Extraction of cellulosic Materials from enset fiber agro industrial residues: Application for polyvinyl chloride (PVC) composite materials development

Sina Tesfaye Kerga

A Thesis Submitted to Department of Materials Science and Engineering

School of Mechanical, Chemical and Materials Engineering

presented in partial fulfilment for the requirements for the Degree of Master's in Materials Science and Engineering

Office of Graduate Studies

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Adama, Ethiopia

Extraction of cellulosic Materials from enset fiber agro industrial residues: Application for polyvinyl chloride (PVC) composite materials development

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Declaration

I declare that this Master Thesis entitled "**Extraction of cellulosic Materials from enset fiber agro industrial residues: Application for polyvinyl chloride (PVC) composite materials development**" is my own work and has not been submitted to any university for similar purpose. The references used in this proposal are duly recognized by proper citations.

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I/we hereby certify that the recommendation and suggestion given by the thesis review committee are appropriately incorporated into the final thesis/dissertation proposal entitled “Extraction of cellulosic Materials from enset fiber agro industrial residues: Application for polyvinyl chloride (PVC) composite materials development” by Sina Tesfaye.

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We, the undersigned, members of the Board of Reviewers of the thesis open defense by Sina Tesfaye have read and evaluated the thesis/dissertation proposal entitled “Extraction of cellulosic Materials from enset fiber agro industrial residues: Application for polyvinyl chloride (PVC) composite materials development” and assessed the understanding of the candidate about the proposed research. This is, therefore, to certify that the thesis/dissertation proposal is accepted and we recommend the implementation of the thesis.

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LIST OF ACRONYMS

FTIR	Fourier transforms infrared spectroscopy
ENC	Enset nano cellulose
SEM	Scanning electron microscope
PET	Poly ethylene terephthalate
TGA	Thermal Gravimetric Analysis
XRD	X-ray diffraction
PVC	Poly vinyl chloride
CMF	Cellulose microfibers
CNF	Cellulose nanofibers

ABSTRACT

Polyvinyl chloride (PVC) and nanocellulose have been identified as promising candidates for developing sustainable composite materials. This study explores the reinforcement effects of integrating nanocellulose into PVC matrices, with a focus on improving mechanical properties, biodegradability, and overall environmental sustainability. Enset nanocellulose (ENC), obtained from residue sources, was incorporated into the PVC matrix at varying concentrations to assess its impact on the composite's mechanical performance and biodegradability.

The methodology involved extracting cellulose from enset fibers and preparing ENC through hydrolysis. Composite materials were fabricated using a solution casting technique with ENC content ranging from 5% to 25%. Various analytical techniques, including scanning electron microscopy (SEM), X-ray diffraction (XRD), thermogravimetric analysis (TGA), and Fourier-transform infrared spectroscopy (FTIR), were employed to evaluate dispersion, interaction, thermal stability, and functional group identification. Mechanical performance was assessed via tensile testing, and biodegradability tests were conducted to determine the degradation rate under different environmental conditions.

Results showed that incorporating nanocellulose significantly enhanced the mechanical properties and biodegradability of PVC composites. Improved tensile strength and faster biodegradation rates were observed, confirming the reinforcing effect of nanocellulose and its potential for environmentally friendly applications. SEM and XRD analyses indicated strong interfacial adhesion between nanocellulose and PVC.

This research contributes to the ongoing efforts in the development of sustainable composite materials by leveraging the unique properties of PVC and nanocellulose. The findings underscore the potential of PVC/nanocellulose composite packaging applications where both performance and environmental considerations are significant.

Keywords: **Composite material, Polyvinylchloride (PVC) matrix, Enset, Enset NanoCellulose (ENC)**

CHAPTER ONE

1. INTORDUCTION

1.1Background

Polyvinyl chloride (PVC) is the oldest thermoplastic polymer, and it is a significant product in the chemical sector. It is highly resistant to acids, alkalies, salts, alcohols, vents, oxidants, and paraffinic solvents, making it an inexpensive and simple-to-assemble construction material. It has the greatest range of application, and its use has increased more quickly than that of other plastics for a number of reasons, apart from the cost of the raw materials. This is due to PVC's extended lifespan and ease of fabrication. Compared to most other thermoplastics, PVC offers superior strength,rigidity, and exceptional chemical resistance to a broad variety of corrosive fluids. It also responds differently to functional additives, allowing for the production of materials that are both flexible and rigid and helpful in specified engineering applications (Venkatachalam et al., 2023). Because of its special qualities and adaptability, PVC is a widely used packaging material. Each year, Europe uses more than 400,000 tons of PVC for packaging. Its barrier qualities assist preservation,while its light weight lowers transportation-related emissions. It is safer because it resists breaking, transparent and clear enough to be shaped into a variety of designs (Shaikh et al., 2021).

PVC is still extensively utilized, despite claims that its manufacture has an unhealthy effect on the environment and public health (Galloway et al., 1991). PVC had several environment and safety concerns. Vinyl chloride, or VC, may be extremely harmful to people’s health. Early in the 1930s, research discovered that VC has aesthetic properties, which they have since explored. It may quickly kill animal at concentrations considerably higher than 8 to 12%. Excessive VC inhalation destroys the liver's capacity, which encourages the growth of tumours. According to (Van Hassel et al., 2024), it is also a carcinogen and also thought to be a pollutant. It creates certain chemicals during processing, or decomposition, such as dioxins and hydrogen chloride, which might harm the atmosphere. Environmental groups have expressed concern of its widespread use due to these difficulties (Silva et al., 2024).

Globally, the amount of plastic garbage is increasing due to changes in people's production and consumption patterns and economic development.

The toxicity and non-biodegradable nature of plastic items make the impact they have on ecological systems and the human population extremely serious due to the massive volume of plastic consumption and trash created in the environment. This is why it is necessary to eliminate plastic garbage from the environment right away. Burning, reusing, or recycling are some more methods for getting rid of these plastics. Because of the drawbacks, disposing of plastic garbage by burning and recycling methods is not a desirable solution(Kalali et al., 2023).

Researchers are very interested in removing plastic waste from the environment and in discovering new source of raw materials with mechanical and physical qualities to those of synthetic fibers. As a result, the product of plastic composites reinforced with natural fiber has expanded dramatically in the modern era. Matrix properties have been applied to high-density polyethylene (HDPE), low-density polyethylene (LDPE), polypropylene (PP), polyvinyl chloride (PVC), and other plastic polymers (Barkoula et al., 2010). The well-known benefits of polymeric matrix composites (PMCs) are their low cost and straightforward production procedure. Affordable goods may be produced by manufacturers of natural fiber reinforced plastic composites (NFPCs) using various production procedures.

The combination of PVC and natural fibers offers a promising alternative, minimizing drawbacks while preserving advantages. Natural fibers are those that have not undergone chemical modification and source from minerals, plants, or animals. Natural fibers are cost-effective, biodegradable, and green, making them popular as reinforcing fillers in composite material (Arif et al., 2021).

This study focuses on the extraction of nanocellulose from enset fiber and used as reinforcement in composite materials. A solution casting method was used to produce nanocellulose- reinforced PVC nanocomposites with weight percentages ranging from 5% to 25% enset nanocellulose in the range of 5%.SEM, XRD, FTIR, TGA, biodegradability and tensile test were used to analyses biodegradability, physical and mechanical property of composites and fiber, respectively. The nanocomposites showed strong interfacial bonding, homogeneous nanocellulose dispersion, and improved thermal stability, suggesting their potential for the commercial market of packaging.

1.2 Statement of the problem

Plastic pollution has emerged as one of the most critical global challenges due to its long-lasting environmental impact. It can take hundreds of years for plastics to decompose, during which they contaminate soil, water, and air, posing significant risks to both human health and ecosystems. In Ethiopia, the increasing investment in plastic manufacturing and importation has exacerbated the problem. Although some plastics are recycled, the majority are not, leading to a rapid accumulation of non-degradable waste that contributes to widespread pollution. The persistent nature of plastics in the environment results in significant ecological damage, such as the ingestion of plastic particles by wildlife, which can lead to poisoning and death (Keller & Dexter, 2012).

Moreover, plastics, including polyvinyl chloride (PVC), are derived from non-renewable petroleum resources. The extraction and processing of these resources contribute to environmental degradation and greenhouse gas emissions. PVC production is associated with the release of toxic chemicals such as dioxins and vinyl chloride, which pose serious health risks to humans, including carcinogenic effects. The non-biodegradable nature of traditional plastics like PVC means that they accumulate in landfills and natural environments, where they persist for centuries, creating an ever-growing waste management problem(Grosu, 2022).

Enset, a crucial crop in Ethiopia, is highly resilient and adaptable, making it essential for regional food security. Enset fiber, primarily composed of cellulose, hemicellulose, and lignin, is ideal for producing nanocellulose, which can be used to produce high-performance, sustainable composite materials. Nanocomposites, with their enhanced properties, are being developed as a novel packaging material due to their potential applications in various industries, including reinforcing materials, paper, food additives, cosmetics, and medical devices (Abdalkarim et al., 2021).

This thesis explores the use of enset fiber residues as a source of nanocellulose, a novel approach to transforming agricultural waste into sustainable composite materials. The study aims to enhance the mechanical properties and biodegradability of these composites, addressing environmental concerns and improving material performance. The successful extraction and application of enset nanocellulose in PVC composites provide a new pathway for waste valorization and the development of eco-friendly composite materials, thereby reducing the environmental impact of agricultural waste.

1.3 Objectives

1.3.1 General objective

The general objective of this study is extraction of cellulosic materials from enset fiber agro industrial residues:application for polyvinyl chloride (PVC) composite materials development.

1.3.2 Specific objectives

- ✓ To isolate cellulose microfibers (CMF) and cellulose nanofibrils (CNF) from enset fiber
- ✓ To develop composite materials based on PVC polymer matrix
- ✓ To analyze morphology, biodegradability, thermal stability, and mechanical property of extracted samples.

1.4 Significance of the study

Primarily extraction of cellulosic materials from enset fiber agro-industrial residues preferred for environmental restoration processes with its attractiveness as a sustainable source of biomaterials comes mainly from their low-cost, availability and chemical composition using of this residue.

This research will allow our country to use our widely and sustainably available enset fiber for packaging applications by combining it with plastic products. It has benefits such as a reduction in greenhouse gas discharges, non-degradation properties, and nonrenewable sources like oil and gas, while enhancing mechanical and physical properties. There will be possibilities to reduce environmental pollution and keep the environment safe from landfill waste. Furthermore, it offers an income for many people and families in developing countries, both in the form of formal employment and informal economic activities. Nowadays, there are already campaigns on plastic waste, yet the problem of plastic waste is still pressing worldwide. Additionally, to extract cellulose and nanocellulose, which have a good yield of cellulose, and study their significant properties when synthesizing PVC composites.

Therefore, we can say that in addition to providing new information in this field, it holds significant implications by utilizing abundant natural resources, reducing land waste, minimizing resource wastage, lowering product costs, and addressing environmental issues, all while enhancing the properties of PVC.

1.5 Scope of the study

This study aims to explore the extraction of cellulosic materials from enset agro-industrial residues, a significant agricultural crop, for the development of polyvinyl chloride (PVC) composite materials for packaging purposes. The research will focus on optimizing fiber quality and yield through various extraction techniques. The extracted cellulosic materials will be characterized by their physical, chemical, and morphological properties to determine their suitability as fillers or reinforcements in PVC-based packaging composites. Specific formulations will be developed for packaging applications, considering factors like fiber content, size and chemical treatment with PVC matrix to enhance mechanical strength, thermal stability, and mechanical properties. Environmental impact assessment will evaluate the sustainability of enset residues as a renewable resource for packaging materials and potential reductions in agricultural waste. The study aims to advance sustainable materials development by effectively using enset agro-industrial residues, enhancing the performance, biodegradability, and eco-friendliness of PVC composite materials for packaging applications

CHAPTER TWO

2. LITERATURE REVIEW

2.1. Composite material

Composite is a substance composed of two or more distinct phase with bulk properties which is significantly distinct from of any of its component parts. It refers to the combination of the two materials in which the resultant traits are better than those of each individual components when using separately, for example. This type of composite material is made up of two constituents: the matrix and the reinforcement. High strength and stiffness, as well as low density that reduces weight in the final product, are the advantages of composite materials over other available materials such as metal, plastic, acting as a matrix (Saxena et al.,2011).

Metals, ceramics, or polymers can be used for the matrix phase while fibers, particles, or other fillers can be used for the reinforcing phase. Reinforcements are used in the manufacturing of components that require excellent wear resistance, and softer reinforcements like graphite and molybdenum are used to obtain characteristics like lubrication. The characteristics of composite materials are determined by the reinforcements and the matrix, which serves to maintain the reinforcement particles in place as well as give them shape and support. In general, reinforcements affect mechanical and physical characteristics or any other tailored characteristics improved from the matrix material. A wide range of characteristics can be acquired by combining many different possible reinforcements and matrix, to alter material characteristics to meet specific requirements (Baillie & Jayasinghe,).

There are several types of composite materials based on the matrix used. This includes Polymer Matrix Composites (PMCs), Metal Matrix Composites (MMCs), Ceramic Matrix Composites (CMCs), Fiber Reinforced Composites and Particulate Composites.

