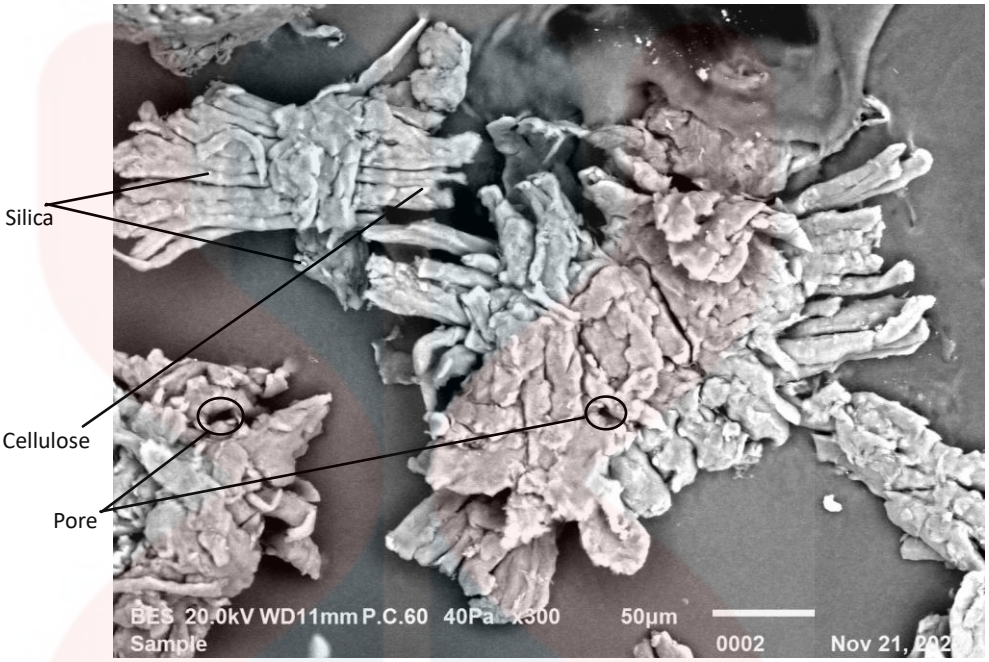
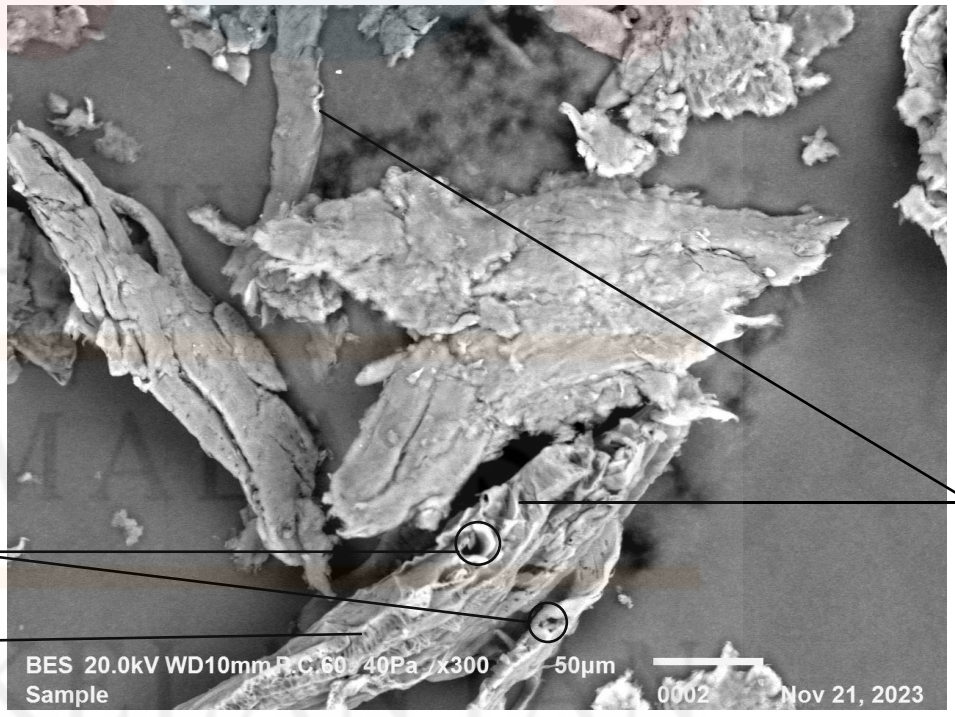
Sample	SEM image
RH 1 (4.7)	
DH 1 (4.8)	

