

CELLULOSE BASED BIOPLASTIC: ADVANCEMENT AND FUTURE PROSPECTS

Submitted in partial fulfillment of the requirement for
the award of bachelor of technology degree in
Biotechnology

By

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(DEEMED TO BE UNIVERSITY)

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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the Bonafide work of Rakesh S (40230038) who carried out the project **"CELLULOSE BASED BIOPLASTIC: ADVANCEMENTS AND FUTURE PROSPECTS"** under our supervision from August-2023 to April-2024.

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I, Rakesh S hereby declare that the project report entitled **“CELLULOSE BASED BIOPLASTIC: ADVANCEMENT AND FUTURE PROSPECTS”** done by me under the guidance of **Dr. V. RAMESH KUMAR** (Internal) and **Dr. D INBAKANDAN** (External) at Centre for ocean research, Sathyabama research park, Sathyabama institute of science and technology, are submitted in partial fulfillment of the requirements for the award of bachelor of technology degree in biotechnology.

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ABSTRACT

In the past few years, there has been a rising apprehension regarding the sustainability of the environment and the negative consequences associated with conventional petroleum-derived plastics. Consequently, there has been an increased interest in biodegradable alternatives. This study offers a comprehensive study of bioplastic derived from cellulose, source, characteristics, sustainability, advancing progress, and utility. We explore the degradability and compostability of cellulose bioplastics, emphasizing their capacity to mitigate plastic pollution and advance a circular economy. The cellulose was extracted from the natural residue such as sugarcane bagasse, coconut coir and corn cob through alkaline treatment and acid treatment. It was characterized through ATR-FTIR and XRD by confirming the presence of C-O-C glycosidic band. The cellulose was dissolved using zinc chloride method. The cellulose solution was blended with polymer and chitosan which results in a gel like structure. It was then casted in glass slide and cured in hot air oven. The casted bioplastic was peeled from glass slide. Then bioplastic was standardized and further evaluated with different testing analysis and characterization.

KEYWORDS:

Cellulose, bioplastic, alkaline treatment, polymer, formulation, comparative analysis

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LIST OF ABBREVIATIONS

PLA	Polylactic acid
PBAT	Polybutylene adipate terephthalate
PBS	Polybutylene succinate
PCL	Polycaprolactone
PHAs	Polyhydroxyalkanoates
CMC	Carboxymethyl cellulose
BC	Bacterial cellulose
CNC	Cellulose nanocrystal
CNF	Cellulose nano fibers
ZnCl ₂	Zinc chloride
NaOH	Sodium hydroxide
DMSO	Dimethyl sulfoxide
HCL	Hydrochloric acid
H ₂ SO ₄	Sulfuric acid
NaClO	Sodium hypochlorite
H ₂ O ₂	Hydrogen peroxide
PVA	Polyvinyl alcohol
ATR-FTIR	Attenuated total reflection-Fourier transform infrared
XRD	X-ray diffraction

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CHAPTER - 1

INTRODUCTION

Plastics present significant hazards to both the natural surroundings and human well-being. The production and elimination of plastics contribute to the contamination and degradation of the environment, encompassing the detriment of marine life and ecosystem(Halden, 2010). Plastics contain detrimental chemicals that possess the capability to permeate into food and water, thereby resulting in unfavorable health consequences, including cancer, congenital disorders, compromised immunity, and disruption of the endocrine system(Guthrie *et al.*, 2023). The utilization of improper disposal methods, such as landfill and incineration, exacerbates the environmental challenges caused by plastics(Quayle, 1992). The extensive utilization and limited recycling of plastics result in large amounts of waste that contribute to environmental pollution. In order to tackle these concerns, it is imperative to urgently undertake measures that advocate for the application of plastic substitutes, secure disposal of plastic waste, and the formulation of sustainable and eco-friendly recycling methodologies(Nithin *et al.*, 2017).



Fig 1.1 Representation of soil pollution due to plastic

Bioplastics have been proposed as a potential solution for the issues caused by conventional plastics. These biodegradable plastics derived from organic sources can aid in mitigating synthetic polymer pollution and its adverse impact on the habitat (Zhang *et al.*, 2020). Nevertheless, concerns have been raised regarding the possible implications on food security and ecosystems when bioplastics are derived from agricultural materials (Scarpa *et al.*, 2010). In order to tackle this issue, a sustainable and environmentally friendly process has been developed to convert

leftover microalgae into bioplastic film, which has shown promising results in terms of its tensile strength, elongation at break, and resistance to water (Ren, 2003). Furthermore, researchers have investigated the biocompatibility of organic semiconducting polymers, thereby enabling the production of low-cost and mass-produced sensors for biological sensing (Hassan *et al.*, 2019). Although biodegradable plastics hold the potential to effectively manage the accumulation of plastic waste, The effectiveness of these approaches hinges on the presence of economical waste categorization technologies and the commitment to investing in organic waste treatment facilities. (Shen *et al.*, 2020).

Cellulose, a polysaccharide, is the most abundant biopolymer that is recognized by humans. It is apparent in a life forms range of species, incorporating bacteria, animals, and plants. The production of biodegradable cellulose-based bio-plastics, exemplified by the "cellophane film," necessitates the implementation of certain chemical treatments upon cellulose xanthate (Venkatachalam *et al.*, 2020).

During the late 19th century, Parkesine, a pioneering flexible form of nitrocellulose, was employed as a substitute for ivory. The origin of its name can be traced back to its creator, Alexander Parkes. By dissolving cotton Fibers in nitric acid and ethanol, the cellulose was synthesized. This substance, known as collodion, could be shaped under pressure or cast into thin layers. Subsequently, celluloid, which employed camphor, an inflexible, adhesive, volatile aromatic crystalline compound ($C_{10}H_{16}O$), as a plasticizer, eventually progressed and superseded parkesine. In comparison to Parkesine, celluloid is less prone to fracturing due to the presence of the camphor plasticizer. At the close of the 19th century, celluloid emerged as an alternative to ivory, particularly for the production of pool balls, and served as a medium for both still and moving images in the form of photographic film. Additionally, it found application in the creation of windshields. Another derivative of cellulose, known as cellulose acetate, is manufactured through the dissolution of cellulose in a blend of acetic anhydride, sulfuric acid, and acetic acid. Subsequently, sheets are either cast or Fibers are commonly spun using an acetone solution. Cellulose acetate gained significant usage as a film stock during the early 20th century. Moreover, restructured cellulose is obtained from woody fiber through a two-step method involving the dissolution of the crystalline structure with sodium

hydroxide (NaOH), followed by the formation of a gel-like substance using acid(Mooney, 2009),(Stevens, 2020).

This overview explores the realm of cellulose-derived bioplastics, aiming to provide a comprehensive understanding of their origins, manufacturing processes, characteristics, and extensive range of applications, including materials for packaging, agricultural films, medical devices, textiles, and various others. Given the precarious situation we are in as we strive to reduce plastic waste and safeguard our planet's ecosystems, bioplastics made from cellulose provide a ray of hope. This investigation aims to elucidate their importance and role in the ongoing transition towards more sustainable and environmentally responsible materials, thereby paving the way for a future that is greener and more pristine.

1.1 OVERVIEW OF CELLULOSE

Cellulose, an abundant and environmentally adaptable raw material, has garnered significant interest in the industrial sector (Chen *et al.*, 2023). Its abundant availability and versatility make it an attractive option for the construction of multifunctional materials characterized by exceptional properties (Chen *et al.*, 2023). For instance, materials based on cellulose exhibit remarkable electrical conductivity and shielding efficiency, making them suitable for electromagnetic shielding(Califano *et al.*, 2020). Additionally, the incorporation of cellulose nanofibers into nanocomposites yields materials with superior visual transparency, chemical resistance, tensile strength, biodegradability and tensile strength (Saikia, *et al.*, 2019). Moreover, cellulose can be extracted from agro-food waste and utilized in the production of sustainable materials, such as cellulose dispersed in a polylactic acid matrix. Overall, cellulose presents a promising solution for the development of sustainable materials in various applications, including electromagnetic shielding, nanocomposites, and waste utilization (Rathinamoorthy *et al.*, 2022).

1.1.1 SOURCE OF CELLULOSE:

It can be derived from a multitude of sources from agriculture residues, bacterial biosynthesis, wood-based biomass, aquatic organisms and industrial waste. The basic sources of cellulose mainly revolve around plants that are cultivated specifically for the production of cellulose Fibers, such as (fig 1.2) jute, wood, kenaf,

cotton, hemp, and flax. On the other hand, secondary sources of cellulose typically involve agricultural leftover, where lignocellulosic biomass is generated as a

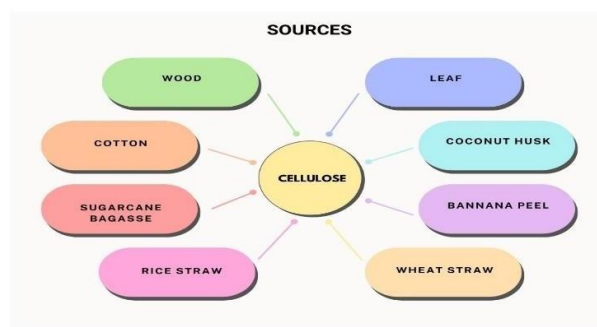


Fig 1.2 Representation of sources of cellulose

byproduct of crops primarily harvested for food and biochemical production his encompasses straw derived from maize, rice crops, and wheat(Pennells *et al.*, 2020). Another source of cellulose involves residual matter obtained from industrialized biomass, such as residues from the food and beverage sector, compacted sugarcane bagasse, sludge from the paper manufacturing process, and municipal waste(García *et al.*, 2016). However, in past years, there has been increasing in exploring alternative sources of cellulose in order to alleviate the pressure on wood-based biomass and enhance the sustainability of the production process. Agricultural residues have emerged as a promising alternative owing to their inherent advantages such as their initial low value, renewable nature, and rapid generation(BOUFI,2017).

1.1.2 PROPERTIES OF CELLULOSE

Cellulose, being an inherent polymer, displays intrinsic attributes such as remarkable crystallinity, a complex nano-network structure, a superior elastic modulus, a substantial tensile strength, notable permeability, biodegradability in the natural environment, and commendable biocompatibility(Li *et al.*, 2022). Cellulose nanofibrils exhibit remarkable tensile characteristics(Peng *et al.*, 2018). Cellulose also presents exceptional thermal, mechanical, and sustainable attributes.