2.1.1. Metal Matrix Composite Materials

The metallic composites (aluminium/boron fibers and aluminium/carbon fibers) are also the final (Sauer et al., 2013). Packaging, automotive, light structures, civil engineering, aviation, sports, biomedicine, thermos mechanical components, and aerospace are just a few of the application areas that are impacted by these composite materials. Metal matrix composites (MMCs) consist of a metal or an alloy as the continuous matrix and a reinforcement that can be particle, short fiber or whisker, or continuous fiber.

2.1.2 Ceramic Matrix Composite Materials

Ceramic Matrix Composite (CMC) is a composite material with ceramics as a matrix material. These materials offer high strength, stiffness, chemical inertness, and low density. They are brittle and hard, often mixed with non-metals like oxygen, carbon, or nitrogen (Fenetaud & Jacques, 2023). They have low fracture energy, low toughness, and low thermal and mechanical shock resistance. However, they are brittle, fragile, and can resist high temperatures. Even small internal faults or surface imperfections can negatively affect CMC (Chang et al., 2023).

2.1.3. Polymer Matrix Composite Materials

A polymer matrix composite (PMC) is a composite material composed of a variety of short or continuous fibers bound together by an organic polymer matrix. PMCs are designed to transfer loads between fibers of a matrix. Some of the advantages of PMCs include their lightweight, high stiffness, and their high strength along the direction of their reinforcements. Other advantages are good abrasion resistance and good corrosion resistance (Dagdag et al., 2919; Khalil et al. 2013). Polymers that are often used as composites are thermoplastic polymers, thermosetting polymers, or elastomers. Polymers, which are a source of a wide variety of low-priced raw materials, offer many advantages these include low specific weight, enhanced stability against corrosion, improved electrical and thermal insulation, ease of shaping and economic mass production, and attractive optical properties, e.g. fiber optics, glazing applications, etc(Amrollahi et al., 2019).

However, they have some serious shortcomings: exhibiting quite often poor mechanical stiffness and strength, and poor resistance against heat being sensitive to aging i.e. change of the physical, chemical, and mechanical properties by light, heat, oxygen, and moisture being combustible exhibiting large values of coefficient of thermal expansion, which can generate high levels of internal frozen-in stresses.

Polymer matrix composites are classified based on their level of strength and stiffness into two distinct types which is reinforced and advanced composites.Reinforced plastics confers additional strength by adding embedded fibrous matter into plastics composites consists of fiber and matrix combinations that facilitate strength and superior stiffness. They mostly contain high-performance continuous fibers such as high-stiffness glass (S-glass), graphite, aramid, or other organic fibers (Ahmadijokani et al., 2020; Arabpour et al., 2020).

Compared with conventional materials, composites offer: lightweight, high specific strength and stiffness, high toughness (impact strength), damping ability (attenuates noise and vibrations/shocks), high fatigue resistance (improves fatigue life), corrosion resistance, low and controllable coefficient of thermal expansion (CTE) (i.e. good dimensional stability), ablative shielding (El Gouri et al., 2009).

2.1.3 Thermoplastic matrix in natural fiber Composites

A wide variety of matrices and processing techniques may be used to produce composites. Thermoset matrix, thermoplastic matrix, and biodegradable matrix are the three general categories into which matrix used for natural fiber-reinforced systems fall. Thermoplastic polymers are the most widely used matrix in natural fiber-reinforced polymer composites. Polymers made of plastic that can be melted at high temperatures, solidified at low temperatures, and molded or reshaped under certain circumstances are called thermoplastics. In comparison to thermoplastics, thermoset polymers are less commonly used because of their more expensive manufacturing costs and complex curing requirements (Mohanty et al., 2005; Sreekumar & Thomas, 2008). PLA, polyvinyl chloride(PVC), polypropylene (PP), polyethylene (PE) - (HDPE & LDPE), and polyethylene (PE) are common thermoplastic matrix materials used in natural fiber reinforced polymer composites(Sreekumar & Thomas, 2008).

Table 1: Melting temperature of different thermoplastics (Rajendran Royan et al., 2021).

Thermoplastics	Melting Temperature (^{O}C)
Polypropylene (PP)	150-160
Low density polyethylene (LDPE)	105-115
High density Polyethylene(HDPE)	120-190
Poly ethylene terephthalate(PET)	235-260
Polyvinyl chloride (PVC)	212
Acrylonitrile butadiene styrene	190-250

2.1.3.1. Poly vinyl chloride (PVC)

One of commonly used thermoplastic polymers is PVC(Lewandowski & Skórczewska, 2022).It has numerous beneficial qualities, including low flammability, low coat, and formulation flexibility, but it's use is restricted by qualities like weak impact toughness and low heat-softening temperature. The heat stability studies are therefore extremely important for PVC consumers. Rubber is frequently added to make PVC composites stronger. Additionally, several elastomers have been created to increase PVC's impact toughness (Bao et al., 2021).However, they frequently cause polymeric composites to lose strength, have a lower heat resistance modulus, and be more complex to produce.

PVC is known to be soluble in a range of organic solvents, such as cyclohexanone, tetrahydrofuran, and toluene. PVC is insoluble in water. The glass transition temperature (Tg) of PVC is around 85°C.Tg is the temperature at which an amorphous polymer transitions from a hard, brittle state to a soft, rubbery state. The melting temperature (Tm) of PVC is around 212°C. The chemical structure of PVC consists of a repeating unit of vinyl chloride monomers (CH_2=CHCl) connected by covalent bonds However, they frequently cause polymeric composites to lose strength, have a lower heat resistance modulus, and be more complex to produce (Tomaszewska et al., 2021).

There has been significant advancement in PVC toughening since the use of stiff filler particles. Natural fibers can reduce costs while concurrently achieving good mechanical qualities, thermal stability, and processing properties by toughened PVC with hard filler particles(Murugan et al., 2017).Numerous nanoscale fillers, including as montmorillonite, calcium carbonate, aluminium oxide, and silica, have typically been described in recent years.

2.1.3.2. Plant Fibers for thermoplastic composites

The reinforcing fibers in natural fiber composites (NFCs) commonly derived from carbon dioxide-neutral and renewable fiber sources, such as wood or plants (Al-Oqla et al., 2014).The increasing environmental contamination caused by waste from man-made items has led to a growing interest in using natural plant fibers as an alternative to man-made materials in most applications. As a result, natural fiber reinforcing with composites has become more and more popular. Several current studies demonstrate that a variety of natural fibers from plants and vegetables may be utilized to reinforce polymeric composites (Lotfi et al., 2021). Flax, jute, hemp, sisal, ramie, kenaf, pineapple leaf fibers, and wood fiber are some of the most widely used natural fibers; they have all been extensively studied and are utilized in a range of industrial purposes (Lotfi et al., 2021).The use of natural fibers in polymer composites is attracting the attention of industry executives and researchers. Although they are more environmentally friendly and support sustainable practices, natural fiber-reinforced composites have the potential to have a favourable influence throughout the projected lifespan (2022–2030) and reach more than US$ 12 billion by 2030.

The six forms of natural plant fiber are as follows: wood fiber, seed/fruit fiber, leaf fiber, grass fiber, and straw/stalk fiber(Ramamoorthy et al., 2015). Plant-based natural fibers are classified as the primary or secondary depending on how they will be used. Primary plants are those that are farmed mainly for their fiber, whereas secondary plants produce fibers as a consequence. Jute, hemp, kenaf, and sisal are a few examples of the primary plants. Coir, oil palm, Enset (a fake banana), and pineapple leaf fiber are a few examples of secondary plants.(Ramamoorthy et al., 2015; Sathish et al., 2021).

One prevalent natural polymer that may be found in plant cell walls is cellulose. It is the primary constituent of natural fibers and offers plant tissue shape and strength. Plants may provide cellulose, which is then utilised to manufacture a variety of goods including paper, textiles, and biofuels.

2.2. Cellulose

Cellulose is an organic compound with the formula $(C_6H_{12}O_6)_n$, a polysaccharide which consists of a linear chain of hundreds to thousands of β, 1–4 linked D glucose units. The most prevalent organic polymer on Earth is cellulose. Green plants, numerous types of algae, and oomycetes all have primary cell walls that contain cellulose as an essential structural element. It is secreted by some bacterial species in biofilms. Cotton fiber contains 90% cellulose, wood contains 40%–50%, and dried hemp contains about 57% cellulose (Aziz et al., 2019; George & Sabapathi, 2015).

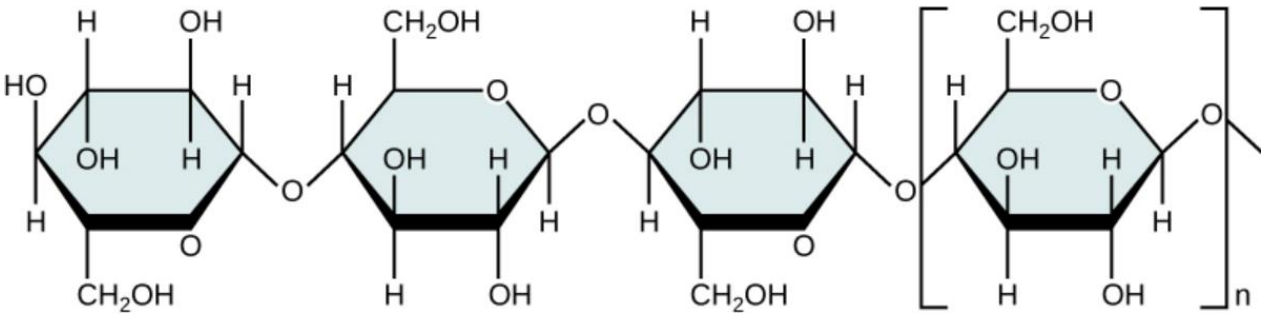


Figure 2. 1: Cellulose chemical structure and symbol(Schulz et al., 2000).

The two main categories of cellulose fibers are synthetic cellulose and natural cellulose. Synthetic cellulose is derived from non-renewable sources, as opposed to natural cellulose, which is derived from plants and animals (Ramamoorthy et al., 2015). The main drawback of synthetic cellulose is its environmental impact.

The production of synthetic cellulose involves the use of chemicals and energy-intensive processes, which contribute to greenhouse gas emissions and other forms of pollution. Additionally, the disposal of synthetic cellulose products can be problematic as they are not biodegradable and can persist in the environment for a long time.

Natural cellulose has several advantages over synthetic cellulose. Firstly, natural cellulose is renewable and biodegradable, making it a more sustainable option. It can be derived from various plant sources such as wood, cotton, or hemp, which can be grown and harvested in an eco-friendly manner.

Secondly, natural cellulose has superior mechanical properties compared to synthetic cellulose, making it more suitable for applications requiring high strength and durability. Furthermore, cellulose-based biomaterials offer important advantages over conventional synthetic materials, making them a promising platform for tissue engineering and other application (Rahman & Bhoi, 2021).

2.2.1. Natural cellulose fibers

Natural cellulose fibers can be extracted from a variety of bio resources, including plants, animals, and minerals. Natural fibers with cellulose bases can be made from a variety of fruits, vegetables, and plants. Figure 2.2, shows natural cellulose fibers derived from plant sources. The chemical constitution , growing region, plant age, and extraction techniques determine the physical and mechanical properties of natural cellulose fibers(Idicula et al., 2005; Peças et al., 2018).

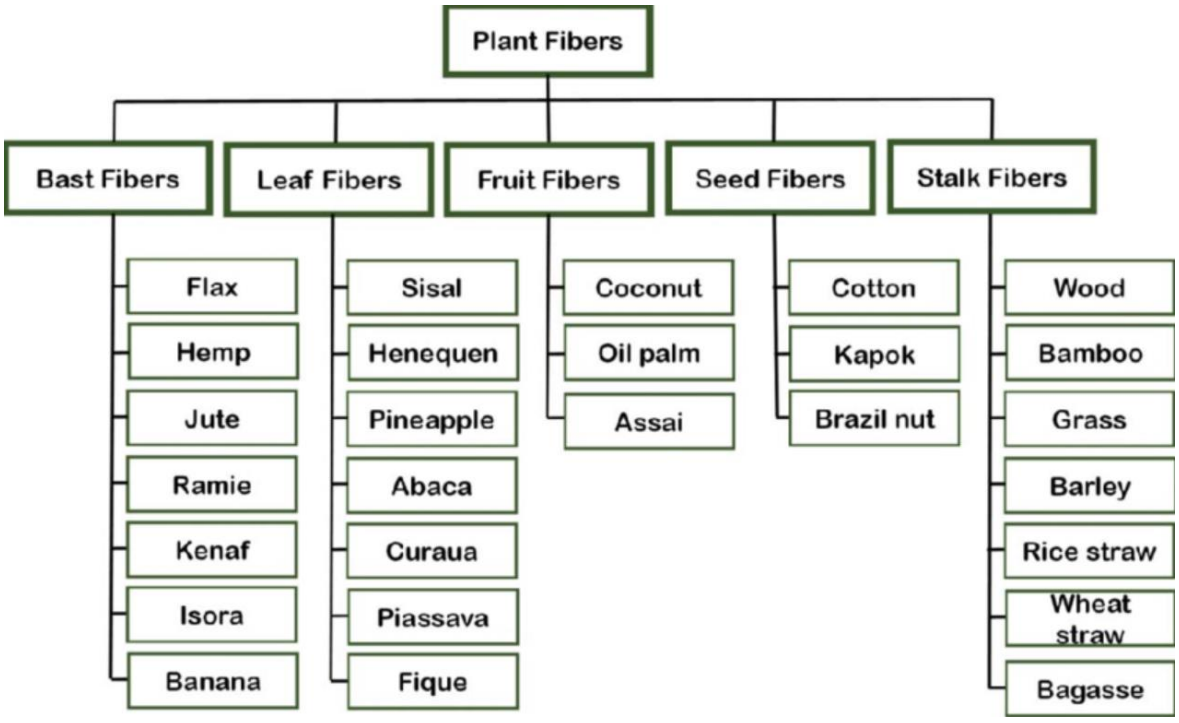


Figure 2. 2: Classification of natural cellulose fibers

Cellulose, a low-density, high-porosity material, has numerous applications in fields like adsorption, oil/water separation, and thermal insulation(Zheng et al., 2021).