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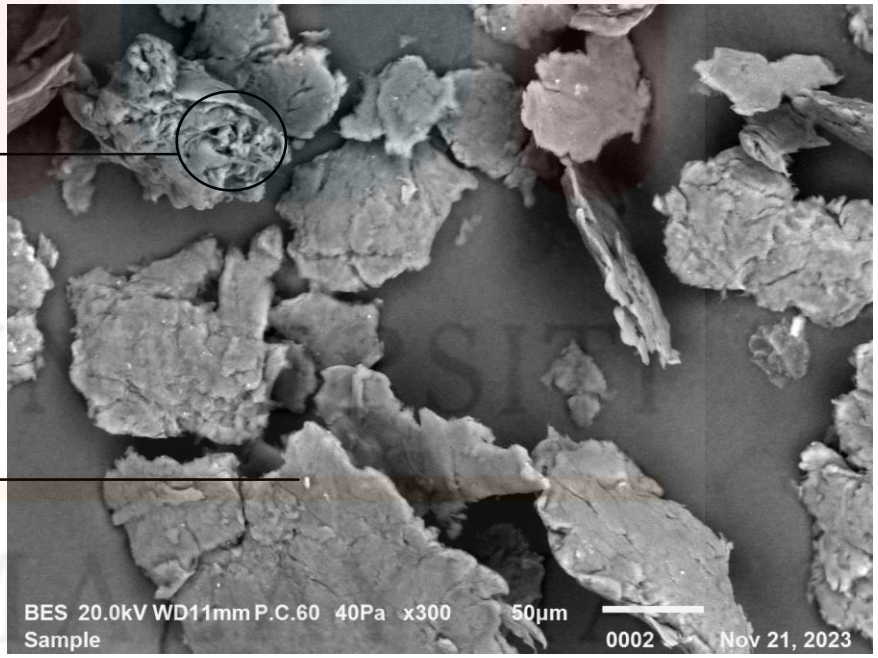
DH 2 (4.9)	
PP 1 (4.10)	

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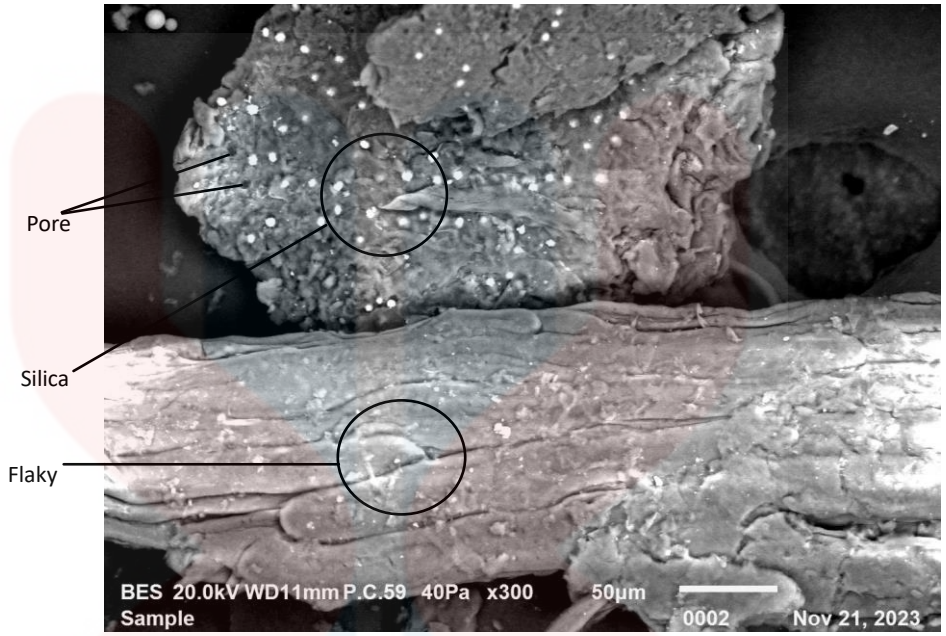
PP 2 (4.11)	