1.2 OVERVIEW OF BIOPLASTIC

The pursuit of alternatives has been initiated in response to the environmental issues that arise from the disposal of artificial plastics. Bioplastics, possessing both functional similarities to synthetic plastics and ecological sustainability, are being promoted as prospective novel materials to combat these issues (Atiwesh *et al.*, 2021). The degradation of most petroleum-based plastics is a slow process due to the presence of crosslinked covalent bonds, especially the carbon-carbon bonds. On the other hand, the cellulose's oxygenated molecular chains can be broken down by naturally occurring yeast, bacteria, and fungi in the soil (Coughlan, 1991).

1.2.1 TYPES OF BIOPLASTICS

1.2.1.1 CELLULOSE BASED BIOPLASTICS

A diverse array of organic matter can be harnessed for the purpose of generating cellulose, encompassing materials such as timber, seed filaments, plant stalk Fibers, vegetation, aquatic organisms (specifically tunicates), photosynthetic organisms, fungi, animals without backbones, and microorganisms (Ahsan *et al.*, 2023). The utilization of plastic derived from cellulose encounters limitations in the market due to the exorbitant expenses involved in its production (Venkatachalam *et al.*, 2020).

1.2.1.2 STARCH BASED BIOPLASTIC

The polysaccharide known as starch is widely present on our planet. Plastics derived from starch are primarily manufactured using wheat, potatoes, rice, and corn, among other starch-rich plant sources. These starch-based plastics are intricate combinations of starch and compostable plastics, including PLA, PBAT, PBS, PCL, and PHAs (Venkatachalam *et al.*, 2020). The composition of this substance involves two distinct categories of polymers: linear amylose and branched amylopectin. (Hernández-Carmona *et al.*, 2017). Linear amylose provides the crucial characteristic of elasticity in bioplastics, whereas the branched structure of amylopectin governs both tensile strength and elongation. Starch, due to its cost-effectiveness, has gained significant popularity as one of the most favourable biopolymers in the production of edible films (Ahsan *et al.*, 2023).

1.2.1.3 PLA BASED BIOPLASTIC

Polylactic acid, which is also referred to as polylactide or PLA, is a thermoplastic material that is commercially available and biodegradable. It is based on lactic acid and is often considered as commonly used degradable aliphatic polyester, it being a heat sensitive polymer, possesses the characteristic of being non–aromatic and aliphatic non-cyclic. It is derived from lactide and lactic acid, with its formation occurring through the polymerization of sugars that are obtained from various sources of agricultural biomass.(Helanto *et al.*, no date). The PLA polymer possesses immense appeal for applications in the realm of biology and medicine, as it has the capability to be converted into filaments that can be harnessed to fabricate textiles or films. It has been sanctioned for direct interaction with food and is employed in edible enclosure items because of its benefits attributes such as elevated transparency, gloss, rigidity, printability, processability, and exceptional ability to impede aroma(Venkatachalam *et al.*, 2020). In contrast to alternative artificial polymers, polylactic acid exhibits a comparatively reduced emission of greenhouse gases(Ahsan *et al.*, 2023).

1.2.2 PROPERTIES OF BIOPLASTIC

The ecological nature of bioplastics does not align with the superior attributes of conventional plastics that determine their ultimate applications. Despite the fact that most bioplastics exhibit reduced mechanical strength, a subset of bioplastics boasts significant tensile strength(Kong, *et al.*, 2023).

1.3 APPLICATIONS

1.3.1 HEALTHCARE INDUSTRY

Cellulosic biopolymers have gained significant traction within the healthcare sector owing to their multifaceted utility and wide-ranging applications. These cellulose-derived biopolymers find extensive utilization in the preparation of various pharmaceutical dosage form and product. They find extensive usage in pharmaceutical contexts, including the development of extended-release and late-release coated dosage forms, osmotic drug delivery system, controlled-release matrices, and bio-adhesive materials. Moreover, cellulose-based biopolymers function as enhancers for compressibility in tablet compression, thickening agents

and stabilizers in liquid dosage forms, binders in granular and tablet formulations, and gelling agents in semisolid preparations. The continuous production of novel derivatives and the exploration of novel applications for cellulose-based biopolymers have further augmented their significance within the healthcare industry. In the forthcoming era, with respect to the implementation of fortified bioplastics in the healthcare sector, there exists a potential for the utilization of bioplastics in biophotonic applications. Bio photonics, as a field of technology, involves the exploitation or creation of photons and light in order to visualize, discern, and scrutinize cells and tissues via the light characteristics of organic structures. By employing reinforced bioplastics in the human body, this particular application could potentially facilitate the process of tissue engineering(Shokri *et al.*, 2013; Kong *et al.*, 2023).

1.3.2 AGRICULTURE INDUSTRY

Cellulose-derived biopolymers hold considerable potential for application within the agriculture sector. These biopolymers possess desirable qualities such as malleability, notable strength, exceptional modulus, biodegradability, and recyclability. Consequently, they find utility across a range of agricultural products and processes. Their application extends to serving as coatings for synthetic Fibers, nonwoven fabrics, and films. Using renewable resources to strengthen bioplastics for use in the plant nursery industry would serve a comparable purpose to bioplastics in biodegradable mulch films, which enhance soil fertility through the decomposition of the strengthened bioplastics in the soil. Despite this, it is of utmost importance to thoroughly investigate the elements and gases emitted to prevent any harmful effects on agricultural crops(Peng *et al.*, 2020; Kong *et al.*, 2023).

1.3.3 PACKAGING INDUSTRY

Cellulosic bioplastics have demonstrated potential for utilization in the packaging sector. These biodegradable substances provide an environmentally friendly alternative to traditional synthetic polymer, which constitute to the volume of waste. Numerous investigations have examined the utilization of cellulose, including bacterial cellulose (BC) and carboxymethyl cellulose (CMC) in the creation of packaging materials. The integration of CMC obtained from agricultural waste, such as sugar cane bagasse, has been examined to reduce expenses and enhance the

characteristics of biodegradable films(Yaradoddi *et al.*, 2020). Bacterial cellulose, a naturally occurring biopolymer produced by bacteria, has been employed for the manufacture of antimicrobial packaging materials that have the potential for utilization in the fields of food, and medicine (Zahan *et al.*, 2020). Furthermore, the combination of cellulose acetate (CA) with polysaccharides, such as carrageenan (CG) and sodium alginate (SA), has led to the creation of cost-effective and environmentally friendly packaging films for food (Rajeswari *et al.*, 2020). Moreover, the incorporation of BC with CMC and polyvinylpyrrolidone (PVP) has been examined to enhance the thermoplastic properties and longevity of packaging materials (Bandyopadhyay, Saha and Saha, 2020). In summary, these investigations underscore the promise of cellulose-based bioplastics as sustainable packaging solutions across various industries (Rovera *et al.*, 2020).

CHAPTER – 2

LITERATURE SURVEY

(Rani *et al.*, 2018) Bio nanocomposites Reinforced with Cellulose Nanofibers Derived from Sugarcane Bagasse. The article explores the potential of bio nanocomposites reinforced with cellulose nanofibers extracted from sugarcane bagasse. Sugarcane bagasse, a byproduct of sugar production, offers a sustainable and cost-effective source of cellulose. These nanofibers, when incorporated into polymer matrices, enhance the mechanical, thermal, and barrier properties of the resulting bio nanocomposites. This research signifies a promising avenue for the utilization of agricultural waste in the development of eco-friendly materials with applications in various industries such as packaging, automotive, and construction. The study underscores the importance of sustainable materials development for a greener future.

(Aguilar *et al.*, 2019) Magnetic bioplastics based on isolated cellulose from cotton and sugarcane bagasse.

The study delves into the creation of magnetic bioplastics utilizing isolated cellulose from both cotton and sugarcane bagasse. By integrating magnetic particles into bioplastic matrices derived from these cellulose sources, researchers aim to enhance functionalities for diverse applications. This innovative approach not only utilizes renewable resources but also introduces magnetic properties, expanding potential applications in fields like biomedicine, electronics, and environmental remediation. The research underscores the versatility and sustainability of bioplastics, offering a promising avenue for eco-friendly material development with advanced functionalities.

(Shahi *et al.*, 2020) Eco-Friendly Cellulose Nanofiber Extraction from Sugarcane Bagasse and Film Fabrication.

The article investigates an eco-friendly method for extracting cellulose nanofibers from sugarcane bagasse and subsequently fabricating films. Through a sustainable process, researchers aim to harness the abundant cellulose in sugarcane bagasse, a byproduct of sugar production. These extracted nanofibers serve as a renewable and biodegradable material for film fabrication, offering potential applications in packaging and other industries. This research highlights the importance of environmentally friendly approaches in material extraction and processing, paving the way for the development of eco-conscious alternatives to conventional plastics.

(Melesse *et al.*, 2022)Extraction of Cellulose from Sugarcane Bagasse Optimization and Characterization.

The study focuses on optimizing the extraction of cellulose from sugarcane bagasse while thoroughly characterizing the resulting material. By fine-tuning extraction parameters, researchers aim to enhance the efficiency and yield of cellulose extraction from this abundant agricultural waste. Characterization techniques such as spectroscopy and microscopy are employed to analyze the structure, purity, and properties of the extracted cellulose. This research not only offers insights into improving extraction processes but also provides valuable information on the structural and functional attributes of cellulose from sugarcane bagasse, paving the way for its utilization in various applications spanning from bioplastics to biomedical materials.

(Alizadeh Asl *et al.*, 2017)Synthesis and Characterization of Carboxymethyl Cellulose from Sugarcane Bagasse

The article delves into the synthesis and comprehensive characterization of carboxymethyl cellulose (CMC) derived from sugarcane bagasse. Through a multi-step synthesis process, researchers aim to transform cellulose from sugarcane bagasse into CMC, a versatile and eco-friendly polymer with numerous industrial applications. Characterization techniques such as spectroscopy, microscopy, and rheology are employed to analyze the chemical structure, morphology, and rheological properties of the synthesized CMC. This research not only contributes

to the valorization of sugarcane bagasse but also provides valuable insights into the structural and functional attributes of CMC, offering potential applications in areas ranging from food and pharmaceuticals to textiles and cosmetics.

(Liu *et al.*, 2006) Isolation and Characterization of Cellulose Obtained from Ultrasonic Irradiated Sugarcane Bagasse.

The research focuses on the isolation and subsequent characterization of cellulose obtained from sugarcane bagasse through ultrasonic irradiation. This innovative method utilizes ultrasonic energy to break down the lignocellulosic structure of sugarcane bagasse, facilitating the extraction of cellulose fibers. Characterization techniques such as spectroscopy and microscopy are employed to analyze the structural and morphological properties of the isolated cellulose. This study sheds light on the potential of ultrasonic irradiation as a green and efficient approach for cellulose extraction from agricultural waste, contributing to the development of sustainable materials for various applications.