Natural cellulose, derived from bacterial and plant-based sources, offers advantages over synthetic cellulose like availability, cost-effectiveness, usability, biocompatibility, negligible toxicity, mild immune response, and mimicking native tissues. Researchers are particularly interested in its use in polymer engineering systems for reinforcement in composite materials, as it mimics native tissues(Baghaei & Skrifvars, 2020).

Factors that have a significant effect on the characteristics of cellulose include the type of plant, the time of harvest, and the section used. Natural fibers have a higher tendency to absorb moisture, which impacts the dimensional stability of the composite specimen and causes micro cracking. It has a number of negative effects on the characteristics and long-term effectiveness of composites(Fang et al., 2013; Patel et al., 2012).

To overcome these limitations, readily available cellulose must be transformed into nano/micro-cellulosic forms or combined with suitable polymers to create composites that can enhance their desired properties. Nano form cellulose is superior to cellulosic micro fibrils because it has a higher surface-to-volume value and a nano-scale size effect(Subbiah et al., 2005).

Cellulosic nanostructures, which are among the many nanostructures derived from cellulose, have received a lot of attention because of their superior mechanical strength, flexibility, ability to be functionalized and blended, biodegradability, chirality, thermos stability, and low thermal expansion.

Cellulose nanofibers (CNF) have been proposed as a potential matrix for a variety of applications, including pharmaceuticals, energy storage, packaging, drug delivery, bio imaging and biomedical materials, protective coatings, barrier membranes, and filtration media (Moon et al., 2011; Pääkkö et al., 2007; Podsiadlo et al., 2005).

Cellulose nanofibers (CNFs) are a less abrasive, low-cost, low-density renewable material with qualities that include purity, biocompatibility, crystallinity, wettability, and a surface-tuneable structure. These attributes significantly impact a variety of environmental applications.

Their biocompatibility is particularly notable in various biomedical applications, such as the development of scaffolds for tissue engineering, which also enhances their utility in environmental applications(Torres et al., 2012).

The structure of cellulose nanofibers (CNF) is more stable due to its highly crystalline nature. When combined with other low-crystallinity materials like polyvinyl alcohol (PVA) and polyvinyl acetate (PVAc), the resulting nanocomposite gains stiffness and high strength, in addition to the native properties of the added materials. The high crystallinity found in these nanofibers also improves their ability to bind to different volatile compounds present in water and the atmosphere. Adsorbents based on cellulose nanofibers can be employed in the clean-up of water and air pollution. Additionally, their sorption properties can be utilized in sensors, catalysis, and molecular-level compound separation applications. The structure of these nanofibers can accommodate a wide range of guest nanoparticles, including Au, Ag, Pb, Ni, TiO2, CuO, and SiO2, thanks to their high porosity, intact crystalline structure, surface area, and mechanical stability (Frone et al., 2016; Jonoobi et al., 2010).

2.2.2. Cellulose Extraction

Extracting refers to the transfer of a compound from its initial state to another phase, achievable through methods such as liquid-liquid, solid-phase, acid-base, ultrasound-assisted, and heat reflux extraction. Cellulose extraction involves separating and isolating cellulose from its natural source .This process addresses two key issues: reducing plant or agricultural waste and converting this waste into beneficial products (Johar et al., 2012; Sheltami et al., 2012).

Agricultural waste contains high concentrations of cellulose, hemicellulose, lignin, xylose, ash, crude proteins, and other organic compounds. Burning agricultural waste, which is a common but harmful practice, poses serious health risks due to pollution and smoke, with part-per-million (ppm) concentrations of between 12 and 60. This practice results in double waste because it destroys valuable straw and contributes to hazardous pollution (Ajila et al., 2012; Madurwar et al., 2013).

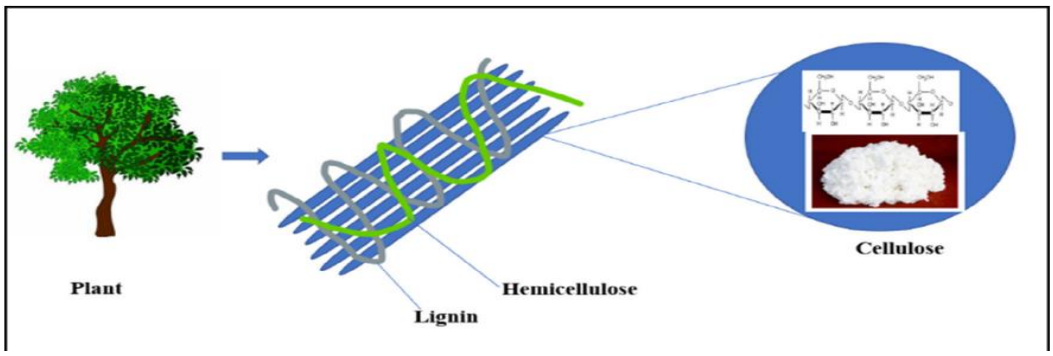


Figure 2. 3: Cellulose extraction from plant biomass(Chopra, 2022).

Figure 2.3 shows that the main component of a plant's structure is cellulose, whereas lignin serves as an adhesive to keep hemicellulose and cellulose joined and hemicellulose serves as a medium to link cellulose and lignin together (Jagadesh et al., 2022).

Cellulosic fiber extraction can be achieved on a nano or microscale using several chemical or mechanical methods. Chemical extraction methods are used to efficiently isolate cellulose. As showen in figure 2.4 the extraction process typically involves three main phases: pulping, bleaching, and pre-hydrolysis.

Pulping involves heating the fiber with an alkali such as NaOH, which helps to break down the lignin and hemicellulose, making the cellulose more accessible. Alkaline treatment with NaOH is particularly effective in reducing amorphous components, including lignin and hemicellulose, resulting in a higher purity of cellulose. This method is favored due to its efficiency in breaking down non-cellulosic components, making the cellulose more accessible for subsequent applications.

Bleaching, the final stage uses chemicals like H_2O_2, sodium chlorite, or ozone to obtain pure, white cellulose fibers (poursorkhabi et al., 2013). Bleaching not only removes residual lignin but also enhances the whiteness and purity of the cellulose fibers, making them suitable for various industrial applications.

These chemical treatments ensure that the cellulose extracted is of high quality, suitable for a wide range of applications in industries such as textiles, paper, and bio-composites.

The use of these specific chemicals is driven by their effectiveness in breaking down complex plant structures and yielding high-purity cellulose, which is crucial for producing materials with desirable properties such as high tensile strength, low density, and biocompatibility(Chopra, 2022; kahawita & samarasekara,2016).

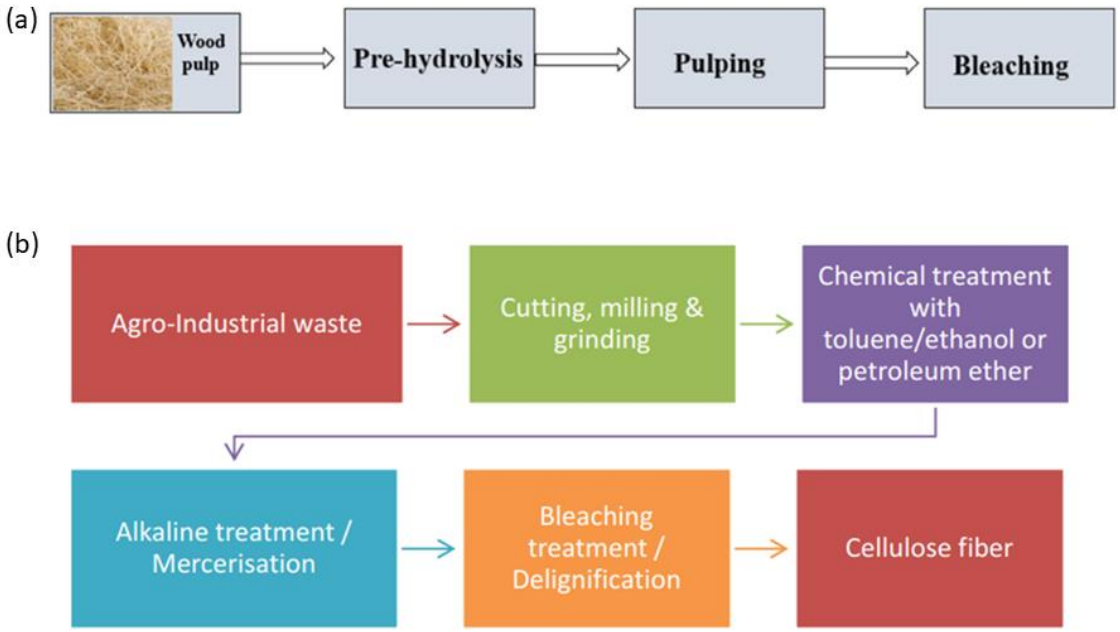


Figure 2. 4: (a) Cellulose extraction from plant biomass and (b) Simplified flowchart for extraction of cellulose fibers (Chopra, 2022)

2.2.3. Potential plant sources for CNF

The Musaceae family includes the native South-East Asian Musa plant (Musa acuminata) (Aziz et al., 2011; Li et al., 2010). This plant produces significant biomass, including bunches, pseudo-stems, leaves, and stalks, which are rich in fibers (Zuluaga et al., 2007). In tropical countries like Malaysia and South India, where bananas are widely cultivated, about 88.84% of the plant's waste is discarded after fruit harvesting, despite its high fiber content (Ramesh, 2018). The extracted banana fibers exhibit high tensile strength, low elongation, light weight, fire resistance, moisture absorption, low density, and high tensile modulus, making them valuable for various industrial applications (Gupta et al., 2020; Suparno, 2020).

The abaca plant (Musa textilis), which has become famous for its strong tensile strength and resilience to bending and salinity, is another well-known example. Some definitions classify abaca fibers as stem fibers, whereas others classify them as leaf fibers(Nurnasari & Nurindah, 2017).

Fout point five to 7.5 meters is the maximum height that the plant can grow. Its stem diameter ranges from 12.5 to 20 cm, and it may generate fibers that are 1.5 to 2.5 meters long and 12.5 to 30 cm wide. The outside portion of the fiber is stronger than the inside, and its quality changes based on where it is located on the stem. Additionally, abaca fibers float well, which makes them appropriate for use in maritime environments. (Suliyanthini, 2021).High tensile strength and resistance to banding and seawater characteristics abaca fiber.The frond's fiber quality is influenced by where it is located on the stem, with the outer part of the frond being more robust than the inner part. Abaca fiber also has excellent flotation qualitites (Suliyanthini, 2021).

Five locally superior abaca varieties were released in October 2019 from Talaud, North Sulawesi, where abaca is widely planted (Nebangka et al., 2020). Maize, a widely grown crop in Asian nations, is a rich source of natural fiber from its stems, leaves, and skins. Cornhusks contain 46.15% cellulose, 33.79% hemicellulose, and 8.92% lignin, with a tensile strength of 169.49 MPa (Herlina Sari et al., 2018).

The fiber can be altered by treating it with sodium hydroxide (NaOH) solution, which decreases hydrophilic characteristics but increases crystallization, tensile strength, and thermal resistance. (Sari et al., 2020; Sari et al., 2017).

Research by (Moon et al., 2016) has demonstrated the versatility of CNFs in various applications, including pharmaceuticals, energy storage, packaging, drug delivery, bioimaging, biomedical materials, protective coatings, barrier membranes, and filtration media. The low cost, low density and renewable nature of CNFs make them highly attractive for these applications. In environmental applications, the high crystallinity of CNFs enhances their ability to bind with various volatile compounds found in water and the atmosphere, making them effective for pollution clean-up efforts(Menon et al., 2017)focused on the biocompatibility and mechanical strength of CNFs, which are crucial for biomedical applications, especially in tissue engineering scaffolds.

Furthermore, combining CNFs with other low-crystallinity materials, such as polyvinyl alcohol (PVA) and polyvinyl acetate (PVAc), results in nanocomposites with enhanced stiffness and strength(Trache et al., 2024).

The high porosity and mechanical stability of CNFs also enable them to accommodate a wide range of guest nanoparticles, including Au, Ag, Pb and Ni.This property expands their applications in sensors, catalysis, and molecular-level compound separation. The diverse plant sources and the innovative extraction techniques used in these studies underscore the significant potential of cellulose nanofibers in advancing various industrial and environmental technologies(Jayeoye et al., 2023)..