Figure 4.7, 4.8, 4.9, 4.10 and 4.11: SEM images of treated RH 1, DH 1, DH2, PP 1 and PP 2 in the magnification of 300x.

4.3.3 X-ray Diffraction

Cellulose, an essential biopolymer found in the complex cell walls of plants, has a structural intricacy marked by the simultaneous presence of crystalline and amorphous regions. In X-ray diffraction (XRD) investigation, the presence of well-defined diffraction peaks at specific angles indicates a material's crystalline properties. The XRD analysis, shown in Figure 4.12, reveals a distinct and precisely defined peak at an angle of $2\theta = 22°$, which corresponds closely to the results given by Khawas and Deka (2016). This observation confirms the presence of highly organized crystalline formations within the celluloses being studied, revealing the extremely ordered character of these regions.

It is essential to mention an apparent lack of a noticeable highest point at $2\theta = 18°$

in all analyzed samples. According to Gopinathan et al. (2017), this vital feature demonstrates the successful removal of lignin and hemicellulose, two key components contributing to cellulose's non-crystalline parts. The careful removal procedure is essential in cellulose processing since it consistently increases the purity and crystallinity of the material, resulting in improved characteristics and usefulness.

Exploring the field of quantitative evaluation, the crystalline index, as explained by Han et al. (2013), is a crucial and essential instrument. In this context, a higher value of the crystalline index indicates that the cellulose sample has a more precisely arranged, dense, and crystalline structure, suggesting a more significant structural organization. On the other hand, a reduced crystalline index indicates a cellulose sample with a less organized structure with more amorphous sections. This measure serves as a valuable tool for understanding the intricate relationship between the structural organization of cellulose and its performance characteristics.