(Rahimi Kord Sofla *et al.*, 2016) A comparison of cellulose nanocrystals and cellulose nanofibres extracted from bagasse using acid and ball milling methods.

The study conducts a comparative analysis between cellulose nanocrystals (CNCs) and cellulose nanofibers (CNFs) extracted from bagasse utilizing acid and ball milling methods. Through these techniques, researchers aim to isolate and characterize CNCs and CNFs from bagasse, evaluating their properties and potential applications. Characterization methods such as spectroscopy, microscopy, and rheology are employed to assess the structural, morphological, and rheological properties of the extracted nanocellulose materials. This research provides valuable insights into the efficiency and effectiveness of acid and ball milling methods for producing CNCs and CNFs from bagasse, contributing to the advancement of sustainable nanocellulose-based materials for various industrial applications.

(Sun *et al.*, 2004) Isolation and characterization of cellulose from sugarcane bagasse.

The study focuses on the isolation and subsequent characterization of cellulose obtained from sugarcane bagasse. Various methods, such as chemical, mechanical, or enzymatic treatments, are employed to extract cellulose fibers from the lignocellulosic matrix of sugarcane bagasse. Subsequently, the isolated cellulose is thoroughly characterized using analytical techniques such as spectroscopy, microscopy, and rheology to determine its chemical composition, structure, morphology, and properties. This research aims to provide valuable insights into the composition and quality of cellulose derived from sugarcane bagasse, which can inform its potential applications in various industries, including papermaking, textiles, biofuels, and bio composites.

(Abdullah *et al.*, 2021) Structure and thermal properties of microcrystalline cellulose extracted from coconut husk fibre.

The research focuses on investigating the structure and thermal properties of microcrystalline cellulose (MCC) extracted from coconut husk fibre. Through a series of chemical and mechanical treatments, cellulose is isolated from coconut husk fibers and processed into MCC. Structural analysis techniques, such as spectroscopy and microscopy, are employed to characterize the morphology and crystalline structure of the extracted MCC. Additionally, thermal analysis methods, including thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC), are utilized to assess the thermal stability and properties of the MCC. This study provides valuable insights into the structure-property relationships of MCC derived from coconut husk fibre, which can inform its potential applications in various industries, including pharmaceuticals, food, and composites.

(POORNACHANDHRA *et al.*, 2023) Coconut Husk Fiber: A Low-Cost Bioresource for the Synthesis of High-Value Nanocellulose.

The study explores coconut husk fibre as a low-cost bioresource for synthesizing high-value nanocellulose. Utilizing an environmentally friendly extraction process,

researchers isolate nanocellulose from coconut husk fibre, a readily available agricultural waste. Through a combination of mechanical and chemical treatments, the cellulose fibers are broken down into nanoscale dimensions. Characterization techniques such as spectroscopy and microscopy are used to analyze the structure and morphology of the nanocellulose. This research highlights the potential of coconut husk fibre as a sustainable source for producing valuable nanocellulose, which can find applications in various industries including packaging, textiles, and biomedical materials.

(Fahma *et al.*, 2011)Effect of pre-acid-hydrolysis treatment on morphology and properties of cellulose nanowhiskers from coconut husk.

The research investigates the effect of pre-acid-hydrolysis treatment on the morphology and properties of cellulose nanowhiskers extracted from coconut husk. Through this treatment, researchers aim to enhance the efficiency of cellulose nanowhisiker isolation from coconut husk fibers. The study evaluates changes in the morphology, size, and crystallinity of the nanowhiskers using microscopy and spectroscopy techniques. Additionally, the mechanical and thermal properties of the nanowhiskers are characterized to assess their suitability for various applications. This research provides valuable insights into optimizing the extraction process of cellulose nanowhiskers from coconut husk, enhancing their potential for use in industries such as packaging, textiles, and biomedical materials.

(Nascimento *et al.*, 2014)A novel green approach for the preparation of cellulose nanowhiskers from white coir.

The research presents a novel green approach for the preparation of cellulose nanowhiskers from white coir, a natural fibre obtained from the husk of coconuts. This innovative method aims to extract cellulose nanowhiskers in an environmentally friendly manner, minimizing chemical usage and waste generation. By utilizing sustainable and biocompatible solvents and processing techniques, the study focuses on preserving the natural properties of cellulose while maximizing yield and purity. Characterization techniques such as microscopy, spectroscopy,

and rheology are employed to analyze the structure, morphology, and properties of the obtained nanowhiskers. This research offers a promising avenue for the sustainable production of cellulose nanowhiskers from white coir, with potential applications in various industries including packaging, textiles, and biomedical materials.

(Rosa *et al.*, 2010) Cellulose nanowhiskers from coconut husk fibers: Effect of preparation conditions on their thermal and morphological behaviour

The study investigates the effect of preparation conditions on the thermal and morphological behaviour of cellulose nanowhiskers derived from coconut husk fibers. By varying parameters such as acid concentration, reaction time, and temperature during the preparation process, researchers aim to optimize the properties of the resulting nanowhiskers. Thermal analysis techniques, such as thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC), are employed to evaluate the thermal stability and properties of the nanowhiskers under different conditions. Additionally, microscopy and spectroscopy methods are used to assess changes in the morphology and structure of the nanowhiskers. This research provides valuable insights into the relationship between preparation conditions and the thermal and morphological behaviour of cellulose nanowhiskers from coconut husk fibers, offering opportunities for their tailored application in various fields such as composites, packaging, and biomedical materials.

(Wu *et al.*, 2019) Preparation and characterization of cellulose nanofibrils from coconut coir fibers and their reinforcements in biodegradable composite films

The study focuses on the preparation and characterization of cellulose nanofibrils (CNFs) derived from coconut coir fibers, as well as their incorporation as reinforcements in biodegradable composite films. Through a series of mechanical and chemical treatments, cellulose nanofibrils are extracted from coconut coir fibers, aiming to achieve high purity and uniform morphology. The resulting CNFs are then characterized using various analytical techniques, including microscopy, spectroscopy, and rheology, to assess their structure, morphology, and properties.

Subsequently, the CNFs are incorporated into biodegradable polymer matrices to fabricate composite films, and the mechanical, thermal, and biodegradation properties of these films are evaluated. This research offers insights into the potential use of CNFs derived from coconut coir fibers as sustainable reinforcements in biodegradable composite materials, contributing to the development of eco-friendly alternatives for various applications such as packaging, textiles, and biomedical devices.

(Jia *et al.*, 2016) Preparation of carboxymethyl cellulose from corncob

The research focuses on the preparation of carboxymethyl cellulose (CMC) from corncob, a renewable agricultural residue. Carboxymethylation, a chemical modification process, is employed to introduce carboxymethyl groups onto the cellulose backbone of corncob fibers, resulting in the synthesis of CMC. The preparation process typically involves alkaline treatment followed by etherification with sodium monochloroacetate under controlled conditions. Various parameters such as reaction time, temperature, and reagent concentrations are optimized to achieve desired CMC properties. Characterization techniques including spectroscopy, chromatography, and rheology are utilized to analyze the structure, composition, and properties of the synthesized CMC. This research offers insights into the utilization of corncob as a sustainable feedstock for the production of value-added cellulose derivatives like CMC, with potential applications in industries such as food, pharmaceuticals, textiles, and cosmetics.

(Ach, 1993) BIODEGRADABLE PLASTICS BASED ON CELLULOSE ACETATE

Biodegradable plastics based on cellulose acetate are developed using cellulose, a renewable and abundant natural polymer, as a starting material. Cellulose acetate is synthesized through the esterification of cellulose with acetic anhydride or acetic acid under controlled conditions. This process modifies cellulose's hydroxyl groups with acetyl groups, resulting in improved solubility and processability. The resulting cellulose acetate can be further processed into various forms, such as films, fibers, or pellets, using conventional plastic processing techniques. These biodegradable

plastics offer potential applications in packaging, agriculture, textiles, and biomedical fields, providing a sustainable alternative to conventional petroleum-based plastics while addressing environmental concerns related to plastic pollution. Additionally, their biodegradability ensures they can be decomposed by microorganisms in the environment, reducing long-term environmental impact.

(Arief *et al.*, 2021) The concentration of sorbitol on bioplastic cellulose based carrageenan waste on biodegradability and mechanical properties bioplastic

The concentration of sorbitol in bioplastic cellulose based on carrageenan waste significantly influences both biodegradability and mechanical properties, with higher sorbitol content generally enhancing biodegradability by increasing porosity and providing a carbon source for microbial activity, while also improving flexibility but potentially compromising mechanical strength and stability, necessitating careful optimization to achieve a balance between these properties for specific application requirements.

(Chadijah *et al.*, 2018) Determination of the optimum concentration cellulose bagasse in making film bioplastic

Determining the optimum concentration of cellulose bagasse in bioplastic film production is pivotal for achieving desired properties. Balancing factors like mechanical strength, flexibility, biodegradability, and cost efficiency is essential. Through systematic experimentation, the ideal cellulose bagasse concentration can be pinpointed, ensuring optimal film performance while maximizing the use of renewable resources and minimizing environmental impact.

(Kostag *et al.*, 2021) Sustainable biomaterials based on cellulose, chitin and chitosan composites

Sustainable biomaterials based on composites of cellulose, chitin, and chitosan represent a promising avenue for eco-friendly materials development. Cellulose, a renewable polymer from plant sources, combined with chitin and chitosan, derived

from crustacean shells, offers biodegradable and biocompatible properties. These composites exhibit enhanced mechanical strength, barrier properties, and biodegradability, making them suitable for various applications in packaging, biomedical devices, and environmental remediation. By harnessing natural resources and employing green processing methods, these biomaterials contribute to reducing reliance on fossil-based plastics and mitigating environmental pollution.

(Lubis *et al.*, 2017) Utilization starch of jackfruit seed (*Artocarpus heterophyllus*) as raw material for bioplastics manufacturing using sorbitol as plasticizer and chitosan as filler

The utilization of starch from jackfruit seeds (*Artocarpus heterophyllus*) as a raw material for bioplastics manufacturing, with sorbitol as a plasticizer and chitosan as a filler, presents a sustainable approach to bioplastic production. Jackfruit seeds, an abundant agricultural byproduct, offer a renewable source of starch suitable for bioplastic synthesis. Sorbitol, a biodegradable polyol, enhances the flexibility and processability of the bioplastic, while chitosan, derived from crustacean shells, serves as a filler to improve mechanical strength and biodegradability. This eco-friendly formulation not only reduces dependence on fossil fuels but also addresses environmental concerns associated with traditional plastics, offering a biodegradable alternative with potential applications in packaging, agriculture, and biomedical fields.