2.2.4 Applications of Cellulosic Materials

If the level of substitution with functional groups is modest, cellulosic materials are well investigated for sustainable applications (Courtenay et al., 2018).

The demand for sustainable products has increased awareness of cellulose use in recent years. Because cellulose can be transformed into biodegradable and nontoxic polymers from sustainable natural resources, it can replace plastics made from petroleum (Peng et al., 2020).

Due to their promising mechanical and barrier qualities, these products are being employed more and more in food packaging as coatings, self-standing films, and paperboard. Since virgin cellulose and its derivatives include a variety of functionalities, these building blocks offer a special platform for chemical modification via covalent functionalization to give cellulose stable and long-lasting functions (Aziz et al., 2022; Vroman & Tighzert, 2009).

Research on CNFs as a reinforcement agent in composite materials is a key area of focus (Woehl et al., 2010). These nanoparticles have a high aspect ratio, allowing good stress transfer between filler and matrix, a high contact surface due to nanometric dimensions, hydroxyl groups on the surface, and are primarly derived from renewable resources, making them ideal for functionalization reactions (Klemm et al., 2011).

2.2.5. Enset fiber

The pseudo-stem and leaves of enset plants are the source of enset fiber, a certain type of plant fiber. Harvested as agricultural waste, the fibers are sun -dried in Ethiopian rural regions before being used to make sacks, bags, mats, sieves, ropes, cordage, and tying materials for construction projects(in place of nails). Depending on the pseudo-stem's height, the extraction method, and the final use, these fibers may extend for up to six meters.

During extraction, they often lose some of their length, often to the extent of one or two meters. Furthermore, it can potentially be utilized in a range of applications due to its durability and versatility (Brandt et al., 1997).

Although cultivated enset grows across Ethiopia, mostly at higher elevations, it is found in Ethiopia's southwest highlands. It is also found in the country's central and Southern regions. In order to maintain food security, enset is currently the principal crop of indigenous African system that is sustainable. Approximately 20% of the population in Ethiopia, primarily residing in the southern Ethiopian highlands, is dependent on enset (Teli & Terega, 2017).

Figure 2.5: Enset ventricosum plants.

2.2.5.1. The characteristics of Enset fiber

Enset fibers are significant categories of natural fibers, primarily consisting of cellulose, hemicellulose, lignin, pectin, and other extractives (Rohit & Dixit, 2016).

The most crucial component is the intermediate layer, known as cellulose, which is composed of helical cellular micro-fibrils and controls the mechanical characteristics of the fiber in its ultimate form (Kumar et al., 2005).

Table 2: Chemical composition of enset fiber (Dejene, 2024).

Enset fiber	Cellulose	Hemicellulose	Lignin	Ash
Composition	64.46	22.47	6.88	5.66
S.D (%)	[0.23]	[0.27]	[0.55]	[0.02]

As Table 2 shows, in contrast to many other well reported plant fibers, enset fiber showed a considerably similar percentage of cellulose and lower concentration of lignin.

Due to its exceptional thermal stability up to temperatures of about 260 °C, as indicated by (Teli & Terega, 2017), this fiber has a greater chance of being utilised in composite materials and the proper technological application.

2.3. Environmental impact of PVC solid waste

Plastic is being used at a rapid rate because of its many benefits, which include its high chemical stability, affordability, and versatility. Plastic materials are frequently used for packaging and other typical applications due to their versatility. Industries are getting more interested in the Plastic manufacturing industry as a result of the increased production of commodities utilizing plastics. The majority of our everyday lives are now dominated by plastics, and the global output of plastic has significantly expanded in recent years. The amount of things produced using diverse plastic materials has increased dramatically. This massive rise in plastic product production also leads to increased garbage production, which creates additional issues (Gu & Ozbakkaloglu, 2016).

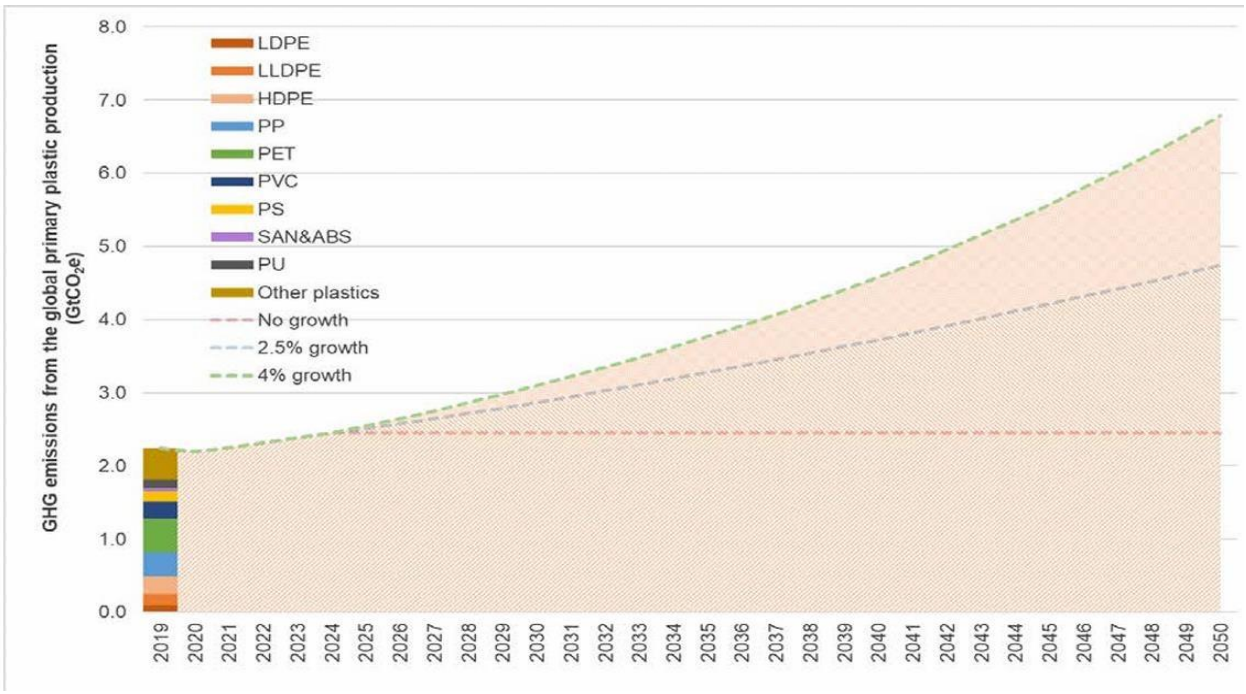


Figure 2.6:GHG emissions from global primary plastic production between 2019 and 2050 under BAU No (Karali et al., 2024)

GHG emissions decreases to around 3.59 GtCO2e and 5.13 GtCO2e in 2050 with 2.5%/yr and 4%/yr increase in plastic manufacturing, respectively, under BAU scenarios with a decarbonised electric grid. Emissions from primary plastic manufacturing would still be high in 2050, even if a decarbonized grid would result in a 25% reduction in emissions. The total greenhouse gas emissions from primary plastic manufacturing, however, may still make up 17-21% and 22-26% of the remaining carbon budget, respectively, providing a 50% and 67% chance of keeping Below the 1.5°C barrier (Karali et al., 2024).

Plastic pollution has become one of the most pressing environmental issues as the world's ability to handle the rapidly increasing supply of throwaway plastic items gets overburdened. Plastic pollution is especially evident in developing nations in Asia and Africa, where waste collection systems are either nonexistent or insufficient (Alqattaf, 2020).

In regular solid waste disposal sites, conventional plastics have a low biodegradability and would require hundreds of years to break down.

The majority polymers are now linked to environmental issues. Most of them are made from petroleum, which is then consumed and released back into the environment after use. Large amounts of carbon dioxide are produced as a byproduct of their manufacture, which requires a fair amount of energy. Most people agree that carbon dioxide either causes or at least contributes to global warming. Many substitute materials have been developed to mitigate these issues. Biodegradable polymers have become more and more significant in recent years, especially in view of the increasing threat to the environment caused by plastic waste. Living organisms such as bacteria, yeast, fungus, and other enzymatic agents may break down biodegradable polymers (Avella et al., 2005).

In a natural environment like a landfill, they are made to be easily destroyed and to eventually disappear (Ishigaki et al., 2004). Consequently, in order to decrease the damage that plastic wastes bring to the environment, there is a considerable interest in substituting biodegradable polymers for some or all synthetic plastics in a variety of applications. Biodegradable polymers that are known to be mostly biodegradable include cellulose, starch, polylactic acid, keratin, and similar natural polymers.

Through partial breakdown, partially biodegradable plastics made by combining biodegradable and non-biodegradable polymers can successfully lower the amount of plastic trash generated. They are more beneficial than fully biodegradable ones because of their better qualities and financial benefits(Fredi & Dorigato, 2021).

2.3.1. The factors that need consideration in bio-composite manufacturing

Packaging materials like metal, glass, and plastic are used to protect items from damage. However, non-biodegradable plastics like PET, PVC, PE, and PS cause waste issues. Biodegradable polymers, made from renewable resources, are gaining attention as eco-friendly alternatives due to affordability and availability(Chen et al., 2023).Multiple variables in thermoplastic-natural fiber composites must be taken into account in order to optimize and identify the most effective features of bio-composites. The possibility of producing composites with subpar mechanical properties throughout the production process is one of the challenges in the developmental of bio-composites. The weakening of interface adhesion is caused by a variety of elements involved in the manufacture of bio-composites.

In addition, those two categories may be utilized to group the important considerations that need to be made while producing bio-composites(Aziz et al., 2023) .

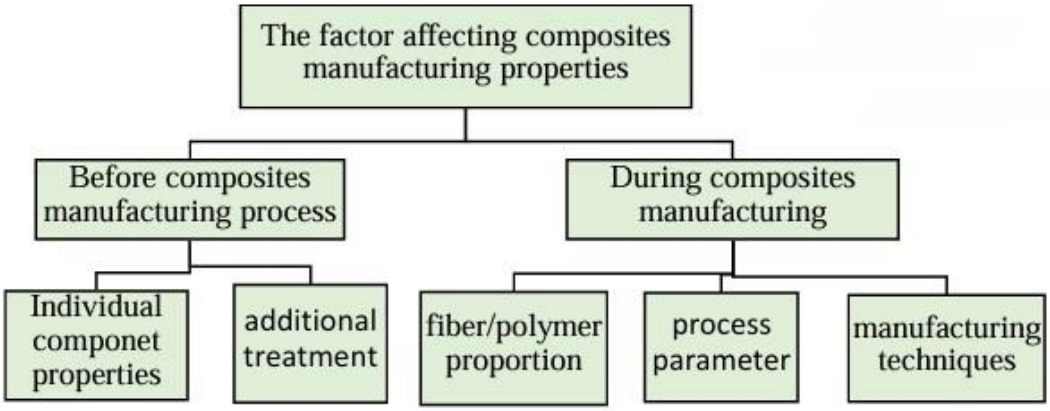


Figure 2. 7: Factor which affect the properties of composites (Cionita et al., 2024)

2.4 Plastic manufacturing methods

2.4.1 Injection molding Method

Injection molding is a leading technique for making plastic parts by melting plastic and injecting it into precise molds. Once cooled and solidified, the plastic takes on the mold's shape, making it perfect for mass-producing components with high accuracy and consistency. However, it may struggle with geometries that demand uniform cooling to prevent issues like warping or internal stresses.It's used widely, from small items like buttons and bottle caps to large parts for cars and furniture(Khosravani & Nasiri, 2020).

2.4.2 Blow molding Method

Blow molding is another key method in plastic manufacturing, particularly for shaping hollow products such as bottles and containers. It starts with melting plastic, which is then formed into a tube called a parison. This parison is placed in a mold and inflated with air, shaping the plastic to fit the mold. After cooling, the plastic keeps its hollow form defined by the mold. Blow molding is efficient, producing uniform hollow shapes quickly. Achieving precise wall thickness and dimensional control can also be problematic, especially for complex shapes or containers with intricate features(Belcher, 2011).

2.4.3 Solution Casting Method

The most effective mechanical reinforcement from ENCs occurs in composites created through casting. This is due to the extended process of solvent evaporation during sample preparation, allowing particles extended time to self-assemble into networks. Over the past two decades, extensive research has been conducted on this method, resulting in a well-established theory that showcases the full potential of cellulose nanoparticles as agents for enhancing mechanical strength, stiffness, and other critical properties of PVC matrices. This approach holds promise for advancing materials for diverse applications such as packaging(Sucharitpong et al., 2020).

The solution casting method is distinctive, especially in research involving nanocellulose-reinforced PVC nanocomposites. It offers advantages over traditional plastic manufacturing methods, particularly in specialized applications. In solution casting, PVC and nanocellulose are dissolved in a solvent to create a uniform mixture.

This solution is then cast into a mold or onto a substrate, where the solvent evaporates, leaving a seamless composite film or layer(Sheng et al., 2017; Sucharitpong et al., 2020).