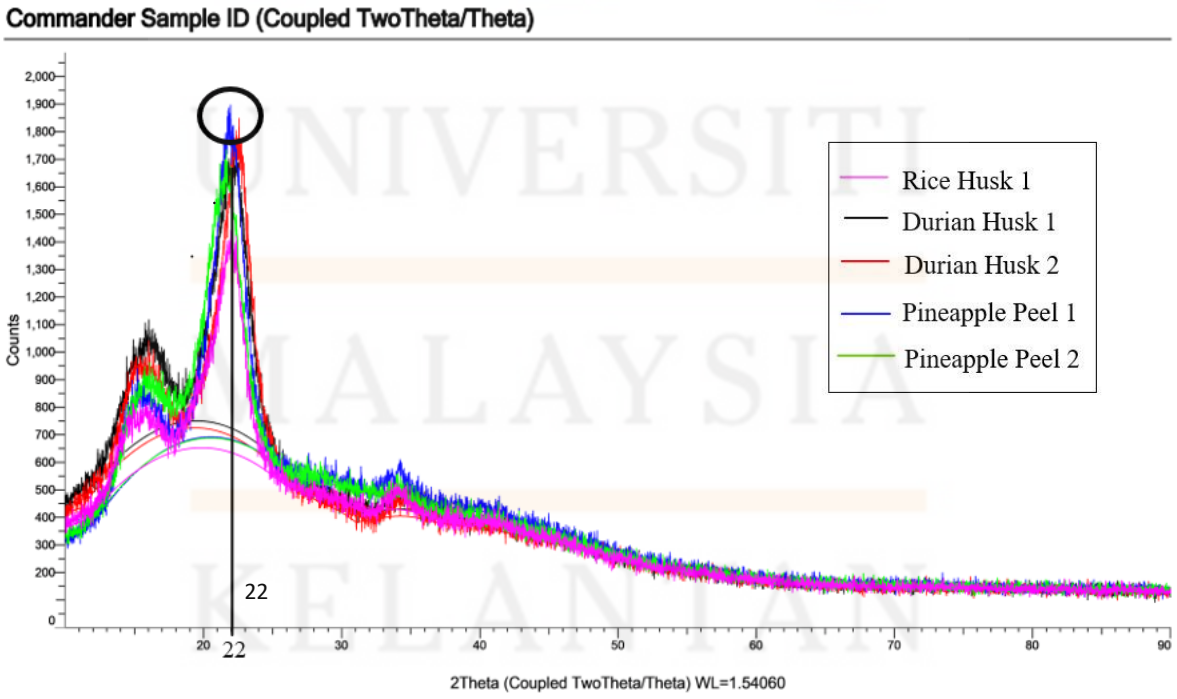


Figure 4.12: XRD diffraction pattern of cellulose from RH, DH, and PP.

(The circle part indicates the highest peak among all the samples which is PP 1)

Sample	Crystallinity	Amorphous
RH 1	44.60%	55.40%
DH 1	45.20%	54.80%
DH 2	34.20%	65.80%
PP 1	36.10%	63.90%
PP 2	34.30%	65.7%

Table 4.4: Crystallinity and amorphous of cellulose of each sample.

Pelissari et al. (2014) highlighted an essential part of cellulose's behavior by stressing how mechanical treatment dramatically affects the degree of crystallinity in cellulose. Selective removal of the amorphous parts of celluloses by automated means can improve the material's crystalline nature. Among the five cellulose samples we analyzed, we found a significant range in crystallinity, with DH 1 having the highest index at 45.20% and DH 2 having the lowest at 34.20%. Compared to DH 2, DH 1 has a more ordered and compact structure, highlighting the considerable influence of processing procedures on cellulose crystallinity.

There are far-reaching ramifications for such variations in cellulose crystallinity. High crystallinity cellulose is a good barrier material because of its inter-fibrillar solid connections and ability to create a dense network. Paper and paperboard are two products that greatly benefit from this quality. High-crystalline cellulose, for example, has a tremendous reinforcing effect on paper materials owing to its better fiber-fiber bond

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strength. Paperboards, grease-proof papers, improved retention, and dry and wet strength result from using high crystallinity cellulose as a wet-end addition.

In addition, crystallinity has a significant impact on the mechanical properties of cellulose. Proof of concept for the efficient use of high crystalline cellulose as a plastic reinforcement came from Khalil et al. (2014). The strength and stiffness of films made from high crystalline cellulose are exceptional, with strengths exceeding 200 MPa and stiffness reaching about 20 GPa, respectively. These features make cellulose a more versatile material and apply to many fields, such as biocompatible polymers, packaging, and materials research.

4.3.4 Thermal stability analysis

Using thermogravimetric analysis, the thermal stability of chemically treated cellulose is determined over a temperature range of 30 °C to 550 °C. The left limit is the temperature at which one begins to lose weight. The correct limit is the temperature at which weight loss concludes. An inflexion point is a directional alteration in the curve.

Due to initial thermal decomposition and moisture evaporation, the curve indicates that initial weight loss occurs between 38 and 120 degrees Celsius. Water retention occurs in cellulose because of its hydrophobic nature. Under a temperature of 200 °C, hemicellulose and lignin degrade. The thermal depolymerization of hemicellulose and cleavage of glycosidic linkages in cellulose occurs within the temperature range of 268-300 °C (Li et al., 2015). The wide range of temperatures from 270 °C to 400 °C indicates cellulose degradation (Khawas & Deka, 2016).