(Lee *et al.*, 2021) Double-crosslinked cellulose nanofiber-based bioplastic films for practical applications

Double-crosslinked cellulose nanofiber-based bioplastic films represent a promising advancement for practical applications. By employing two separate crosslinking mechanisms, these films exhibit enhanced mechanical strength, water resistance, and thermal stability compared to traditional cellulose-based materials. The first crosslinking step involves chemical crosslinking of cellulose nanofibers, while the second step involves physical crosslinking through hydrogen bonding or electrostatic interactions. This dual-crosslinking approach imparts durability and

resilience to the bioplastic films, making them suitable for a wide range of applications including packaging, biomedical devices, and coatings. Additionally, their renewable and biodegradable nature further enhances their appeal as eco-friendly alternatives to conventional plastics.

(Chen *et al.*, 2020) Dissolution and structure change of bagasse cellulose in zinc chloride solution

The dissolution and structural alteration of bagasse cellulose in zinc chloride (ZnCl_2) solution occur through a multi-step process, initiated by the swelling of cellulose chains due to hydrogen bonding interactions with ZnCl_2 molecules. As the concentration of ZnCl_2 increases, it disrupts intermolecular hydrogen bonds within the cellulose structure, leading to the dissolution of cellulose chains into the solution. Concurrently, the crystalline structure of cellulose undergoes disruption, primarily affecting the amorphous regions and resulting in a more disordered and amorphous structure compared to its native state. This understanding of the dissolution and structural changes in ZnCl_2 solution is crucial for applications involving cellulose dissolution and the production of cellulose-based materials.

(Steven *et al.*, 2022) Facile Preparation of Cellulose Bioplastic from *Cladophora* sp. Algae via Hydrogel Method

The facile preparation of cellulose bioplastic from *Cladophora* sp. algae via the hydrogel method offers a sustainable and efficient approach to bioplastic production. This method involves the conversion of cellulose-rich algae biomass into a hydrogel through a simple and environmentally friendly process. The hydrogel is then processed into bioplastic films using methods such as casting or compression molding. The resulting bioplastic exhibits favourable properties such as biodegradability, mechanical strength, and barrier properties, making it suitable for various applications in packaging, agriculture, and biomedical fields. This innovative approach not only utilizes renewable resources but also addresses environmental concerns associated with conventional plastics.

(Păușescu *et al.*, 2022) Bio-Based pH Indicator Films for Intelligent Food Packaging Applications.

Bio-based pH indicator films offer a sustainable solution for intelligent food packaging applications. These films are composed of biodegradable materials derived from renewable sources, such as cellulose or starch, integrated with natural pH-sensitive indicators. The pH indicator allows for real-time monitoring of food freshness and quality by changing colour in response to fluctuations in pH levels. This technology enables consumers to easily assess food safety and freshness, reducing food waste and promoting sustainable consumption practices. Moreover, the biodegradable nature of these films aligns with environmental concerns, offering an eco-friendly alternative to traditional packaging materials. Overall, bio-based pH indicator films hold promise for revolutionizing intelligent food packaging with their combination of sustainability and functionality.

(Sharif Hossain *et al.*, 2016) Nano-cellulose derived bioplastic biomaterial data for vehicle bio-bumper from banana peel waste biomass.

Nano-cellulose derived bioplastic biomaterial from banana peel waste biomass offers a sustainable solution for vehicle bio-bumper applications. By utilizing nano-cellulose extracted from banana peel waste, a renewable and abundant resource, bioplastic biomaterials can be engineered to possess enhanced mechanical properties, such as high strength and impact resistance. Additionally, nano-cellulose reinforcement enhances the biodegradability and eco-friendliness of the bioplastic, aligning with sustainability goals. Data on the mechanical strength, durability, and biodegradability of these nano-cellulose-based bioplastics indicate their suitability for vehicle bio-bumpers, providing both performance and environmental benefits in automotive applications.

CHAPTER - 3

AIM

To develop an environmentally sustainable cellulose-based bioplastic, aiming to replace conventional petroleum-based plastics and contribute to reducing environmental pollution.

SCOPE:

- To investigate and optimize suitable resources for cellulose
- Cellulose extraction process
- Characterization of cellulose
- Formulation of cellulose-based bioplastic

CHAPTER – 4

MATERIALS AND METHOD

4.1 MATERIALS

- Sugarcane bagasse
- Coconut coir
- Corncob
- NaOH
- DMSO
- H₂O₂
- HCl
- ZnCl₂
- H₂SO₄
- NaClO
- Urea
- PVA
- chitosan.

4.2 METHODS

PREPARATION:

The corn cob samples were washed, dried and cut manually into small pieces of about 1cm and was dried in a hot air oven.

4.2.1 EXTRACTION PROCESS OF CELLULOSE FROM CORN COB:

4.2.1.1 PRE-ALKALINE AND ALKALINATION:

Once the drying process is complete, the dried corn cob was immersed in a solution consisting of 2.5% NaOH, allowing them to soak overnight at room temperature. By removing tiny amount of lignin, hemicellulose, wax, and oils through pre-alkaline treatment it improves the raw materials overall quality and purity. The corn cob content was then put on the steam pressure pot with NaOH solution at 170°C, 0.7 MP for 90 min. After the pressure is released, the contaminants and lignin are removed from the fibers, causing explosive defibrillation and fragmentation of the fibre structure. This process also releases the shorter crystalline particles and disassembles the cellulose's initial configuration, separating the outer cellulose microfibrils. After that, the pulp was rinsed many times with distilled water in order to get rid of any last bits of alkaline residue(Jia *et al.*, 2016).



Fig 4.1 Representation of pre alkaline and alkaline process for corn cob

4.2.1.2 BLEACHING:

The cellulose residue was separated via filtration, washed thoroughly with water to neutral, dried and tested. H₂O₂ was added to bleach the crude cellulose. Lignin is

alkali soluble and hemicellulose in water soluble polysaccharide. So, both the lignin and hemicellulose are removed during alkaline and bleaching(Jia et al., 2016).



Fig 4.2 Representation of bleaching process for corn cob

4.2.1.3 ACID TREATMENT AND DRYING

The content is then treated with HCl to get microcrystalline cellulose and further was bleached to eliminate all contaminants and the fibre bundles. After that, the mixture is continuously rinsed with distilled water to eliminate all traces of acid until the pH is neutral. The acid treated content is kept for drying to remove the excess moisture content and was grinded into fine powder using mortar pestle(Jia et al., 2016).



Fig 4.3 Representation of acid treatment and drying process for corn cob

PREPARATION OF SUGARCANE BAGASSE:

The sugarcane bagasse samples were washed, dried and cut manually into small pieces about 1cm and was dried in hot air oven

4.2.2 EXTRACTION PROCESS OF CELLULOSE FROM SUGARCANE BAGASSE:

4.2.2.1 PREALKALINATION:

The sugarcane bagasse, are washed thoroughly with water and dried completely. Once the drying process is complete, the dried contents are immersed in a solution consisting of 2% NaOH, allowing them to soak overnight at room temperature. This pre-alkaline treatment serves the purpose of extracting a minute quantity of lignin, hemicellulose, wax, and oils that may be present on the external surface of the raw materials, thereby enhancing their overall quality and purity(Rani *et al.*, 2018).

4.2.2.2 ALKALINATION:

The treated content is autoclaved with 8-10% of NaOH at a pressure around 15lb. The pressure was released resulting in explosive defibrillation and the shattering of the fibre structure with the removal of impurities and lignin. This procedure additionally breaks down the original arrangement of cellulose, separates the outer cellulose microfibrils, and reveals shorter crystallites. The resulting pulp was subsequently rinsed multiple times with distilled water to eliminate any remaining alkaline residue(Poornachandhra *et al.*, 2023).

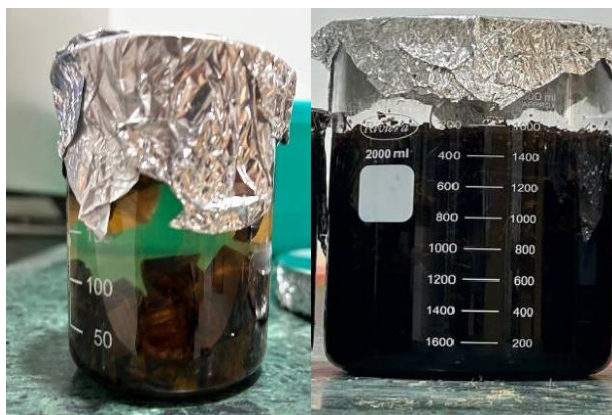


Fig 4.4 Representation of pre alkaline and alkaline process for sugarcane bagasse

4.2.2.3 BLEACHING:

The resulting pulp was then bleached by soaking it on 8% of H_2O_2 for 24h and was washed with distilled water to remove remaining sticking substance. Lignin is alkali soluble and hemicellulose is water soluble polysaccharide. Hence this process leads to the removal of both lignin and hemicellulose (Rani *et al.*, 2018).



Fig 4.5 Representation of bleaching process for sugarcane bagasse

4.2.2.4 ACID TREATMENT:

The bleached content is treated with 10% HCl to remove all the impurities and release the fiber bundles. The solution was further washed with distilled water to remove the acid content completely till it reaches the neutral pH. The acid treated content is kept for drying to remove the excess moisture content. Then the dried content is grinded into fine powder using mortar pestle (Rani *et al.*, 2018).



Fig 4.6 Representation of acid treatment and drying process for sugarcane bagasse

PREPARATION OF COCONUT COIR:

The coconut coir was cut into pieces with length ranging from 0.5 to 1 cm before they were exposed to any treatments.

4.2.3 EXTRACTION PROCESS OF CELLULOSE FROM COCONUT COIR:

4.2.3.1 PREALKALINE AND ALKALINE TREATMENT:

A pre-treatment of soaking was applied to coconut fibers in order to remove contaminants and waxy materials that were covering the fiber cell walls outside. The primary goal of these treatment was to get rid of non-cellulosic materials like lignin and hemicellulose. Coir fibres were autoclaved at high pressure (20 pounds) and temperature (121 °C) for 1.5 hours after being soaked in 2% NaOH for 2 hours at 25 °C. It is well known that solid biomass lignin is transformed into a soluble liquid phase by the alkaline delignification process(Poornachandhra *et al.*, 2023a).