One major benefit of solution casting is its ability to ensure uniform dispersion of nanocellulose within the PVC matrix. This enhances mechanical properties and biodegradability, supported by detailed analyses. By using a common solvent for both materials, solution casting achieves molecular-level blending that's hard to replicate with other methods like extrusion or injection molding. This results in strong bonding between PVC and nanocellulose, improving overall performance. Furthermore, solution casting is straightforward and cost-effective, making it ideal for laboratory research and development. It allows precise control over film composition and thickness, crucial for maintaining consistency in experiments. Its ability to produce thin films is advantageous in packaging and other applications where efficiency and material performance are key(Acquavia et al., 2021; Mathew & Oksman, 2014). Thus, solution casting is pivotal for creating high-quality nanocomposite films with superior mechanical strength and environmental sustainability. Its potential in advancing composite materials is highlighted in studies on nanocellulose-reinforced PVC, showing promise for sustainable material development.

Table 3: Summary of Literature gap

Researcher	Raw material	As response	Objective	Gap
(Joshi & Marathe, 2010)	Wood fiber and PVC	Tensile and impact strength	To investigate the properties of composite made from PVC reinforced from WF composites reinforced with WF	• reduction in mechanical property
(Abraha et al., 2023)	Enset Fiber-Reinforced PLA-Based Biocomposites	Tensile, flexural, impact strength and water absorption	Potential of sustainable and unutilized agro-waste natural fibers (enset fibers) as reinforcement	• Further research is needed to optimize the extraction and processing
(Maou et al., 2021)	Date palm fibers (DPFs), PVC and HDPE	Tensile, Flexural strength, water absorption, TGA and (DSC)	To investigate the effect of chemical modifications of DPFs on the thermo-physical properties of PVC-HDPE composites	• Exploring Practical Application of Chemically Modified DPFs in PVC-HDPE Composites
(Shankar et al., 2020)	Teak sawdust, PVC, Calcium carbonate	Tensile, compressive, bending and impact strength	To evaluate the mechanical properties of a WPC made using teak sawdust, PVC, and calcium carbonate	• Lack of Process Development and Comparative Analysis • Insufficient investigation of interfacial adhesion.
(Joglekar et al., 2021)	Citrus Maxima fibers-based PVC	Tensile modulus, breaking strength, yield stress	To effectively utilize the waste fiber portion of the Citrus Maxima to create PVC-based composites with improved mechanical properties.	• Limited Optimization of Processing Parameters • • No comparison with natural fiber reinforcements.

2.6. Research gap

Plant fibers are a fascinating alternative source for extracting cellulose derivatives because they come from residues with unique physical and chemical properties and are abundant, showing great potential for various important applications. Various publications have pointed out that PVC polymer offers advantages such as being inexpensive, resistant to fire, and versatile in formulation, but its practical use is limited due to drawbacks like poor impact resistance and inability to decompose naturally.

These raw materials contain a significant amount of organic matter that could be valuable in multiple industries if utilized properly. There has been limited exploration in the literature on using diverse raw materials in packaging. Enset fiber is one such material with potential for this purpose, found in regions like Ethiopia, which leads in both quality and quantity of enset production globally. While there has been extensive research on composite materials strengthened by natural fibers such as kenaf, palm oil, bamboo, jute, sisal, coconut, pineapple fibers, and other additives, there remains a gap in the literature concerning the use of PVC composites reinforced specifically with enset fiber, particularly in nanocomposite form, for packaging applications.

This research investigates the extraction and use of cellulosic materials from enset agro-industrial residues to produce PVC composite materials. It emphasizes significant improvements in the strength and ability to break down naturally, achieved by mixing enset nanocellulose into PVC mixtures.

CHAPTER THREE

3. MATERIALS AND METHODS

3.1. Chemicals and Materials

Enset fibers were collected from a local farm in Hawassa, South Ethiopia. Before analysis and extraction, the pods were washed with tap water and distilled water, sun-dried and then ground. The chemicals, i.e., distilled water, tetrahydrofuran (THF) 99.5% purchased from HiMedia, Mumbai, India, sulfuric acid (H2SO4, 95–97%), hydrogen peroxide (H2O2, 30%) solution (Merck), glacial acetic acid, and sodium hydroxide (NaOH, 7%), were purchased from Sigma Aldrich. Commercial PVC powder (Yerosen pipe factory), Sabic's 25 kg powdered PVC product, standardized for industry requirements, is pristine white with a K-value ranging from 65 to 68 and meets international trade specifications, ensuring authenticity and reliability. All chemical products were used as received without modification.

3.2. Cellulose extraction from Enset fibers

The non-cellulosic components in enset fibers were initially eliminated to extract cellulose. To carry out this, ground 30 g of raw enset fiber was washed with distilled water for 1h at 60^0C under stirring. After that, an alkaline pre-treatment with 4% w/w NaOH solution at temperature of 80°C for 2 h under agitation at 500 rpm was conducted to reduce the amorphous concentration, which includes lignin and hemicellulose. For the alkaline treatment, the dewaxed sample was adjusted to 1:10 (g/mL) with NaOH solution. Using distilled water, the samples' excess alkali was removed. The subsequently obtained alkali-treated enset fibers were then bleached for a further two hours at 80 °C using a solution consisting of equal parts (v:v) of aqueous sodium chlorite (1.7 wt.% $NaClO_2$ in water) and acetate buffer (27 g NaOH and 75 mL glacial acetic acid, diluted to 1L distilled water). The procedure described above produced a pure white product known in this study as bleached enset fibers (cellulose fiber).

3.3. Enset nanocellulose (ENC) isolation

The ENC were prepared via the hydrolysis method.18 grams of the pretreated dry cellulose enset cellulose powder was preheated with various acid concentrations with the fiber to solution ratio of 1:15 (g/mL) for different reaction times at various temperatures under agitation at 500 rpm. This was followed by an immediate quenching of the reaction by the addition of a tenfold amount of distilled water then resulting suspension was filtered, centrifuged, and dialyzed using cellulose membrane until pH became constant. This was further centrifuged at 6000 rpm at room temperature for 15 min and this step was repeated several times until a constant pH in the range of 5–6 was reached. The suspension was further dialyzed against deionized water for 5 days to neutralize and hence eliminate all the free acid molecules, including the non-reactive sulfate group and salts, in addition to soluble sugars. After dialysis, the acquired suspension was ultra-sonicated for 5–15 min at 60°C resulting in an aqueous homogenized dispersion. Then filtered the powder out using filter paper and dried at room temperature. This was eventually named as the ENC, and ENC yield was measured. Then 35% H_2SO_4 acid concentration was used to convert microcellulose into nanocellulose and to remove the remaining nanocellulose components, at 60 °C for 30 minutes stirring time was choosed because of its great yield of ENC.

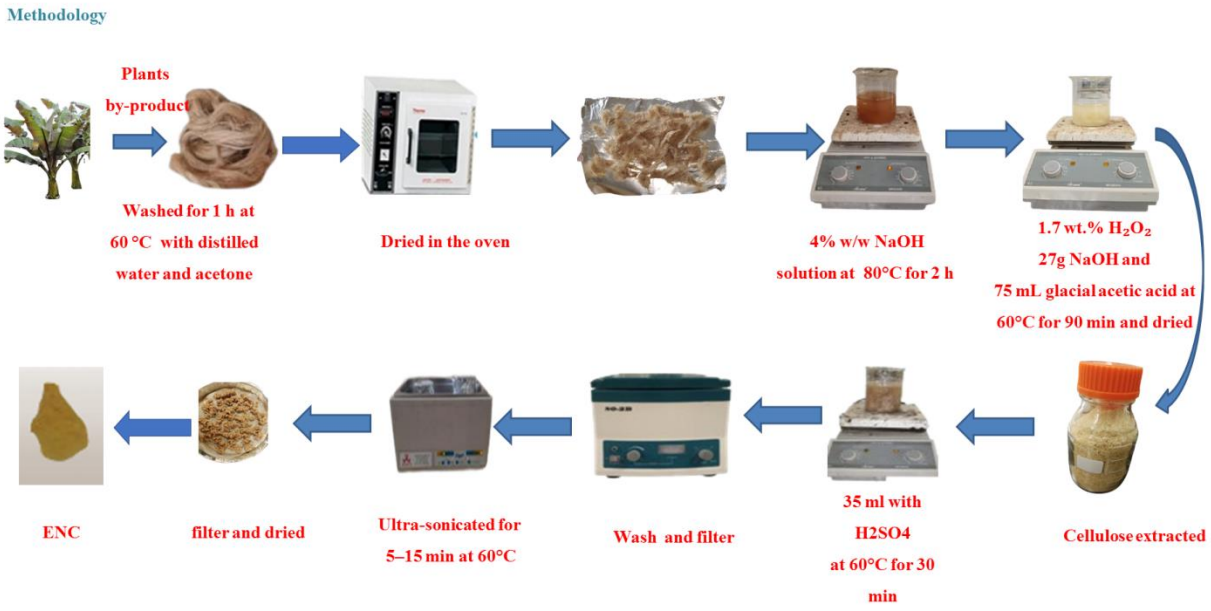


Figure 3. 1:Enset cellulose and Enset nanocellulose (ENC) extraction

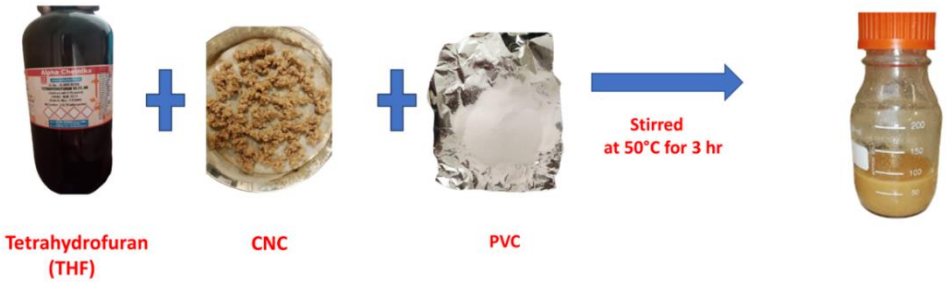
3.4. Preparation of composite films

The composite materials were prepared using the solution casting technique. PVC was dissolved in THF at 2:1 (g/mL). Films of thickness 3.22 mm with different enset nanocellulose contents (5, 10, 15, 20, and 25% by wt. %) mixed with PVC dissolved THF were stirred at 50ºC for 3 hours. After this, the solution was sonicated for 30 minutes in order to get homogenous solutions. The solution was cast into a sample holder and allowed to dry at room temperature for 24 hours. Then, the prepared composite films were peeled off from the sample holders.

Table 4: The sample design or mixing ratio of PVC/Enset Nano cellulose composite (wt%)

Sample Name	**PVC (wt%)**	**Enset nanocellulose (wt%)**
1	**100**	**0**
2	**95**	**5**
3	**90**	**10**
4	**85**	**15**
5	**80**	**20**
6	**75**	**25**

A.



B.

C.

0%	5%	10%	15%	20%	25%
					

Figure 3. 2 A: Sample preparation of composites B: and C: Prepared samples at different ratio of ENC

3.4. Sample Characterization

Morphological, phase, thermal stability, biodegradability, mechanical test, and functional group analysis were analyzed after sample preparation. Functional groups of all samples were recorded at a spectral width ranging from 4000 to 600 cm−1, using FTIR spectrometry (Perkin-Elmer Spectrum 2000) equipped with ATR accessory. The morphological features of the outer surface of samples were evaluated using scanning electron microscopy (SEM - HIROX SH 4000 M) at 15 kV, all samples were vacuum coated with carbon.

Three samples were tested and various images were recorded; size determination was done using Image J software. The thermal stability were evaluated by thermo gravimetric analysis (TGA, Discovery TGA from TA instruments). Samples of ca. 5−10 mg were heated to 700 °C under nitrogen atmosphere at a heating rate of 10 °C/min. The crystalline structure of cellulosic materials was examined using X-Ray diffractometer (D2 PHASER diffractometer, BRUKER) Cu Kα radiation; λ = 0.15406 nm; 40 kV, 10 mA; 2θ range: 10–40°).

3.5. Mechanical measurement

Tensile strength and Elongation properties measurements were performed using an Instron 4465 Universal Tensile Tester according to ASTM D668M, WP 310 Materials testing. The tensile tests were conducted at a crosshead speed of 10 mm/min and 5KN.

3.6. Biodegradability test

3.6.1. Swelling in water environment

To test the swelling properties of films in water, 1 × 1 cm^2 samples (W1) were weighed and placed in 100 mL of distilled water. The swelling test was carried out according to ASTM D2765-95C(Deepa et al., 2016). The swelling ratios (SW) of the films were calculated using the following equation:

$$\%SW = \frac{W_2 - W_1}{W_1} \times 100 \ldots\ldots\ldots\ldots\ldots\ldots (1)$$

Where $W_2$the sample's weight after swelling and W_1 is the weight of the dry sample. The swelling property of NC film was performed in distilled water.

3.6.2. Degradation studies in Acidic Environment

A strip of 1 cm x 1 cm was cut from the film, precisely weighted, and put into a 30% concentration of sulfuric acid to investigate the effect of the acid on the film. The percentage weight loss was calculated for 25 days.(Mostafa et al., 2018).

$$\%WL = \frac{W_2 - W_1}{W_1} \times 100 \ldots\ldots\ldots\ldots\ldots\ldots (2)$$

Where: W_2 is the sample's weight after acid treatment and W_1 is the weight of the dry sample.