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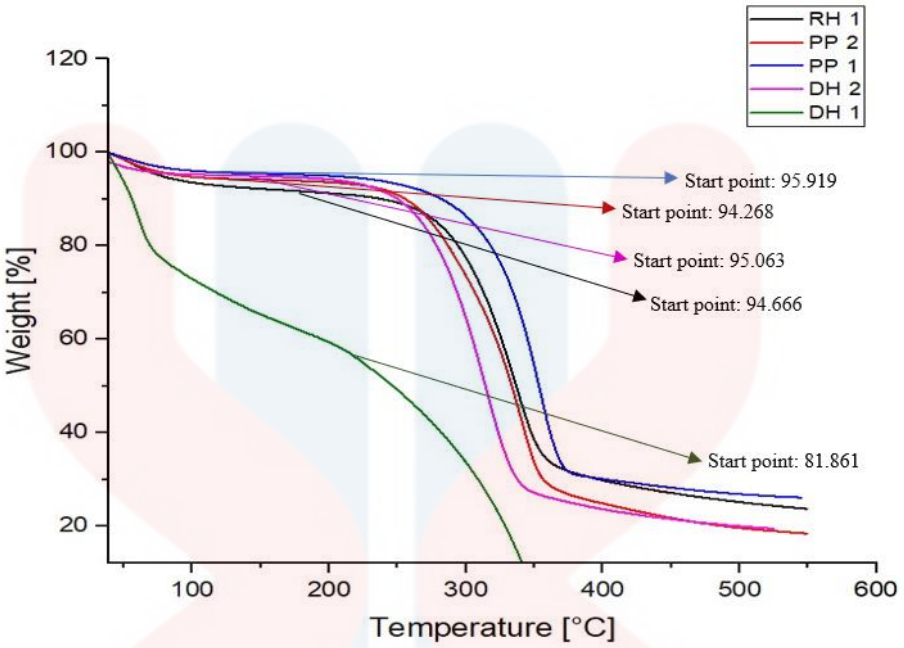

Figure 4.13: Thermal stability of the treated celluloses.

Sample	Starting Point (Y-axis)	Second Point (Y-axis)	Weight Loss 1st Quarter (%)	Second Point (Y-axis)	Last Point (Y-axis)	Weight Lost 2nd Quarter (%)
RH 1	94.666	90.479	4.187	90.479	32.964	57.515
DH 1	81.861	59.246	22.615	59.246	32.445	26.801
DH 2	95.063	94.177	0.886	94.177	27.372	66.805
PP 1	95.919	94.788	1.131	94.788	32.323	62.465
PP 2	94.268	92.251	2.017	92.251	28.900	63.351

Table 4.5: The weight loss percentage of each sample in TGA analysis.

Table 4.5 displays the weight loss % of each sample in the TGA study. RH experienced a weight reduction of 4.187% in the first quarter and 32.964% in the second quarter. DH 1 experienced a weight loss of 22.615% in the first quarter and 26.801% in

the second quarter. DH2 experienced a slight initial decrease in weight of 0.886% during the first quarter, followed by a substantial weight loss of 66.805% during the second quarter. PP1 experienced a weight loss of 1.131% in the first quarter and a weight loss of 62.465% in the second quarter. PP2 experienced a weight reduction of 2.017% in the first quarter and a weight loss of 63.351% in the second quarter.

TGA analysis yields significant data regarding the mass reduction of various samples. Notably, RH had the most crucial weight loss during the second quarter, whereas DH2 had the most minor weight loss during the first quarter but saw an effective weight loss during the second quarter. Both PP1 and PP2 encountered substantial weight reductions during the second quarter. According to the outcome shown in Figure 4.5, RH, DH1, DH2, PP1, and PP2 exhibit varying weight loss percentages in Q1 and Q2. In the first quarter of weight loss, DH1 demonstrates a significant loss of 22.615%, whereas the other categories show much lower losses: 4.187% for RH, 0.886% for DH2, 1.131% for PP1, and 2.017% for PP2. In weight reduction Q2, DH 1 stands out as the lowest at 26.801%, followed by RH at 57.515%, DH2 at 66.805%, PP1 at 62.465%, and PP2 at 63.351%. Upon analysis, it is evident that there is minimal disparity between the DH and PP samples when comparing the effects of different pretreatments. However, an exception can be observed in the case of DH 2.