Fig 4.7 Representation of pre alkaline and alkaline process for coconut coir

4.2.3.2 BLEACHING:

To get rid of any remaining lignin, sodium hypochlorite (NaClO) bleaching was applied for 20 minutes. The chlorine residue was eliminated from the bleached fibers by giving them a thorough water wash. Until the residue turned ivory yellow or white, this process was repeated(Wu *et al.*, 2019).



Fig 4.8 Representation of bleaching process for coconut coir

4.2.3.3 ACID TREATMENT:

In brief, the bleached coir were treated in a concentrated H_2SO_4 solution (64wt% sulfuric acid in water) at 45°C . The ratio of fibers to acid solution was 1–10 g/ml. Higher the ratio of coir the better their dispersion and stability in the aqueous medium. After treatment, the hydrolysed cellulose samples were washed (Nascimento *et al.*, 2016).



Fig 4.9 Representation of acid treatment for coconut coir

4.2.3.4 DRYING AND GRINDING:

To eliminate the extra moisture content, the acid-treated material is stored for drying. After that, a mortar and pestle was used to break down the dried material into a fine powder(Shahi *et al.*, 2020).

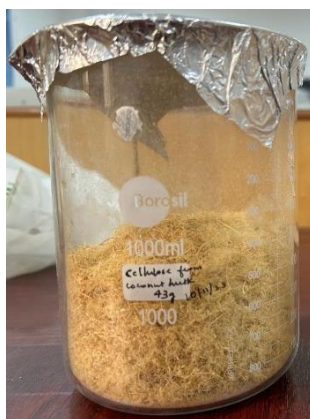


Fig 4.10 Representation of drying process for coconut coir

4.3 CHARACTERIZATION:

4.3.1 FTIR:

FTIR measurements were performed using FTIR analyzer in the range of 4000–600 cm^{-1} . The resolution for the spectra was 4 cm^{-1} . All samples were grinded into small particles and dried in room temperature(Aguilar *et al.*, 2019).

4.3.2 XRD:

XRD patterns of the cellulose were measured using X-ray powder diffractometer. The samples were scanned at the ambient condition over scattering 2 angles from 5° to 90° with the scanning rate of $0.02^\circ/\text{min}$. Crystallinity index of each sample was calculated. X-ray diffraction is used to identify crystalline phases by comparison to a known database, measure atomic spacings (with the use of appropriate standards), measure structural properties of the crystalline phases present (e.g., preferred orientation and particle size), and to probe atomic arrangements in amorphous materials. XRD samples were prepared by pressing raw materials between two glass slides into flattening sheets. The identification of crystalline

materials is often aided by chemical analysis in order to identify the elements present. The mass fractions of the phases present in a sample can be determined by comparison to known standards. Since XRD is relatively quick and is usually a non-destructive technique, it is often one of the first methods employed in the characterization. The electron diffraction pattern is similar to X-ray diffraction pattern through which Bragg condition is satisfied. The Bragg equation ($n \lambda = 2 d \sin \theta$) relates the wavelength (λ) of the electrons, the angle (θ) of the electron beam with respect to a set of diffracting planes, and the spacing (d) between those planes, which is determined by the crystal structure of the material examined. One major challenge when working with electron diffraction is that electrons are often multiply scattered by the sample, which can complicate the interpretation of electron diffraction patterns(Aguilar *et al.*, 2019).

4.4 DISSOLUTION PROCESS:

4.4.1 NaOH/UREA METHOD:

Cellulose can be dissolved using NaOH/urea pre-cooled aqueous solution at a rapid speed. The rapid dissolving of cellulose results from a dynamic process of self-assembly between the macromolecules of cellulose and solvent molecules (NaOH, urea, and water) occurring at low temperatures through the development of new hydrogen bonding networks. This method's primary benefits are its quick disintegration, relative ease of use, affordability, and environmental friendliness.

A solution containing distilled water, NaOH, and urea (7:12:81 by weight) was pre-cooled to -20°C. To create a 4% cellulose solution, a proper amount of cellulose was added right away to the mixture and vigorously stirred for two minutes. The clear resolution that was reached(Qi *et al.*, 2009).



Fig 4.11 Representation of cellulose dissolution process with UREA/NaOH at -20°C

4.4.2 DMSO METHOD:

Dimethyl sulfoxide (DMSO) is a potent solvent that can dissolve cellulose along with a variety of other organic and inorganic substances. Breaking the hydrogen bonds separates the cellulose chains and allows the cellulose to dissolve in DMSO and spread throughout the solvent. 75% DMSO was prepared and kept at stirrer and cellulose was added in powder form. Certain factors are required for the dissolution process to occur, and it might also need for high temperatures and mechanical stirring. Heating quickens the dissolution process by raising the kinetic energy of molecules, while constant stirring guarantees uniform dispersion of cellulose particles in the solvent.



Fig 4.12 Representation of cellulose dissolution process with DMSO

4.4.3 ZINC CHLORIDE METHOD:

The 80% ZnCl_2 was prepared and kept at magnetic stirrer to obtain ZnCl_2 aqueous solution. Then mixture of three cellulose sources at equal ratio were added to ZnCl_2 solution at magnetic stirrer at 80°C for 6 hours(Xiong *et al.*, 2016).

TABLE 4.1: Dissolution of cellulose

S.NO	METHOD	DISSOLVED	PARTIALY DISSOLVED	NOT DISSOLVED
1.	UREA/NaOH	-	-	✓
2.	DMSO	-	-	✓
3.	ZNCL2	-	✓	-

4.5 POLYMER BLENDING AND FORMULATION:

The polymer powder was dissolved in water by using a stirrer at a temperature of 60°C for 1 hour. Equal amount of Chitosan was added to the mixture and was kept in the stirrer for 1 hour. The cellulose solution is added to the polymer mixture with various ratios namely 5:5, 6:4, 7:3, 8:2(Arief *et al.*, 2021).



Fig 4.13 Representation of cellulose solution blended with polymer and chitosan

4.5.1 STANDARDIZATION:

The polymer and chitosan were added with three different cellulose sources, such as corn cob, sugarcane bagasse, coconut coir separately, and the mixture of the three sources together. These were mixed in the ratio of 5:5, 6:4, 7:3, 8:2 at 80-90 C.

TABLE 4.2: Standardization of cellulose incorporated polymer at different ratios

S.NO	NAME OF THE CELLULOSE SOURCE	RATIO	TEMPERATURE
1.	CORN COB	5:5,6:4,7:3,8:2	80°- 90°C
2.	SUGARCANE BAGASSE	5:5,6:4,7:3,8:2	80°- 90°C
3.	COCONUT COIR	5:5,6:4,7:3,8:2	80°- 90°C
4.	MIX	5:5,6:4,7:3,8:2	80°- 90°C

From the following ratios, 6:4 was considered as the optimum ratio to cure the cellulose-incorporated polymer

4.5.2 Characterization through Water contact angle and surface energy.

The water contact angle was used to measure the wetting characteristics of cellulose bioplastics. A goniometer was used to measure the flat surface of the cellulose bioplastic. Ten millilitres of room temperature water were dropped onto the sample's surface. Using the ImageJ software, the picture was taken and the water contact angle was calculated (Steven et al.,2022).Surface energy of bioplastics is essential for understanding their wetting behaviour, adhesion properties, and interactions with other materials. Surface energy refers to the cohesive forces at the surface of a liquid that determine its ability to wet a solid surface(Steven *et al.*, 2022).

4.6 COMPARATIVE ANALYSIS TEST

A swelling test was performed with each individual cellulose-based bioplastic with a mix of three different source cellulose-based bioplastic for 2hrs to 10hrs to study and compare the swelling percentage of cellulose bioplastic(Yaradoddi *et al.*, 2020).



Fig 4.14 swelling test between each individual cellulose-based bioplastic with mixed three different source cellulose-based bioplastic

The results interpret that a mixture of cellulose bioplastic showed minimal swelling percentage when compared to individual sources.

4.7 TEST FOR MIXED CELLULOSE BIOPLASTIC

4.7.1 SOLUBILITY IN SOLVENTS:

Samples were cut into small pieces and placed in a beaker containing different solvents to check the solubility in different solvents. Whether the sample is insoluble, partially soluble or soluble were determined. The solvents used are ethanol, methanol, chloroform and acetone((Yaradoddi *et al.*, 2020)).



Fig 4.15 Solubility test with solvents

4.7.2 DETERMINATION OF OIL PERMEABILITY COEFFICIENT

During the experiment, 1ml of edible oil was added to the centrifuge tube, and the film sample was then sealed within. Additionally, the closed test tube was positioned upside down on the filter paper (which had a known weight), maintained in the middle of the sidewall, and left for a whole day. Following the incubation period, the oil permeability coefficient was computed by applying the following equation to the filter paperweight, which was determined using an electronic weighing machine:

$$P_0 = (\Delta W \times FT) / (S \times T),$$

where FT is the film thickness (mm), S is the film area (m²), and T is the incubation period. P₀ is the oil permeability coefficient (g·mm/(m²·day), and ΔW is the difference in the filter paperweight (g)(Yaradoddi *et al.*, 2020).

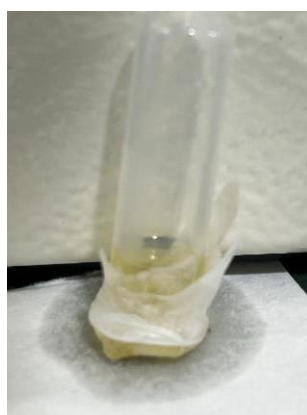


Fig 4.16 Representation of oil permeability test

CHAPTER - 5

RESULTS AND DISCUSSION

5.1 OBTAINED CELLULOSE:

The raw and processed material's chemical makeup and condition of aqueous dispersion are compiled. Following exposure to NaOH, the fibers colour in water changed from dark brown to yellow, indicating a reduction in lignin content. The significant reduction in hemicellulose was brought by the NaOH treatment's disruption and breakage of hydrogen bonds (Shahi *et al.*, 2020). Furthermore, the extractive and ash contents were also decreased. It was discovered that the pure NaOH treatment was insufficient to completely eliminate hemicellulose and lignin. After the samples were treated with NaOH, steam treatment was applied to further remove any remaining lignin. Their lignin concentrations were decreased and it turned pure white following the steam alkaline treatment, leaving no trace of dark lignin residue (Poornachandhra *et al.*, 2023a). The reaction between NaOH and lignin caused aromatic substitution, which is the displacement of side chains in an acidic environment, which is the cause of the decrease in lignin and hemicellulose. Hemicelluloses and lignin showed reductions respectively, suggesting that non-cellulosic components were significantly removed by severe NaOH/NaClO₂ treatment (Rahimi Kord Sofla *et al.*, 2016). Because of the increased NaOH concentration and extended treatment period, the lignocellulose yield reduced after the NaOH treatment (Shahi *et al.*, 2020). The lignocellulose yield dropped following NaClO₂ treatment as a result of the further reduction in hemicellulose and lignin concentrations, reason for the decrease in cellulose yield could be because during NaOH/NaClO₂ treatments, some glycosidic linkages were broken, which caused the polysaccharides to de-polymerize (Rani *et al.*, 2018).