3.6.3. Degradation studies in alkaline Environment

A strip of 1 cm x 1 cm was cut from the film and was precisely weighted and put into 30% concentration of NaOH to investigate the effect of the base on the film. The percentage weight loss was calculated for 25 days(Mostafa et al., 2018).The percentage weight loss was calculated using Eq. (2) after each time period till a constant weight loss was observed.

3.6.4. Degradation Studies in a Saltwater Environment

A strip of 1 cm x 1 cm was cut from the film and was precisely weighted and put into 30% concentration of sodium Chloride to investigate the effect on the film. The percentage weight loss was calculated for 25 days(Deepa et al., 2016).The percentage weight loss was calculated using Eq. (2) after each time period till a constant weight loss was observed.

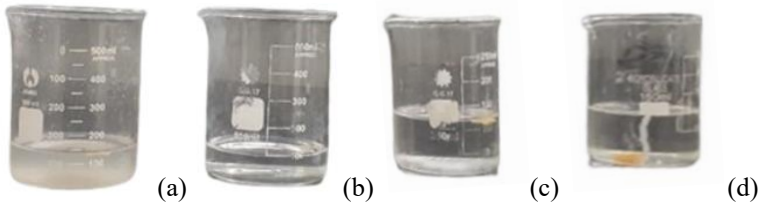
3.6.5. Biodegradation Evaluation in Soil

The biodegradability of the film has been investigated for 25 days on a laboratory scale using the soil burial method according to ASTM D 2216 (Machado et al., 2015; Mostafa et al., 2018). The soil was placed in a plastic container with tiny holes, and specimens with dimensions of 1 cm x 1 cm were buried at a 10-cm depth from the surface to allow the action of the soil.

The physical changes were observed at regular intervals by removing specimens, washing and drying them. Then the percentage weight loss was calculated.

$$WL\% = \frac{W1-W2}{W_1} \times 100 \quad \ldots\ldots\ldots\ldots\ldots\ldots (3)$$

where W1 is the weight of dry sample and W2 is the weight of the soil buried sample.

a) 

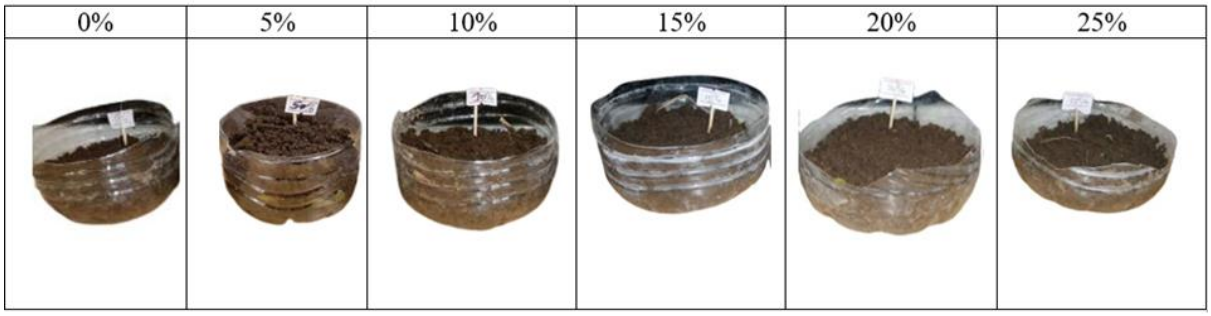
b) 

Figure 3. 3: a) Composite film samples immersed in different Medias for biodegradability test a,NaCl b,NaOH c,H_2SO_4 d,Swelling and b) soil tests at different concentrations

CHAPTER FOUR

4. RESULT AND DISCUSSION

4.1. XRD Analysis

The X-ray diffraction (XRD) patterns showed the crystalline structures of three distinct samples: Enset, Bleached, and ENC. Each plot shows intensity (Y-axis) against diffraction angle 2θ (X-axis), revealing the crystallinity and specific crystalline phases present in the samples.

An XRD analysis was conducted on raw fiber, bleached fiber, and ENCs extracted from ensete fiber. Figure 4.1 illustrates different peak patterns for these samples due to varying chemical compositions at different treatment stages. The raw enset contains non-cellulosic constituents like hemicellulose, extractives, and lignin, which were progressively removed in pretreatment processes, leading to observable semicrystalline amorphous humps and crystalline peaks. Experimental data indicated peaks at 2θ angles of 15.74, 16.80, and 22.65, with crystallinity index (CI) of approximately 44.09%, 62.30%, and 77.25% for raw, bleached, and isolated ENCs, respectively, determined using the Scherrer equation.

The XRD patterns clearly show that crystallinity increases with sequential chemical pretreatments, demonstrating the efficacy of methods in eliminating non-cellulosic components. Amorphous elements such as hemicellulose and lignin dissolve effectively through alkali treatments, significantly enhancing sample crystallinity (Dube, 2022)**.** The remaining amorphous cellulose regions are susceptible to sulfuric acid, where penetration of hydronium ions promotes hydrolysis, leading to the formation of distinct crystallites(Dube, 2022)**.** Figure 4.1 reveals that removing the amorphous cellulose domain increases the CI of ENCs, resulting in narrower and sharper peaks This may be associated with the re precipitation of cellulose after sulfuric acid hydrolysis. The average crystallite size of ENCs was determined as 52.41 nm using the Scherrer equation(Beyan et al., 2021).

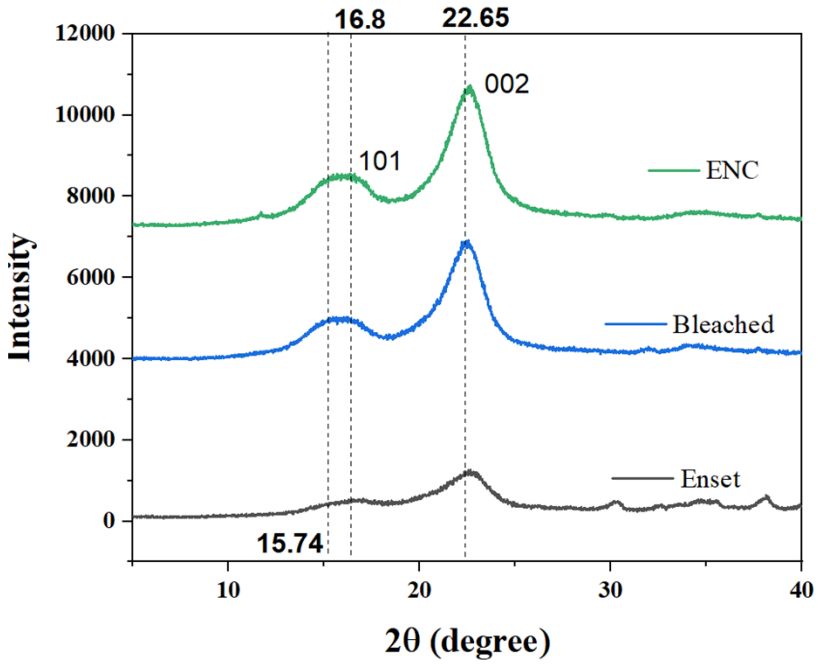


Figure 4. 1: XRD results of enset before and after different treatment.

4.2. Functional group analysis

The FTIR spectra were used to evaluate the effectiveness of chemical pretreatment in extracting enset nanocellulose (ENC) from enset cellulose through acid hydrolysis. The spectra for raw, bleached, alkali-treated, and isolated enset fibers were measured and analyzed. Figure 4.2 shows the spectra and distinctive bands for these fibers. The FTIR spectra of raw, alkali-treated, bleached, and isolated ENCs revealed different bands at 3277.00-3348, 2922.65-2924, 1740, 1600-1634.74, 1414.16, 1315.45-1327.48, 1018.75-1028.75, and 899 (cm^{-1}), indicating the presence of various functional groups corresponding to the chemical structures of cellulose and its derivatives. The defining band at 3277.00-3348 cm^{-1} was produced by the stretching vibration of the O-H group, connected to the intermolecular hydrogen bonds of cellulose (Dube, 2022).

Figure 4.2 shows that as chemical treatment progresses, the transmittance in this region gradually changes. The broad peak at 3277.00-3348 cm^{-1} is associated with the stretching vibrations of the O-H group, indicating the presence of intermolecular hydrogen bonds in cellulose. This peak signifies the hydroxyl groups of cellulose, with its intensity varying according to the degree of treatment, reflecting changes in hydrogen bonding.

The peak at 2922.65-2924 cm^{-1} corresponds to the stretching vibrations of aliphatic C-H bonds, particularly methyl and methylene groups, indicating the presence of cellulose and other polysaccharides in the biomass, with sharper peaks signifying higher cellulose content. The peak at 1740 cm^{-1} is attributed to the stretching of carbonyl groups in acetyl and uronic ester groups in hemicellulose or carboxylic groups in lignin, with its disappearance after treatment indicating the removal of hemicellulose and lignin. The peak at 1634.74 cm^{-1} represents the bending vibrations of O-H groups, primarily from absorbed water, with its intensity indicating moisture content and changes due to chemical treatments. The peak at 1414.16 cm^{-1} corresponds to the C-H deformation vibrations in cellulose and hemicellulose, with changes reflecting modifications in the polysaccharide structure during treatment. Peaks at 1315.45-1327.48 cm^{-1} are associated with the C-O stretching in cellulose and the C-O ring vibrations in lignin, indicating the presence and removal of lignin and hemicellulose components. Peaks at 1018.75-1028.75 cm^{-1} are linked to the C-O-C pyranose ring vibrations in cellulose, indicative of the cellulose backbone structure. The peak at 899 cm^{-1} indicates the β-glycosidic bonds between glucose units in cellulose, signifying the intact cellulose structure and showing the degree of polymerization (Dube, 2022; Puglia et al., 2024; Teli & Terega, 2017).

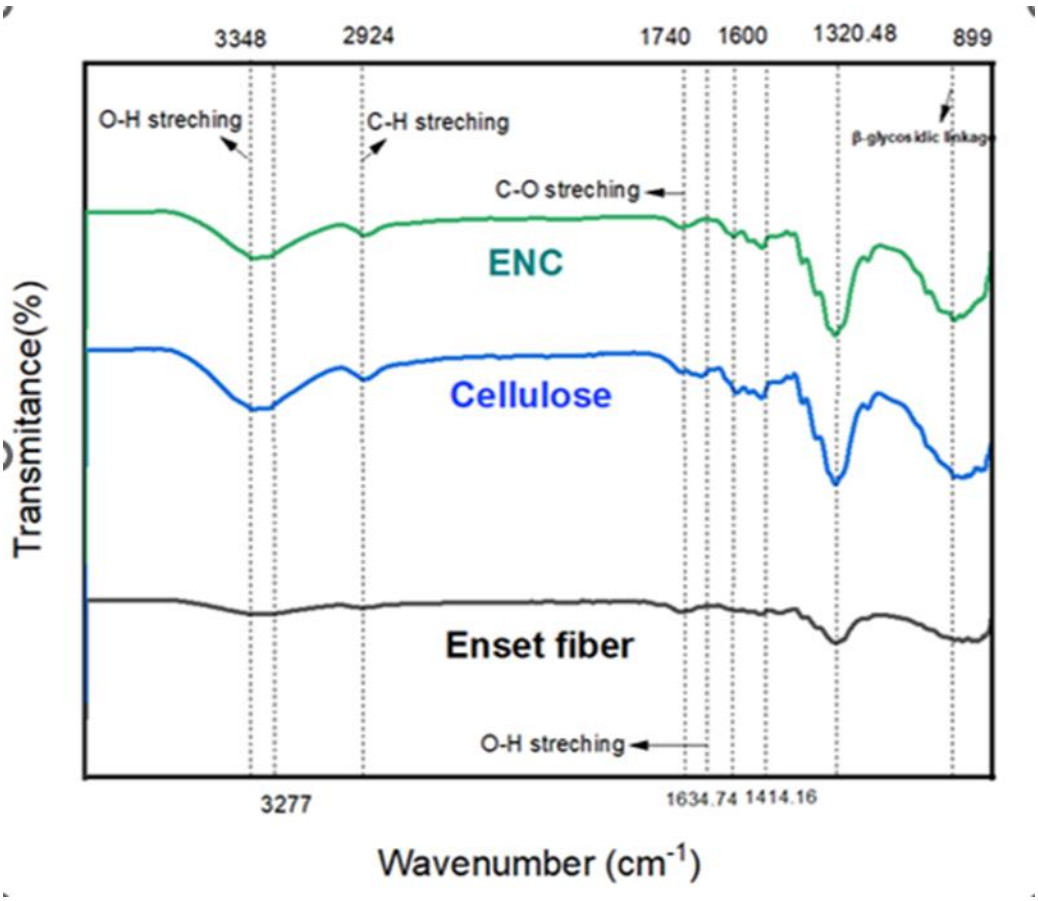


Figure 4. 2:FTIR result of Enset and its different treatment.

