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Dedications

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Abbreviations and Symbols

PMCs	Polymer matrix composites.
MMCs	Metal matrix composites.
CMCs	Ceramic matrix composites.
SiC	Silicon carbide.
Al_2O_3	Alumina.
ZrO_2	Zirconia.
CVI	Chemical vapor infiltration.
CO_2	Carbon dioxide.
PLA	Polylactic acid.
WR	Water retting.
T	Traditional method.
NaOH	Sodium hydroxide.
H2SO4	Sulfuric acid.
FTIR	Transform infrared spectroscopy.
XRD	X-ray diffraction.
ASTM	American Society for Testing and Materials.
σ	Stress (MPa).
ε	Elongation (%).
E	Young's modulus (GPa).
E′	Storage modulus.
E″	Loss modulus.
Tg	Glass transition temperature.
tan δ	dissipation factor.
K_{cv}	Resilience.
CAD	Computer-aided design.
CI	Crystallinity index.
CS	Crystalline size.
λ	Wavelength of the X-ray radiation.
β	Width at half maximum (FWHM).
θ	Theta.
I_{am}	intensity of the amorphous peak.
I_{200}	Intensity of the crystalline peak.
PLA-WR1	PLA based bio-composite reinforced by 1% of yucca fiber extracted via water retting.

PLA-WR3	PLA based bio-composite reinforced by 3% of yucca fiber extracted via water retting.
PLA-T1	PLA based bio-composite reinforced by 1% of yucca fiber extracted via traditional method.
PLA-T3	PLA based bio-composite reinforced by 3% of yucca fiber extracted via traditional method.
UTY	Untreated yucca fiber.
TN3	Treated yucca fiber at 3% NaOH.
TS1	Treated yucca fiber at 1% sulfuric acid.
EUY	Epoxy based bio-composite reinforced by untreated yucca fiber.
EYN3	Epoxy based bio-composite reinforced by treated yucca fiber at 3% NaOH.
EYS1	Epoxy based bio-composite reinforced by treated yucca fiber at 1% sulfuric acid.

General introduction

General introduction

In the era of the world, the environmental crisis caused by the intensive exploitation of fossil resources and the proliferation of plastic waste is a major challenge for humanity [1]. Synthetic materials, although high-performance, are a major contributor to the pollution of terrestrial and marine ecosystems, with an estimated degradation time of several hundred years[2]. Nevertheless, their ubiquity comes with an exorbitant ecological cost: according to the OECD, nearly 460 million tonnes of plastic were produced in 2023, of which less than 10% is recycled, with the rest polluting the oceans, soil and atmosphere via microplastics and CO_2 emissions. This crisis, coupled with the depletion of fossil resources, has catalyzed a global awareness, embodied in international agreements such as the European Green Pact or COP28, which promote a transition to a circular, low-carbon economy. In this context, bio-based materials are emerging as a key solution [3]. Among them, biocomposites, combining a polymer matrix (biodegradable or biosourced) and natural reinforcements, are attracting growing interest. Their potential lies in their ability to reconcile technical performance and sustainability, while making the most of renewable resources such as plant fibers [4]. However, there are major scientific challenges to their widespread adoption, not least the variability of natural fiber properties, which is directly influenced by extraction methods.

Biocomposites combine the advantages of polymers (flexibility, malleability) and natural fibers (mechanical strength, lightness). Their main advantage lies in their partial or total biodegradability, depending on the nature of the matrix, and their low production energy compared with synthetic composites [5]. Nevertheless, their development comes up against technical challenges: variability of fiber properties, sensitivity to humidity, and limited adhesion between the fiber and the matrix. These limitations, often linked to the heterogeneous structure of the fibers and to extraction methods, affect their performance in service. Despite these constraints, their applications are diversifying, from lightweight automotive parts to biomedical implants and thermal insulation materials [6, 7]. Nonetheless, their industrial deployment is hampered by intrinsic limitations. The hygroscopicity of natural fibers, due to the presence of hydroxyl groups (-OH) in the cellulose, compromises their dimensional stability and their adhesion to the polymer matrix. Furthermore, their mechanical strength (Young's modulus of 10-70 GPa, compared with 70-300 GPa for carbon fiber) varies significantly depending on their botanical origin, their maturity and, above all, their extraction method. These parameters, which are often overlooked in

the literature, nevertheless determine the reproducibility of biocomposites, a key issue for their standardization [8].