Fig 5.1 Representation of extracted cellulose in powder form.

5.2 CHARACTERIZATION:

5.2.1 ATR – FTIR ANALYSIS OF CORNCOB:

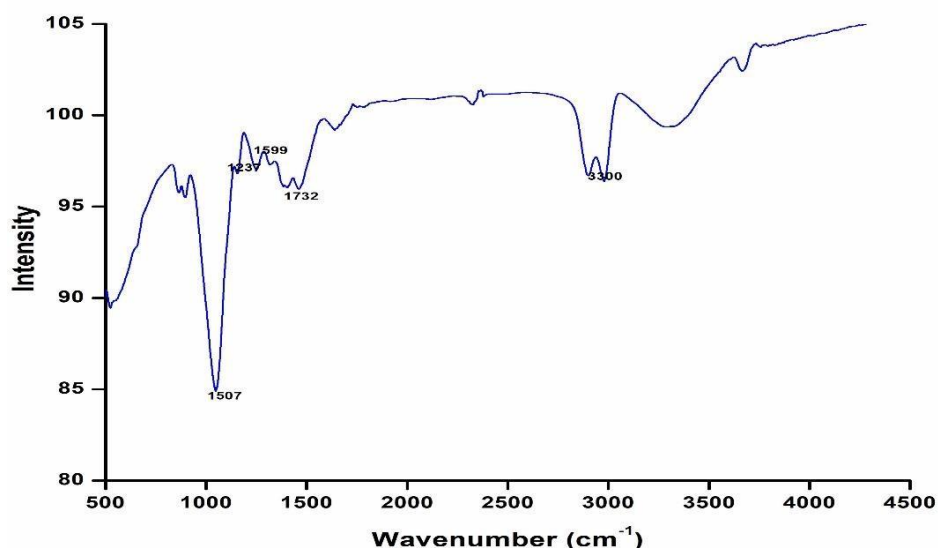


Fig 5.2 Representation of ATR-FTIR graph of corncob cellulose.

After the extraction process, the chemical composition and structural alterations of the corn cob were observed using FTIR spectroscopy. The hemicellulose's acetyl and ester groups, is an indicator of the ester connections of the carboxylic groups of the ferric and p-coumaric acids of lignin and hemicellulose are indicated by the peak in the corn cob's spectrum at 1732 cm^{-1} . (Rahimi Kord Sofla *et al.*, 2016). After being treated with alkali, the peak eventually vanished. The C = C stretching vibrations of the aromatic rings of lignin are responsible for the absorption band that was detected at 1507 cm^{-1} in the treated plant sample. The structure's peak, measuring 1237 cm^{-1} , coincides with the ether, ester, and phenol groups of lignin's C-O axial asymmetric strain(Shahi *et al.*, 2020).The methyl, methylene, and methoxy groups of lignin exhibited C-H deformation, which reflected in the absorption bands at 1599 and 1455 cm^{-1} . Since the peaks were absent from the alkaline-treated corncob's FT-IR spectra, these results lead us to the conclusion that

the alkali pretreatments significantly reduced the quantity of lignin and hemicellulose present in the corncob structure(Poornachandhra *et al.*, 2023a).

5.2.2 ATR – FTIR ANALYSIS OF SUGARCANE BAGASSE:

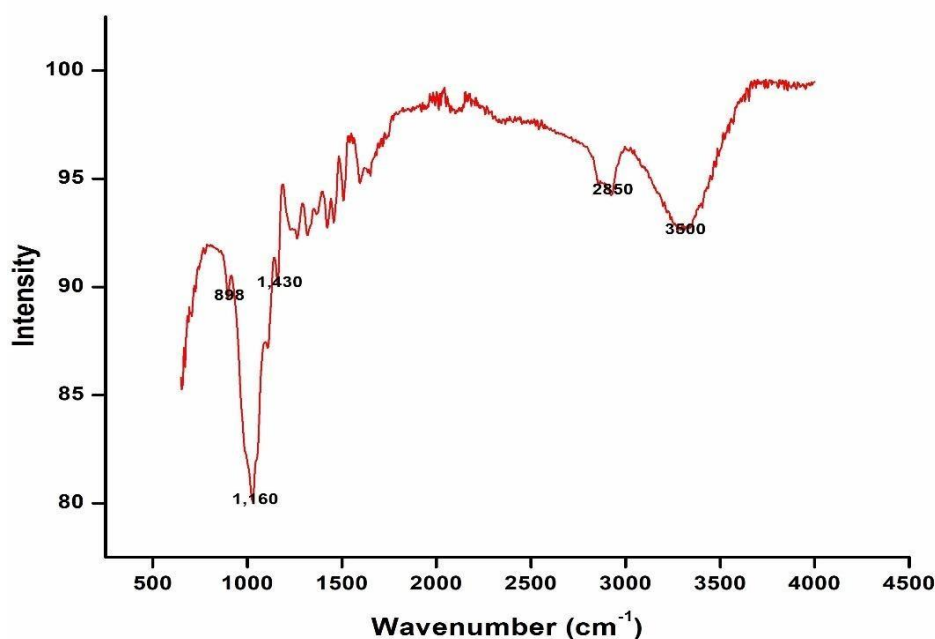


Fig 5.3 Representation of ATR-FTIR graph of sugarcane bagasse cellulose.

From the FTIR analysis of cellulose extracted sugarcane bagasse has a peak at 3200 – 3500cm⁻¹ which indicates O-H stretching vibrations in cellulose molecules and it displays the hydrophilic tendencies of the cellulose. The band between 2850-3000cm⁻¹ indicates the C-H asymmetric stretching due to alkyl groups in the aliphatic bonds of cellulose(Rani *et al.*, 2018). The stretching of the C–O–C glycosidic band was reflected by the peaks at 1,160cm⁻¹. A tiny peak at 1,037cm⁻¹ is ascribed to the C-O-C ether linkage from trace amounts of leftover lignin or hemicelluloses, whereas the C-O-C pyranose ring skeleton is responsible for the peak at 1,054 cm⁻¹(Rahimi Kord Sofla *et al.*, 2016). Peak at 898 cm was attributed to cellulose's b-glycosidic bond. The amorphous and crystalline portions of cellulose linked to cellulosic b-glycosidic connections are represented by two peaks

positioned at $1,430$ and 898cm^{-1} , respectively. These peaks were more pronounced upon the removal of lignin and hemicelluloses(Shahi *et al.*, 2020). Because the majority of the hemicelluloses and lignin were removed during the chemical treatment, this peak vanished entirely. The absence of the peaks at $1,252$ and $1,514\text{cm}^{-1}$ in the cellulose nanofiber spectrum suggests that the majority of the lignin was eliminated and pure cellulose was separated(Rani *et al.*, 2018). The usual structure of cellulose is indicated by the growth of the band at 898 cm in spectra derived from chemically treated samples, whereas the C–O–H out-of-plane bending mode in cellulose is represented by the peak at 670cm^{-1} (Mzimela *et al.*, 2018).

5.2.3 ATR – FTIR ANALYSIS OF COCONUT COIR:

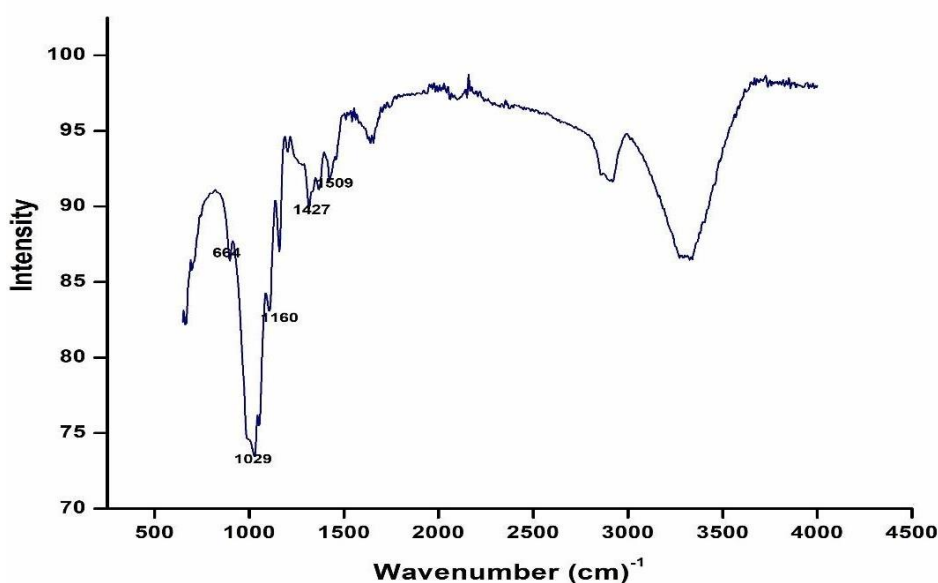


Fig 5.4 Representation of ATR-FTIR graph of coconut coir cellulose.

The peaks at $1,509$ and $1,267\text{cm}^{-1}$ are attributed to C=C aromatic skeletal vibration of lignin and C–O stretching of hemicellulose and lignin, respectively. Treatment reduced lignin and hemicellulose effectively as seen by the elimination of vibrations from standard peaks of coconut coir at $1,720$, $1,509$, and $1,267\text{cm}^{-1}$. The decrease of intensity peaks at $1,720$ and $1,267\text{cm}^{-1}$ was evidence that some lignin and hemicellulose can be removed by increasing time of the NaClO treatment(Nang An *et al.*, 2020).The spectra showed that the delignification time affected the removal

of hemicellulose and lignin. The characteristic peak at 1029cm^{-1} and 1427cm^{-1} in the cellulose spectrum represents the C-O stretching of cellulose. Furthermore, cellulose's C-H deformations and C-O-C stretching vibrations are between 898 cm^{-1} to 1160 cm^{-1} peaks, respectively(Poornachandhra *et al.*, 2023a). The peak at 1160 cm^{-1} indicated the presence of a sulfated group (SO_2) in the spectra, which was most likely brought on by cellulose sulfonation during the acid hydrolysis process using sulfuric acid. The existence of OH bending from absorbed water and C-OH out-of-plane bending in cellulose are indicated by the bands at 664 cm^{-1} and 1630 cm^{-1} , respectively(Nang An *et al.*, 2020). Additionally, as described in his paper, acid hydrolysis revealed the hydroxyl groups bound by intermolecular hydrogen bonding in cellulose, as evidenced by the broadband at 3330cm^{-1} (Nang An *et al.*, 2020). This intermolecular ($\text{O}(2)\text{H}---\text{O}(6)$ and $\text{O}(3)\text{H}---\text{O}(5)$) and intramolecular ($\text{O}(6)\text{H}---\text{O}(3')$) hydrogen bonding, which was caused by the presence of pendant hydroxyl groups, determines the strength and crystallinity of cellulose. Because hydroxyl groups have the potential to quicken the curing of epoxy, cellulose derived from coir fibre is an excellent choice for use in emulsions. As a result, the FTIR analysis showed that the fibers amount of binding components was decreased after the acid and alkali treatments(Poornachandhra *et al.*, 2023a).