Chemical treatment alters these bands, reflecting changes in fiber composition. Figure 4.3 shows that the FTIR spectrum of treated PVC-ENC composite reveals shifts in frequencies and new peaks due to interactions between the solvent and composite matrix. This indicates the formation of new bonds and the presence of several functional groups arising from these interactions, which are not present in the pure PVC spectrum. Such changes demonstrate successful incorporation of ENC into the PVC matrix, enhancing its properties by introducing functional groups from the natural fibers. The results indicate the presence of several functional groups from the interaction between solvents and the composite matrix, signifying the successful formation of new bonds and shifts in frequencies compared to pure PVC. The spectrum showed specific peaks, such as 883.67 cm^{-1} (C-H bending in alkynes), 705.02 cm^{-1} and 769.63 cm^{-1} (C-H out-of-plane vibrations in trans-alkenes), 1157.33 cm^{-1} (C-H wag in alkyl halides), and 1731 cm^{-1} (C=O stretch in carbonyls). These functional groups indicate the composite's improved chemical structure, derived from materials like carbohydrates, starch, and other natural gums.

Their presence points to the successful reinforcement of PVC with enset nanocellulose, enhancing its overall properties(Joglekar et al., 2021; Maou et al., 2021).

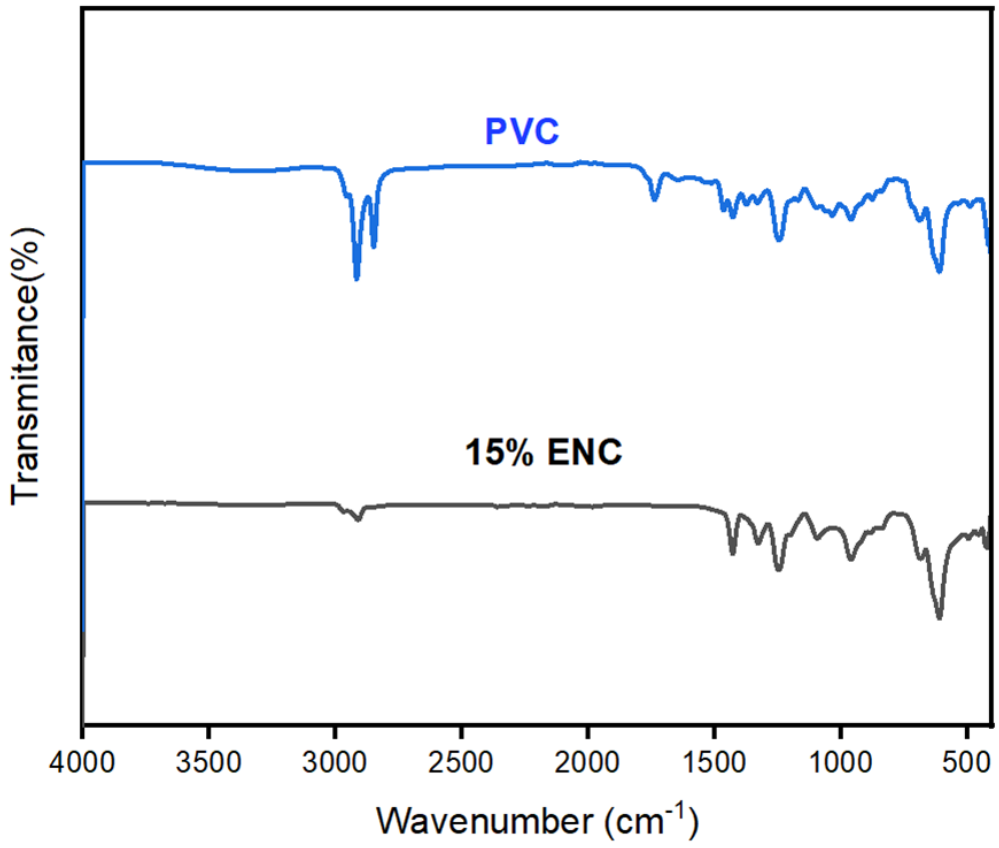


Figure 4. 3: .FTIR for PVC and 15% enset reinforced PVC.

4.3. Thermal stability analysis

This result provides an in-depth analysis of the thermal stability of variously treated enset fibers, including raw enset, bleached, alkali-treated fibers, and nanocellulose (ENCs), using Thermogravimetric Analysis (TGA) and Differential Thermal Analysis (DTA). These techniques measure changes in physical and chemical properties as a function of increasing temperature, providing critical data on the decomposition patterns and thermal stability of the samples.

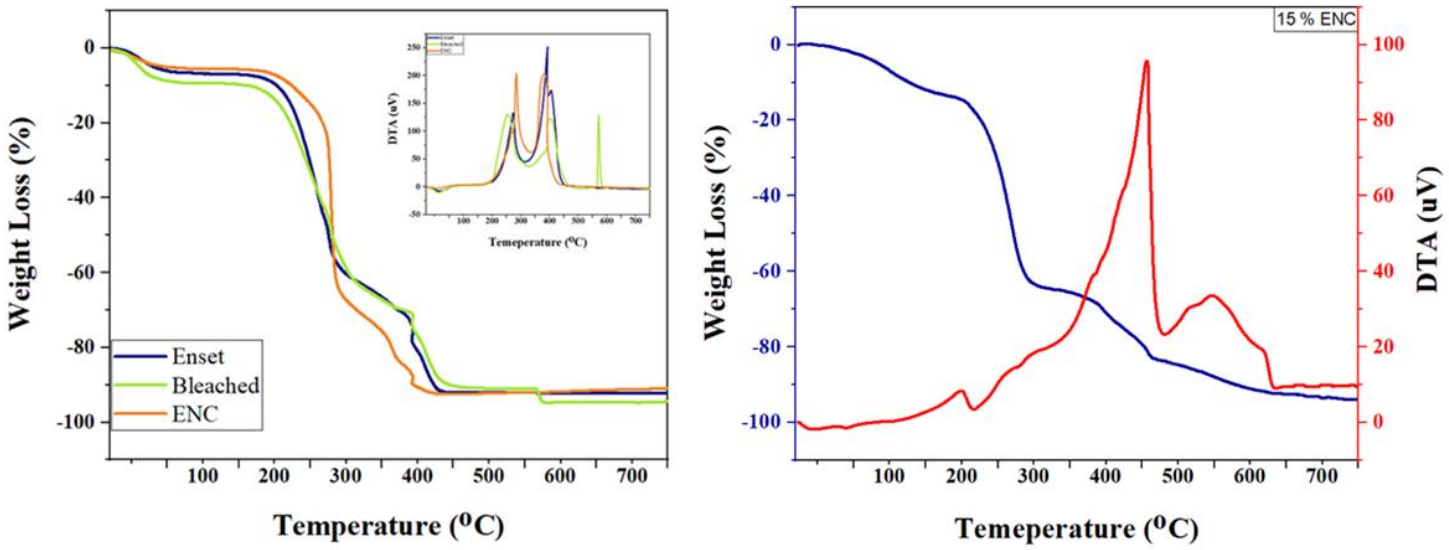
The TGA and DTA results, presented in figure 4.4, display the thermal degradation curves for the different enset samples. The TGA curves illustrate the weight loss of the samples as they are heated, while the DTA curves indicate the heat flow associated with the thermal transitions.

All samples exhibit a minor weight loss (<10%) within the temperature range from ambient to 150°C, primarily due to the evaporation of free moisture and the elimination of low molecular weight components, corresponding to the loss of absorbed water and volatiles. The major decomposition stage occurs between 185°C and 400°C, representing the degradation of cellulosic components through processes like depolymerization, dehydration, and glycosidic bond cleavage. The TGA curves reveal distinct differences in the thermal degradation patterns between raw, alkali-treated, bleached enset fibers, and isolated ENCs. The onset temperature of thermal degradation for ENCs is noted at 185.63°C, which is lower than that of bleached cellulose (247.5°C), alkali-treated fiber (238°C), and raw enset fiber (192.5°C), indicating that ENCs begin to degrade at a lower temperature compared to the other samples. The maximum degradation temperature (Tmax), which is the temperature at which the maximum rate of weight loss occurs, is observed to be 325.63°C for ENCs, lower than bleached cellulose (352.50°C), alkali-treated fiber (338.50°C), and raw fiber (327.50°C). The lower Tmax for ENCs suggests that they degrade more rapidly upon heating(Puglia et al., 2024; Teli & Terega, 2017).

The thermal degradation behavior of the samples is heavily influenced by the presence of different chemical constituents such as hemicellulose, cellulose, and lignin. The higher onset temperatures for bleached and alkali-treated fibers are due to the removal of non-cellulosic components, primarily lignin and hemicellulose, which are known to degrade at lower temperatures(Mashego, 2016; Teli & Terega, 2017). The rapid degradation of ENCs, evidenced by their lower Tmax, can be attributed to the presence of negatively charged sulfate groups introduced during sulfuric acid hydrolysis. These sulfate groups lower the activation energy required for thermal degradation, making ENCs less thermally stable than their untreated counterparts. Compared to previously published studies, these findings provide a detailed comparative analysis of the thermal stability of differently treated enset fibers and highlight the specific thermal behavior of nanocellulose derived from enset.

The significant reduction in thermal stability for ENCs underscores the impact of chemical modifications at the nanoscale level, particularly the substitution of sulfate groups, which facilitates thermal degradation(Dube, 2022).

In summary, the TGA and DTA analyses offer key insights into the thermal stability of enset-derived fibers and nanocellulose, with the observed thermal behaviors closely tied to their chemical composition and structural modifications. These findings have implications for the potential application of these materials in environments where thermal stability is a critical factor.



A. B.

Figure 4. 4: TGA and DTA plots (A). For different treatment of enset and (B), For PVC and 15% enset reinforced PVC.

4.4. Morphological analysis

Figure 4.5 of the thesis discusses the SEM results, providing a detailed morphological analysis of enset fiber, raw cellulose, and enset nanocellulose (ENC). The SEM micrographs, presented in Figure 4.5, reveal significant differences between the morphologies of isolated ENCs and raw and processed enset fibers. The raw enset fiber exhibits a smoother surface due to the presence of extractives such as wax, pectin, hemicellulose, and lignin, as noted by (Dube, 2022).Upon chemical pretreatment, the treated enset fiber displays an uneven size and shape with a rough surface containing fiber bundle fragments. This change is primarily attributed to the chemical pretreatments that break down non-cellulosic components, removing the shielding layer that covers alpha cellulose(Kozłowski et al., 2020).

These shielding layers are predominantly composed of lignin and hemicellulose (Gan et al., 2020).The rough surface and increased porosity observed post-treatment enhance the penetration of sulfuric acid during acid hydrolysis, facilitating the breakdown of cellulose into ENCs. The SEM images demonstrate the distinct rod-like morphology of cellulose nanocrystals and some clusters of ENCs, as described by(Listyanda et al., 2020). The study emphasizes that the pretreatment methods and their conditions significantly impact the characteristics and production of ENCs.

The detailed SEM analysis analyzes the transformative effects of chemical treatments on the structural properties of enset fibers, highlighting the enhanced porosity and roughness that contribute to more efficient acid hydrolysis and nanocellulose formation. The transformation is particularly notable in the ENCs, which display a rod-like morphology characteristic of cellulose nanocrystals, along with some degree of clustering. These morphological changes are crucial because they enhance the effectiveness of acid hydrolysis, improving the accessibility of sulfuric acid to the cellulose fibers. This accessibility is due to the increased porosity and roughness, facilitating deeper acid penetration and more efficient hydrolysis. The removal of non-cellulosic materials not only alters the fiber surface but also plays a significant role in improving the material properties of the final nanocellulose product. The increased surface area and porosity are beneficial for applications that require high reactivity and interaction with other substances, such as in composite materials or as biodegradable fillers(Dube, 2022; Listyanda et al., 2020; Teli & Terega, 2017).

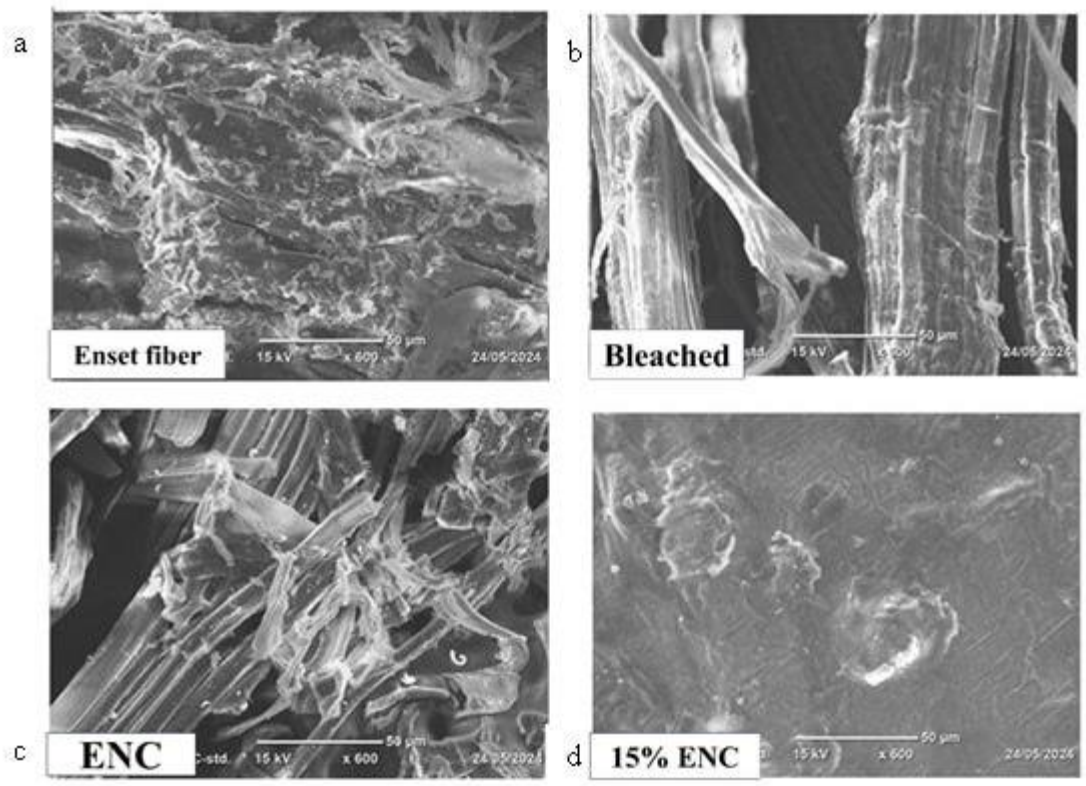


Figure 4.5: SEM characterization result different treatment phases of enset of (A).enset (b).Bleached (c).ENC and (d).15% ENC

4.5. Biodegradability result

Figure 4.6 provides analysis of the biodegradability results of polyvinyl chloride (PVC) composites reinforced with enset nanocellulose (ENC). The study evaluates the degradation behavior of these composites under different environmental conditions, including soil, water, 30% (H_2SO_4, NaOH, and NaCl) environments over a period of 25 days and 1 day for water absorption test.