The TGA analysis emphasizes the diverse weight loss patterns observed in different samples. RH demonstrates exceptional performance in terms of weight reduction during the second quarter, suggesting a potentially more unstable composition. Conversely, DH2 may have initially seen a minor decrease in weight during the first quarter but compensated for it with a significant decline in the second quarter.

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Furthermore, PP1 and PP2 exhibited substantial reductions in weight during the second quarter, indicating alterations in their compositions as well.

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CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The study underscores the significance of cellulose extraction for various applications, particularly in the production of environmentally friendly microbeads. Alkali treatment and bleaching methods show promise in extracting cellulose from rice husk, durian husk and pineapple peels, marking a promising step forward in research. Cellulose fibers offer diverse benefits, including moisture absorption, biodegradability, and sustainability, making them ideal for applications in renewable energy, textiles, and biomedical fields and more.

In this research utilized FTIR spectroscopy to assess the efficacy of sodium hydroxide in purifying cellulose and examined their composition and properties. Sodium hydroxide effectively removed lignin and hemicellulose from the fibers, but impurities remained due to aromatic C=C bonds in lignin. Cellulose exhibited characteristic peaks in the FTIR spectra, including aliphatic C-H stretching and O-H bonding. Cellulose and cellophane, derived from cellulose, have distinct applications, with cellophane prized for its transparency and barrier properties in packaging. The study found consistent percentages of cellulose and cellophane in the samples, with cellophane ranging from 72.45% to 76.32% and cellulose from 64.97% to 71.59%. These findings have significant implications for industries reliant on cellulose-based materials.

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SEM is crucial for evaluating pre-treatment operations, monitoring changes in sample structure, and detecting surface contaminants such oils and waxes. Untreated samples images display non-cellulosic components such pectin, lignin, and hemicellulose on the surface, which contribute to a greater diameter in comparison to cellulose. Nevertheless, following the elimination of lignin and the implementation of alkali treatment, the presence of these components is much diminished, highlighting the efficacy of SEM in assessing alterations in structure.

X-ray diffraction (XRD) analysis confirms cellulose's highly organized crystalline formations, vital for its structural integrity. Absence of a peak at $2\theta = 18°$ indicates successful removal of lignin and hemicellulose, enhancing cellulose purity and crystallinity. The major finding highlights the significant impact of mechanical treatment on cellulose crystallinity, with Pelissari et al. (2014) emphasizing the selective removal of amorphous parts to enhance crystalline nature. Notably, DH 1 exhibits the highest index (45.20%), indicating a more ordered structure compared to DH 2 (34.20%). This finding underscores the importance of processing procedures in influencing cellulose crystallinity, with implications for barrier materials like paper and paperboard.

Thermogravimetric analysis (TGA) of chemically treated cellulose reveals significant insights into their thermal stability. Initial weight loss, attributed to thermal decomposition and moisture evaporation, occurs between 38°C and 120°C. Hemicellulose and lignin degradation is observed around 200°C, while cellulose degradation spans 270°C to 400°C. Notably, RH exhibits substantial weight reduction during the second quarter, while DH2 compensates for minimal initial loss with significant decline later. PP1 and PP2 also undergo notable weight reductions. Overall, TGA analysis underscores diverse weight loss patterns among samples, highlighting

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variations in thermal stability and composition.

The research results suggest that PP1 is the most favourable candidate for producing biodegradable microbeads. The visual appearance and analytical data indicate positive results for PP1 compared to other samples. The results indicate that PP1 has exceptional qualities that are well-suited for manufacturing biodegradable microbeads. Additional scrutiny and verification of the characteristics of PP1 could potentially facilitate its implementation in the advancement of sustainable materials.