5.2.4 XRD ANALYSIS OF CORN COB:

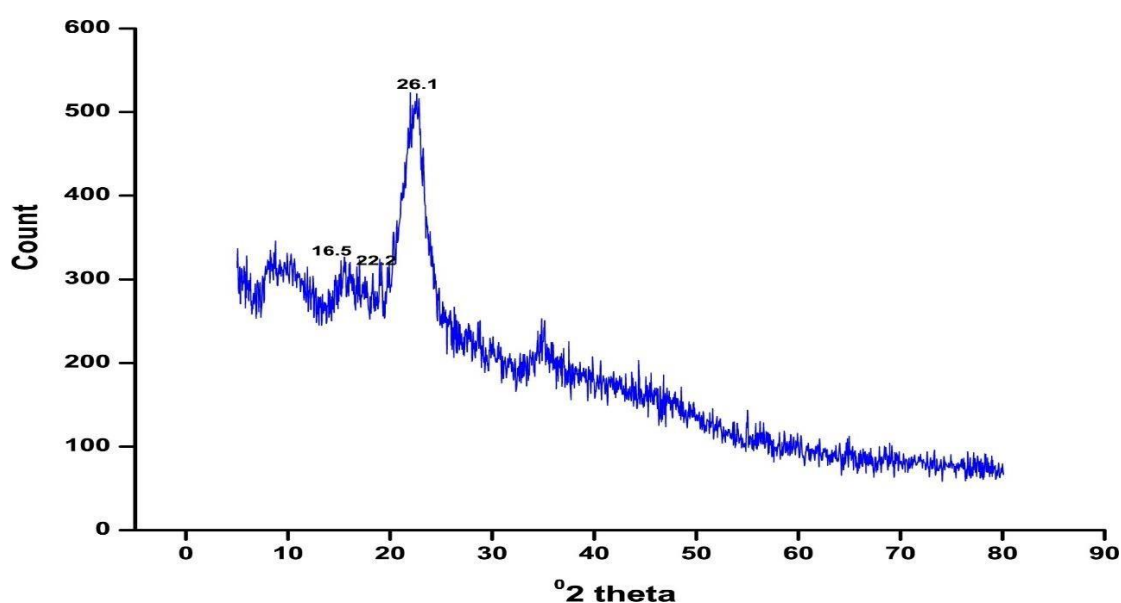


Fig 5.5 Representation of XRD graph of corn cob cellulose.

Figure 5.5 displays the cellulose and corn cob XRD graphs. It was discovered that the crystallinity varied with the physical and chemical conditions used. The cellulose I structure of these samples was typical, exhibiting a broad peak at approximately $2\theta = 16.5^\circ$, 22.5° , and 34.6° . Specifically, cellulose crystallinity has been discovered to be connected with fibre elasticity, thermal stability, and absorption capacity—all crucial properties for industrial applications. Our results are similar to (POORNACHANDHRA *et al.*, 2023a).

5.2.5 XRD ANALYSIS OF SUGARCANE BAGASSE:

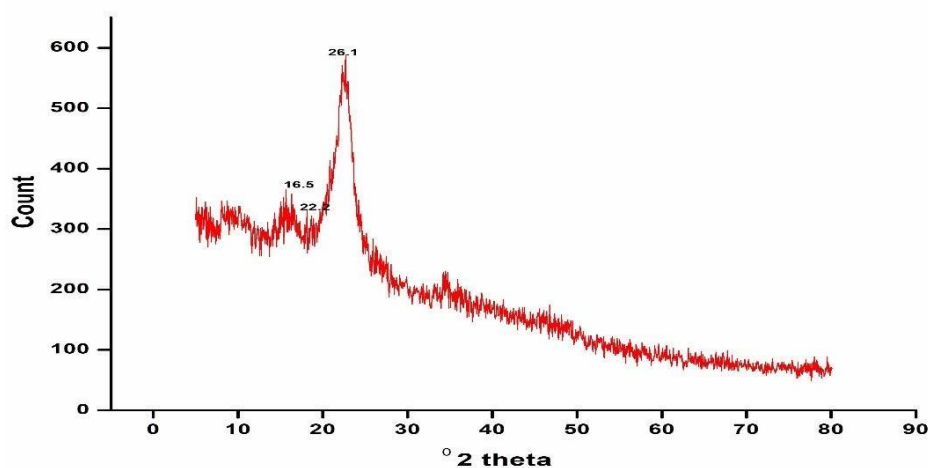


Fig 5.6 Representation of XRD graph of sugarcane bagasse cellulose

Extremely distinct peaks that roughly correspond to a typical cellulose structure at $2\theta = 12.5^\circ$ and $2\theta = 22.5^\circ$ (Mzimela *et al.*, 2018). The primary 2θ diffractions, which are connected to the crystalline cellulose, were found near 16.5° , 26.1° , and 22.2° . The XRD data indicated that the crystalline morphology of cellulose did not change during treatment, but the crystallinity of the fibre was improved over 30% of 264 of SCB-raw. Our results are similar to (RANI *et al.*, 2018).

5.2.6 XRD ANALYSIS OF COCONUT COIR:

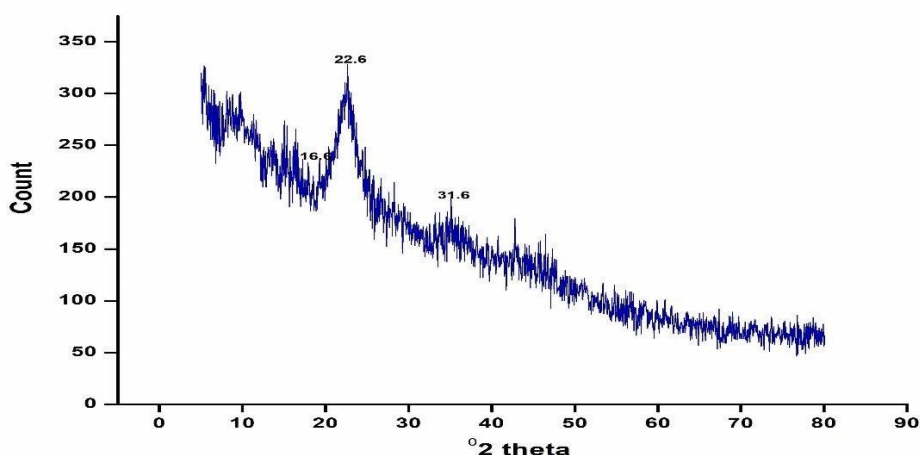


Fig 5.7 Representation of XRD graph of coconut coir cellulose.

The mechanical and thermal properties of cellulose are influenced by its crystalline nature. The produced cellulose's XRD diffraction patterns were obtained at $2\theta = 16.6^\circ$, 22.6° , and 34.6° . The presence of crystalline cellulose was confirmed by the strong crystalline peak at 22.6° with 100% intensity(Nang An *et al.*, 2020). Raw fibre contains a matrix of amorphous lignin, pectin, hemicellulose, and other materials in which components of nanocrystalline cellulose are embedded. During the alkali treatment, the matrix components interacted with sodium hydroxide and started to break down. The high temperature will facilitate the alkali's entry into the interior portion of the raw coir by defibrillating the fibre. These amorphous chemicals and other contaminants were eliminated during the alkali pretreatment and bleaching process, increasing the crystallinity of cellulose(Rani *et al.*, 2018). The more prominent peaks in these samples may be explained by greater molecular hydrogen bonding leading to higher crystallinity. Previous research suggested that the sulfate groups on the cellulose surface would give off a negative charge, which would cause electrostatic repulsion between the individual crystals. This aids in the formation of stable, homogenous cellulose aqueous solutions(Nang An *et al.*, 2020). This suggested that decreasing hydrogen bond formation during the acid hydrolysis process of cellulose synthesis would affect the highly structured crystallites. The degree of hydrogen bonding and compact packing in the produced cellulose can be

related to its high crystallinity index. The XRD graph shows that cellulose modified with sulfuric acid produced well-oriented crystallites(Poornachandhra *et al.*, 2023a).

5.3 DISSOLUTION OF CELLULOSE USING ZINC CHLORIDE:

From the above process cellulose was dissolved and ZnCl_2 solution is obtained. The ZnCl_2 method mechanism may allow Zn^{2+} ions to enter cellulose's interior structure, weakening hydrogen bonds between molecules and upsetting the crystalline area. The insoluble cellulose is filtered out of the partially dissolved cellulose in the gel solution. There are three ways that temperature affects the characteristics of cellulose dissolution. First, cellulose reacts endothermically because of its stronger hydrogen bonds, hence a higher temperature may promote the dissolution of cellulose(Chen, *et al.*, 2020). Second, Zn^{2+} and Cl^- ion thermal transfer may become more severe at high temperatures. This might make it easier for cellulose fibre to clump together with solvents at high temperatures, adding more energy to the resolving process. Third, the solution's viscosity can be decreased at a high temperature, allowing for more uniform distribution cellulose in ZnCl_2 solution(Xiong *et al.*, 2016).



Fig 5.8 Representation of dissolved cellulose in zinc chloride.