Natural fibers, as reinforcements for biocomposites, are attracting growing interest due to their abundance, low density and intrinsic mechanical properties. Derived from renewable sources such as plants (cellulose, lignin) or agricultural residues, they have a lower carbon footprint than synthetic fibers and are more biodegradable at the end of their life [9]. The plant fibers that have been most extensively studied include flax, hemp, jute, sisal and, more recently, yucca fiber, the properties of which vary according to growing conditions, extraction method. The architecture of natural fibers is based on a complex hierarchical structure, with crystalline cellulose playing a key role in their rigidity and tensile strength. However, the presence of hemicelluloses and lignin, although conferring a certain cohesion to the fibers, limits their interaction with polymer matrices due to their hydrophilicity [10]. As a result, chemical treatments such as alkaline hydrolysis or acid oxidation can optimize fiber-matrix adhesion, improving the overall mechanical performance of biocomposites. Despite these advances, the structural variability of natural fibers remains a major challenge for their industrial integration, requiring optimization strategies for both extraction and functionalization [11].

Extracting natural fibers is a key stage in their use as reinforcements for biocomposites. The aim of this operation is to isolate the fiber bundles from the plant structure while preserving or modifying certain physico-chemical properties that are essential for their integration into a polymer matrix. Extraction methods fall into three main categories: mechanical, chemical and biological, each with its own advantages and limitations depending on the intended application. In this context, mechanical extraction relies on processes such as defibration or grinding to separate the fibers without chemical alteration. Although economical and environmentally friendly, this method often produces fibers of heterogeneous size and a surface rich in residual impurities, which can hinder their adhesion to the polymer matrix. In addition, chemical extraction involves the use of chemical solutions to dissolve non-cellulosic components such as hemicelluloses and lignin. This approach produces purer fibers with optimized crystallinity and surface roughness, improving their compatibility with the polymer matrix. However, it requires rigorous control of reaction parameters to avoid excessive fiber degradation and to minimize the environmental impact of chemical effluents. Finally, biological extraction exploits enzymatic or microbial action to selectively degrade undesirable components. Water retting is a more environmentally-friendly alternative, preserving the fibrillar structure and limiting the use of aggressive chemicals. However, these processes require longer treatment times and are often less reproducible than chemical methods. The choice of

extraction method directly influences the morphology, chemical composition and mechanical properties of the fibers, and therefore the final quality of the biocomposites.

This thesis explores the impact of yucca fiber extraction methods on their physicochemical and mechanical properties, and then assesses how these variations affect the performance of biocomposites manufactured by two distinct processes: 3D printing (melt extrusion) and casting molding. The comparative approach adopted aims to identify synergies between extraction and shaping processes, in order to optimize biocomposites for targeted applications. The originality of this work lies in a multidisciplinary approach aimed at optimizing the use of natural yucca fibers in biocomposites. Firstly, an in-depth comparative analysis of four extraction methods was carried out in order to assess their contrasting effects on the microstructure and physico-chemical properties of the yucca fibers. Secondly, a hybrid approach combining additive manufacturing by 3D printing and conventional molding techniques was employed to examine the adaptability of the fibers in complex architectures. Finally, a multiscale characterization of the fibers and biocomposites was carried out, incorporating mechanical, thermogravimetric (TGA), microscopic (SEM) and spectroscopic (FTIR) analyses, enabling an in-depth assessment of their performance and their potential for industrialization.

This thesis is structured in four chapters:

- Chapter 1: Critical review of the literature on natural fibers, biocomposites, and the influence of extraction and manufacturing methods.
- Chapter 2: Detailed description of the experimental protocols (fiber extraction, biocomposite formulation, 3D printing and casting parameters), as well as the machines, analyses and standards used.
- Chapter 3: Yucca fiber characterization results, revealing the influence of extraction methods on chemical composition and mechanical properties.
- Chapter 4: Analysis of biocomposite performance (tensile strength, fracture behavior, moisture absorption), correlated with fiber characteristics and forming processes, as well as fiber extraction methods.

A general conclusion was written at the end of this thesis.

By establishing causal relationships between extraction, microstructure and performance, this thesis contributes to the design of tailor-made biocomposites, aligned with the imperatives of the circular

economy. It also provides the keys to standardizing extraction processes, a crucial issue if these materials are to be industrialized without sacrificing their durability.

Chapiter 1: Natural fibers and bio-composites: state of the art and recent advances

`Chapiter 1 – Natural fibers and bio-composites: state of the art and recent advances

1.1 Introduction

In a global context marked by the need to reduce the environmental impact of industrial materials, natural fibers and bio-composites are emerging as strategic solutions that meet the requirements of a sustainable economy [12]. Natural fibers, derived from renewable and abundant resources, offer a remarkable combination of lightness, mechanical strength and competitive cost. As reinforcements in bio-composites, they make it possible to design hybrid materials that combine technical performance with a reduced ecological footprint, placing these materials at the heart of scientific and technological advances [13].