5.2 Recommendations

The extraction and utilization of cellulose offer great potential for tackling urgent environmental issues and promoting the advancement of sustainable materials research. The study emphasizes the significance of enhancing extraction techniques, defining cellulose fibers, investigating various uses, optimizing processes, and promoting stakeholder collaboration.

Optimizing extraction procedures, such as alkali treatment and bleaching methods, is essential for improving the effectiveness of cellulose extraction from agricultural by-products such as rice husk, durian husk, and pineapple peels. Further exploration of alternate extraction processes has the potential to enhance yields and minimize environmental repercussions.

Furthermore, conducting a thorough analysis and rigorous quality assessment of cellulose is crucial to guarantee their integrity and uniformity for diverse uses. Advanced analytical techniques such as FTIR spectroscopy, SEM, and X-ray diffraction are essential in this context, as they offer vital information about the composition and

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characteristics of cellulose samples.

Furthermore, it is crucial to investigate various uses for cellulose that go beyond the manufacture of microbeads. Sectors such as renewable energy, textiles, healthcare, and packaging have the potential to profit significantly from the distinctive characteristics of materials derived from cellulose. This field primarily focuses on examining the appropriateness of cellulose for uses and creating novel products with improved characteristics.

Optimizing processing procedures is crucial for achieving maximum resource efficiency and minimizing waste generation. This study emphasizes the importance of promoting sustainability in cellulose extraction and production processes and integrating eco-friendly methods throughout the supply chain. By taking this action, we can reduce adverse environmental effects and strengthen sustainability initiatives.

In future studies, conducting biodegradability assessments on microbeads would provide valuable insights into their environmental impact and sustainability, enhancing our knowledge and promoting the development of eco-friendly solutions.

Finally, it is crucial to promote cooperation among studies, industry stakeholders, and policymakers to expedite the advancement and acceptance of cellulose-based solutions. We may enhance multidisciplinary research, promote knowledge sharing, and facilitate technology transfer to tackle complex challenges and bring about good change in the cellulose industry.

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APPENDIX A

The yield of treated cellulose extraction were analyzed by equation below:

Analysis of cellulose yield:

(Weight of extracted cellulose (g) / Weight of the plant powder (g)) x 100%

Table 1: Yield of extraction of chemical treated cellulose fiber from RH, DH and PP using acid and alkaline pretreatment with bleaching process.

Sample	Weight of the plant powder (g)	Weight of extracted cellulose (g)	Yield obtained (%)
RH 1	10.00	7.50	75
DH 1	10.00	7.00	70
DH 2	10.00	6.60	66
PP 1	10.00	7.40	74
PP 2	10.00	6.59	66

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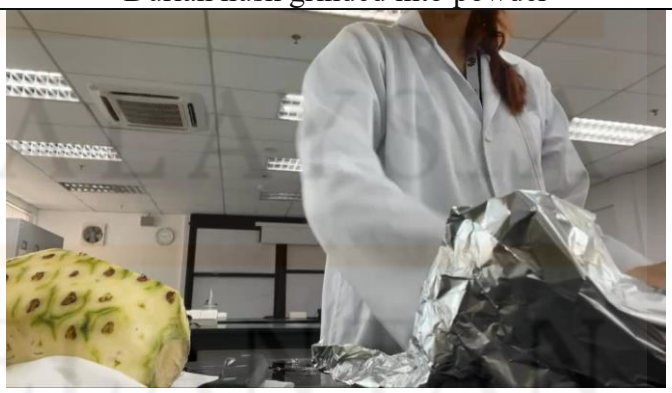
APPENDIX B

Raw materials preparation

Figure	Image
1	 Rice husk from Sekinchan, Selangor
2	 Durian husk grinded into powder
3	 Pineapple Peel fresh cut

4	 Durian peel cut into small pieces before grind
5	 Rice husk obtainedd in ‘sack’

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