5.4 POLYMER BLENDING WITH CHITOSAN AND CELLULOSE SOLUTION

In order to improve the mechanical properties of bioplastics, chitosan and cellulose were added during the manufacture process of bioplastic film. Variations in cellulose

are used to improve the bioplastic film's tensile strength and percent elongation. Cellulose molecules form long, linear chains and are prone to forming hydrogen bonds both inside and between molecules. The tensile strength and percent elongation of bioplastic films are significantly impacted by hydrogen bonds, which are produced from chitosan and cellulose(Chadijah *et al.*,2018). Then PVA polymer is incorporated with cellulose for Strong hydrogen bond interactions between the cellulose and PVA matrix are largely responsible for the enhanced mechanical capabilities of cellulose/PVA/chitosan composites. Because cellulose has a higher degree of crystallinity than PVA(Wu *et al.*, 2019), it has a higher thermal stability than PVA, which means that the overall thermal stability of composites might be effectively increased. When the results are compared to research, they show that all can be released intact and are flexible. However, there is a difference in the colour of the bioplastic film since in the study, bleach was applied, making the film white(Wu *et al.*, 2019).

5.5 STANDARDIZATION:

The cellulose blended with polymer and chitosan are prepared at different ratio and casted. From the ratios 5:5,6:4,7:3 and 8:2. The 6:4 ratio of cellulose solution with chitosan and polymer gives better results. After standardization of ratio, bioplastic prepared with each individual cellulose (corn cob, sugarcane bagasse, coconut coir) and with the combination three cellulose in equal amount.



Fig 5.9 Representation of mixed cellulose bioplastic

5.6 COMPARATIVE ANALYSIS BETWEEN INDIVIDUAL CELLULOSE AND MIXED CELLULOSE BASED BIOPLASTIC:

The swelling test performed to compare the swelling property of each bioplastic for 2-10hrs.

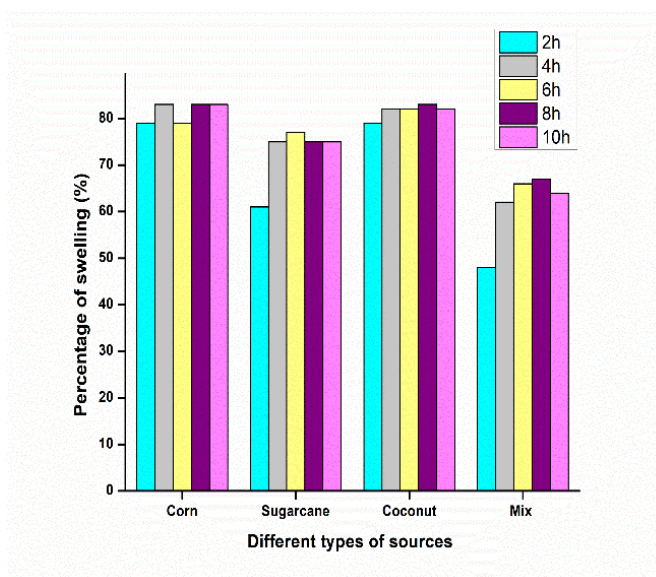


Fig 5.10 Graphical Representation of swelling test for result for comparative analysis.

From the comparative analysis the swelling percentage of mixed cellulose bioplastic is lesser when compared to the other bioplastics.

5.7 CHARACTERIZATION FOR MIXED CELLULOSE BIOPLASTIC:

5.7.1 WATER CONTACT ANGLE:

An equal amount of PVA and chitosan concentration added to the cellulose bioplastic increased the free volume between the cellulose chains. The increment of the formed cross-links hindered the formation of crystalline regions of the cellulose chains. Therefore, more hydroxyl groups were exposed to interact with water and lower the contact angle value The percentage of water solubility of the cellulose bioplastics. As the concentration of Polymer increased, the cellulose bioplastic became more soluble in water. This was due to the lower crystallinity of cellulose structure generated at the sample with higher polymer concentrations. Due to the lower usage of polymer, it prevented the solubility of the cellulose bioplastic in water. Therefore, it also increased the water uptake of cellulose bioplastic(Steven *et al.*, 2022).

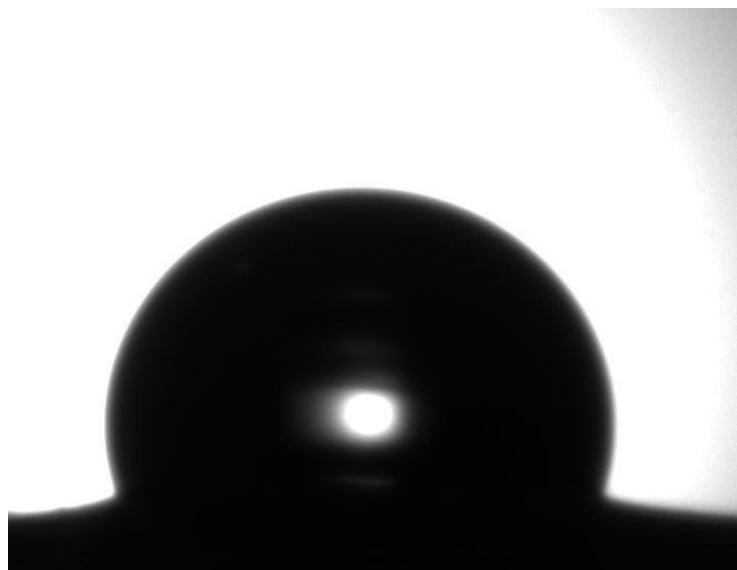


Fig 5.11 Representation of water contact angle for mixed cellulose bioplastic

5.8 TEST FOR MIXED CELLULOSE BIOPLASTIC:

5.8.1 TEST FOR SOLUBILITY IN SOLVENT:

Mixed cellulose bioplastic is placed in different solvents and kept for 24hrs to check its solubility rate,

TABLE 5.1 for solubility in solvent

	ACETONE	ETHANOL	CHLOROFORM	METHANOL
INSOLUBLE	-	-	-	-
PARTIALLY SOLUBLE		✓		✓
SOLUBLE	✓		✓	

After the 24hrs the bioplastic in methanol and acetone are completely dissolved where as in ethanol and chloroform the bioplastic is partially dissolved. The partially dissolving and completely dissolving of bioplastic property show the biodegradability of bioplastic (Yaradoddi *et al.*, 2020).

5.8.2 OIL PERMEABILITY TEST:

Oil permeability of the film, is concerned with the quantity of the lipophilic groups existing in the composite film. The film with the correct permeability is more appropriate in deciding the suitability of the material for packing(Yaradoddi et al., 2020). The film will have better oil and water retention properties relating to its thickness.

$$P_0 = (\Delta W \times FT) / (S \times T),$$

used to determine the oil permeability coefficient. The oil permeability of mixed cellulose bioplastic is $P_0 = 0.011$. The oil permeability of coefficient should be less than one to effectively prevent oil from passing through. A coefficient greater than one would indicate that the bioplastic allows more oil to permeate through it, which could lead to leakage and contamination issue.

After discussing and analyzing the results, ATR-FTIR indicates the presence of bands representing functional groups, confirming the existence of cellulose. Additionally, XRD results show that the crystalline structure of cellulose remains unchanged after chemical treatments. Cellulose dissolution using the zinc chloride method yields superior results compared to the NaOH/urea and DMSO methods. The cellulose solution is blended with polymer and chitosan to enhance elasticity and mechanical strength. The bioplastic is then standardized at a 6:4 ratio after evaluating various ratios. Comparative analysis is conducted among each cellulose-based bioplastic and three mixed cellulose-based bioplastics using a swelling test. The standardized mixed cellulose-based bioplastic is further characterized through water contact angle measurement and tests for solubility in solvents, as well as oil permeability. These findings underscore the importance of excelling globally with strong fundamentals and domain knowledge, coupled with updated modern technology, to effectively address technological challenges. Moreover, they emphasize the significance of considering environmental impacts and promoting sustainability in the development and application of bioplastic materials.

CHAPTER – 6

SUMMARY AND CONCLUSION

In conclusion, this study addresses the immediate need for sustainable alternatives to conventional petroleum-derived plastics by delving into the realm of cellulose-based bioplastics. Through a comprehensive investigation, we have examined the source, characteristics, sustainability aspects, progress in development, and practical applications of cellulose bioplastics. Our research underscores the importance of biodegradability and compostability in combating plastic pollution and promoting a circular economy. By utilizing cellulose extracted from natural residues such as sugarcane bagasse, coconut coir, and corn cob, and employing alkaline and acid treatments, we have demonstrated the feasibility of producing cellulose bioplastics. Characterization techniques including ATR-FTIR and XRD confirmed the presence of essential functional groups in the cellulose structure and crystallinity. The utilization of the zinc chloride method for cellulose dissolution and blending with polymers and chitosan resulted in the formation of a gel-like structure, paving the way for practical applications. Through various testing and analysis, including standardization and characterization, we have contributed to the advancement of cellulose-based bioplastics as viable eco-friendly alternatives. This research not only expands the understanding of bioplastic materials but also highlights the potential for environmentally sustainable solutions in mitigating plastic waste and promoting a greener future.

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Name of the student : Rakesh S

Register Number 40230038

Title of the Thesis : Cellulose based bioplastic: Advancement and future prospects

Internal Guide Name : Dr. V. Ramesh Kumar

External Guide Name : Dr. D. Inbakandan

PO / PSO / PEO / SDG	Justification
PO7	Reduction of agricultural residue such as sugarcane bagasse, corn cob and coconut coir for extraction of cellulose and replacement of petroleum-based plastic by cellulose based bioplastic which leads to achieving environmental sustainability.
PSO3	Extraction of cellulose using bioprocess techniques such as pre alkaline, alkaline with steam explosion treatment. This enables efficient extraction of cellulose for preparation of bio plastic.
PEO1	Cellulose-based bioplastics offer sustainable alternatives to petroleum-based plastics, aligning with global goals for technological excellence. Leveraging strong fundamentals and domain knowledge, modern technologies enhance their properties and applications. By staying updated with advancements, these bioplastics provide effective solutions for environmental and technological challenges. Embracing cellulose-based bioplastics drives innovation towards a more sustainable and resilient future.
SDG15	Cellulose-based bioplastics promote land sustainability by reducing reliance on petroleum plastics. Derived from plant sources, they offer eco-friendly alternatives across industries. This shift minimizes habitat degradation and fosters biodiversity conservation. Embracing cellulose-based bioplastics supports a greener future for life on land.

Your GATE 2024 Result (BT)

Name

RAKESH S

Registration Number

BT24057402296

Gender

Male

Parent's/Guardian's name

SENTHILKUMAR

Date of Birth (YYYY-MM-DD)

2003-08-04

Examination Paper

Biotechnology (BT)

Your GATE 2024 Result (BT)



Photograph



Signature

Marks out of 100⁸

14

Qualifying Marks⁸⁸

36.9

36

35.9

General

OBC-NCL/STB

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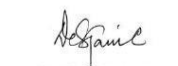
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IIT Kharagpur

Jul-Oct 2021
(12 week course)


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