In addition, the potential of natural fibers as reinforcements is intrinsically linked to their properties, which are themselves strongly influenced by the extraction methodology. This step, which is often underestimated, is a key lever for modulating the chemical composition, morphological structure and crystalline organization of fibers, thereby conditioning their interaction with the polymer matrix and their overall performance in bio-composites [14]. Extraction processes, modify the characteristics of the natural fibers, in terms of their crystallinity index, their compatibility with polymer matrices and their mechanical properties. Consequently, the extraction methodology plays a decisive role in optimizing matrix-fiber interactions, which in turn determines the final properties of the bio-composites [15].

With this in mind, this chapter provides a critical and detailed review of the literature on natural fibers and bio-composites, focusing on the influence of extraction methodologies on the final properties of the fibers and, by extension, on the performance of final bio-composites. The first section examines natural fibers from various aspects: their classification, chemical composition and physical-mechanical properties. The second section explores bio-composites as bio-based materials, detailing their structure, their specific advantages and their applications in various fields. Particular attention is paid to fiber treatment techniques, aimed at improving their compatibility with polymer matrices and overcoming their intrinsic limitations. Finally, the scientific and technological challenges associated with integrating natural fibers into bio-composites are briefly reviewed, while identifying current gaps in the literature and opportunities for future research. This chapter also aims to provide a solid scientific basis for understanding the complex interactions between fiber extraction methodology and bio-composite properties.

1.2 Composite materials

1.2.1 Composite materials definition

Composite materials, frequently referred to simply as 'composites', refer to a class of materials made up of two or more distinct components, combined to produce a new material with properties superior to those of its individual constituents. These components are generally divided into two main phases, matrix and reinforcement [16].

1.2.2 Matrix

In composite materials, the matrix represents the continuous phase, and plays a central role in the structure and mechanical behavior of the final material. It envelops and binds the reinforcing elements (fibers, particles, etc.) while ensuring uniform stress distribution and protection against aggressive environments. The matrix materials are typically polymers, metals or ceramics, and their role is essential in guaranteeing the overall performance of the composite [17].

1.2.3 Matrix classification

Composite matrices are essential for the performance and structural integrity of advanced composites. Several types of matrices exist, including polymeric, metallic and ceramic matrices, each with unique properties that make them suitable for a variety of industrial and technological applications [18].

1.2.3.1 Polymeric matrices

Polymer matrix composites (PMCs) are the most popular due to their light weight, ease of use and relatively low cost [19]. They can be subdivided into:

- **Thermosetting polymers:**

Thermosetting polymer matrices are widely utilized in the production of composites due to their exceptional properties, particularly their outstanding thermal, mechanical, and chemical resistance. These polymers undergo an irreversible curing process when exposed to heat or a hardener, resulting in the formation of a three-dimensional cross-linked structure [20]. This characteristic imparts remarkable thermal stability, making them particularly well-suited for applications requiring high temperature resistance. Additionally, thermosetting polymers demonstrate strong resistance to a broad spectrum of chemicals and moisture, rendering them ideal for use in chemically aggressive environments. Moreover, thermoset polymers exhibit exceptional mechanical strength, particularly in tensile, compressive, and bending tests, owing to their cross-linked network, which effectively prevents plastic deformation and

ensures long-term structural integrity [21]. Common examples of thermoset matrices include epoxy, polyester, and vinyl ester, which are extensively employed in the fabrication of high-performance composites. These materials find critical applications across diverse sectors, including aerospace, automotive, shipbuilding, and protective equipment manufacturing, where durability, resistance to extreme conditions, and structural resilience are essential [22].

- **Thermoplastic polymers:**

Thermoplastic polymers constitute a crucial category of materials in the field of composite manufacturing, valued for their distinct processing advantages, mechanical properties, and recyclability. Unlike thermosetting polymers, thermoplastics do not undergo an irreversible curing process but instead soften when heated and harden upon cooling, allowing for multiple cycles of melting and reshaping [23]. This thermoplastic behavior facilitates diverse manufacturing techniques such as injection molding, extrusion, and additive manufacturing, making these materials highly adaptable for large-scale industrial applications. Thermoplastic polymers exhibit a well-balanced combination of mechanical properties, including tensile strength, impact resistance, and ductility, which can be further enhanced through various reinforcement strategies, such as fiber loading. Additionally, thermoplastics offer superior resistance to chemical degradation, environmental factors, and moisture absorption, making them suitable for use in challenging operational conditions [24]. Notably, certain thermoplastics, such as polyetheretherketone (PEEK), polyamide (PA), and polycarbonate (PC), demonstrate exceptional thermal stability and resistance to aggressive chemicals, further enhancing their applicability in aerospace, automotive, and industrial sectors. Moreover, the inherent recyclability of thermoplastic polymers provides an added environmental benefit, contributing to sustainability in material selection. As a result, thermoplastic matrices are extensively employed in high-performance composite applications, where their ease of processing, mechanical performance, and durability under varied environmental conditions make them invaluable materials for cutting-edge technological applications [25].