The results indicate that as the concentration of cellulose in the PVC composites increases, the rate of biodegradation also increases. This is because nanocellulose acts as biodegradable filler that microorganisms in the soil can break down, thus facilitating the degradation process. Composite samples with higher ENC content showed greater weight loss compared to those with lower ENC content, with the 25% ENC/PVC composite exhibiting the highest degradation rates.

In soil, the composites experienced the highest weight loss, attributed to the rich presence of microorganisms, essential nutrients, and mechanical stress, all of which enhance biodegradation. In water, significant weight loss was observed, although less than in soil, due to the increased surface area available for water absorption, which improved compatibility and dispersion. In an acidic environment (30% H_2SO_4), the composites showed moderate degradation. Sulphuric acid treatment breaks down hemicellulose and lignin, making cellulose more accessible to microbes. The alkaline environment (30% NaOH) also influenced the biodegradation process, with the composites showing similar weight loss to the acidic conditions due to the breakdown of complex structures. In a saline environment (30% NaCl), the composites exhibited the least weight loss, likely due to the lack of mechanical stress and fewer microorganisms(Kaczmarek & Bajer, 2007).

The results showed that soil tests demonstrated the highest biodegradation rates, followed by water, sulphuric acid, sodium hydroxide, and sodium chloride. This variation in degradation rates shows the influence of specific environmental factors on the biodegradability of the composites. For instance, soil's mechanical stress can create micro-cracks in the composite materials, increasing the surface area for microbial attack and enhancing biodegradation(Kaczmarek & Bajer, 2007; Kadry & Abd El-Hakim, 2015).

This result shows that incorporating enset nanocellulose into PVC significantly enhances the biodegradability of the composite materials, particularly in soil environments. The study provides valuable insights into the potential for producing more environmentally friendly materials by enhancing the biodegradability of nanocellulose.

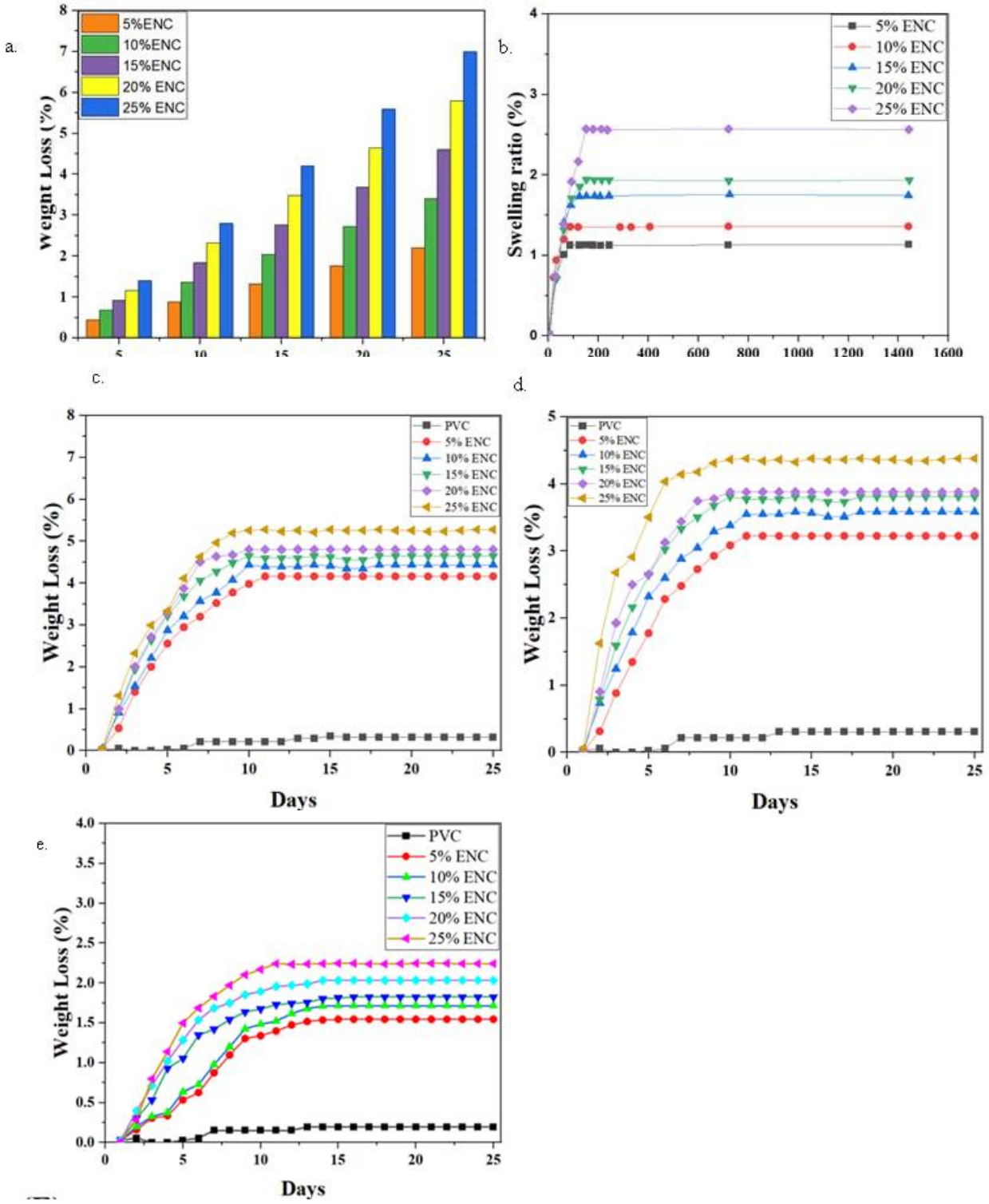
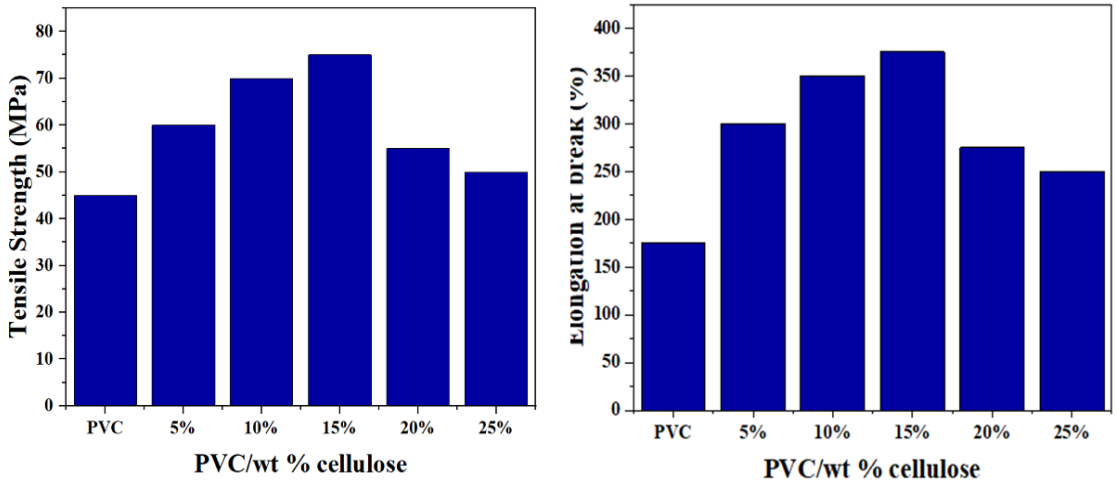


Figure 4. 6: Biodegradability result of PVC and the composites in (A) Soil, (B) Water absorption (C) 30% H_2SO_4, (D) 30%NaOH, (E) 30%NaCl

4.6. Mechanical test results



(A). (B).

Figure 4. 7: The value of mechanical properties (A). tensile strength and (B).elongation % at break of nanocomposites

Figure 4.7 discusses the mechanical test results, specifically tensile strength and elongation at break, of PVC composites reinforced with varying percentages of enset nanocellulose (ENC). The goal is to evaluate how the incorporation of nanocellulose affects the mechanical properties of PVC.

The tensile strength of the PVC composites increases significantly with the addition of nanocellulose, peaking at 20% ENC concentration with a maximum tensile strength of 75 MPa. This enhancement is due to the strong interfacial interaction between the nanocellulose fibers and the PVC matrix, which improves load transfer efficiency and reinforces the composite structure. However, beyond 20% ENC, the tensile strength begins to decrease slightly. According to (Ng et al., 2017) this due to the agglomeration of nanocellulose particles at higher concentrations, which creates stress concentration points and weakens the composite structure.

The elongation at break of the PVC composites also improves significantly with the addition of nanocellulose, reaching its highest at 15% ENC with an increase of 365.7%. This increase in elongation is due to the flexibility imparted by the dispersed nanocellulose within the PVC matrix, allowing the composite to withstand greater deformation before breaking. Similar to tensile strength, the elongation at break decreases beyond 20% ENC due to nanocellulose agglomeration, which limits the mobility of the polymer chains and reduces the material's ductility(Listyanda et al., 2020; Ng et al., 2017).

CHAPTER FIVE

5. CONCLUSION AND RECOMMENDATION

5.1. Conclusion

In this work, enset fiber reinforced PVC composites were successfully made using solvent casting method with different weight percentages of nanocellulose (5-25%). nano cellulose was prepared by acid hydrolysis from enset fiber waste and used as reinforcement which the results showed that PVC/ENC blend films was found to have good dispersion of the nanocellulose in the PVC to form nanocomposites with acceptable biodegradation and mechanical properties are achievable. The acid hydrolysis method can be used for industrial production of nanocellulose due to low cost, easy and fast

XRD patterns clearly showed that crystallinity increases with sequential chemical pretreatments, demonstrating the efficacy of methods in eliminating non-cellulosic components.SEM images confirmed the strong interfacial bonding of nanocellulose into the PVC nanocomposite and effective removal of the amorphous region during the nanocellulose preparation. The Fourier transform infrared spectroscopy (FT-IR) result suggests a reduction and total removal of lignin, hemicellulose, and other amorphous regions of enset fibers. It was found the biodegradation of the prepared PVC nanocomposites increases with the increasing amount of enset nanocellulose in the nanocomposites.

In addition to this, utilization of enset fiber play significant role on pollution and health issues by reducing the use of synthetic fiber in composite and can be used as reinforcement for PVC matrix for packaging application without affecting the desired mechanical properties of the structure in light weight structural application. renewable feedstock utility, cost efficiency, easy processability, less energy consumption, non-hazardous and easy degradability makes the product available in the market with lesser price than the market competitors. In general, this study has provided enough evidence to showcase enset fiber was a good new alternative natural fiber till now not effetely utilized by an industry and as a best lignocellulosic feedstock for producing and biodegradable, good tensile strength and low cost nanocellulose composite of PVC. Therefore, it is concluded that this study greatly supports the government’s mission of a green environment.

5.2. Recommendations

- Identifying the molecular weight of the extracted cellulose is an important matter, as it will provide valuable information about the degree of polymerization (DP) that is the number of monomeric units in a macromolecule or polymer of the nanocellulose. With the molecular weight known, strategies can then be developed to enhance the DP of the nanocellulose. As future work, the plan is to determine the molecular weight of the synthesized nanocellulose using appropriate analytical techniques, such as gel permeation chromatography (GPC) or viscometry. Once the molecular weight is identified, various methods can be explored to increase the DP of the nanocellulose.

- Research on the combination of PVC and enset nanocellulose offers a potential solution to improve biodegradability while managing mechanical properties. Increasing enset nanocellulose content in PVC formulations can enhance biodegradability, but excessive incorporation may decrease tensile strength. Excessive nanocellulose content can lead to aggregation or low dispersion of nanofillers, affecting the PVC matrix's tensile strength. Optimizing nanocellulose content in PVC-Enset nanocellulose composites could help design eco-friendly materials with tailored properties while minimizing environmental impact.

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