1.2.3.2 Metallic matrices

Metal matrix composites (MMCs) are a class of composite materials wherein a metal matrix is reinforced with fibers or particles to enhance their mechanical, thermal, and electrical properties. These composites leverage the high-temperature strength and dimensional stability of metals, while incorporating the mechanical strength, wear resistance, and sometimes corrosion resistance of reinforcing materials [26]. Common metal matrices, such as aluminum, copper, titanium, and magnesium, are extensively utilized in industries such as aerospace, automotive, and electronics, owing to their lightweight

nature, excellent thermal conductivity, and ability to endure extreme environmental conditions [27]. The fabrication of MMCs typically involves processes such as melting and casting, compression molding, and coating, all of which contribute to the development of materials with superior performance characteristics. Reinforcements, including ceramic fibers and silicon carbide particles, play a pivotal role in significantly improving the wear resistance and overall durability of MMCs. These composite materials are widely applied in high-performance sectors, where the integration of light weight, mechanical strength, and thermal conductivity is paramount for advancing technological capabilities [28].

1.2.3.3 Ceramic matrices

Ceramic matrix composites (CMCs) represent a class of advanced materials wherein a ceramic matrix is reinforced with fibers or particulates to enhance their mechanical, thermal, and environmental performance. These composites combine the intrinsic high-temperature stability, wear resistance, and chemical inertness of ceramics with the enhanced toughness, fracture toughness, and damage tolerance provided by the reinforcing phases [29]. Common ceramic matrices, such as silicon carbide (SiC), alumina (Al_2O_3), and zirconia (ZrO_2), are extensively employed in demanding industries such as aerospace, automotive, and energy, due to their ability to maintain structural integrity under extreme thermal, mechanical, and corrosive conditions. These materials exhibit exceptional resistance to high-temperature oxidation, thermal shock, and chemical degradation, making them particularly suitable for applications in turbine engines, brake systems, and thermal protection systems[30]. The fabrication of CMCs typically involves sophisticated processing techniques such as chemical vapor infiltration (CVI), hot pressing, reaction bonding, and slip casting, all of which facilitate the development of composite materials with superior mechanical strength, dimensional stability, and resistance to failure under stress [31].

1.2.4 Reinforcement

Reinforcements within composite materials are integral to significantly enhancing the mechanical, thermal, and wear-resistance properties of the matrix. These reinforcements, typically incorporated in the form of fibers, particles, or nanotubes, are strategically selected to confer specific, desirable characteristics to the composite, such as increased stiffness, elevated tensile strength, enhanced flexibility, or improved thermal conductivity [32]. The primary function of reinforcements is to augment the matrix's capacity to endure external loads, thus improving its structural integrity and enabling the composite to better withstand various operational stresses. This not only bolsters the mechanical performance but also contributes to the composite's overall durability, longevity, and resilience. The selection of appropriate reinforcements is highly contingent upon the nature of the matrix and the specific requirements of the

intended application [33]. For instance, various reinforcements can be employed to optimize particular properties, such as mechanical strength under tension, impact resistance, or thermal and chemical stability, depending on the performance criteria. Moreover, reinforcements play a pivotal role in influencing additional critical properties, including thermal and electrical conductivity, as well as enhancing corrosion resistance. This versatility allows for the tailoring of composite materials to meet the rigorous demands of diverse, often extreme, operating environments. By carefully selecting and incorporating suitable reinforcement materials, it is possible to precisely modulate the composite's performance to achieve optimal functionality for high-performance applications, thus broadening the scope of their use in advanced engineering sectors [34].

Regarding fibers, a variety of options are available to meet specific needs, including carbon fibers, glass fibers, and natural fibers such as flax, jute, and hemp (**Figure 1.1**). Each fiber type offers distinct advantages in terms of lightness, strength, and environmental impact, making them suitable for diverse industrial, aerospace, and ecological applications. The selection of fiber type is crucial, as it not only determines the mechanical properties of the composite but also influences its environmental footprint, an increasingly important consideration in sustainable material design [35].

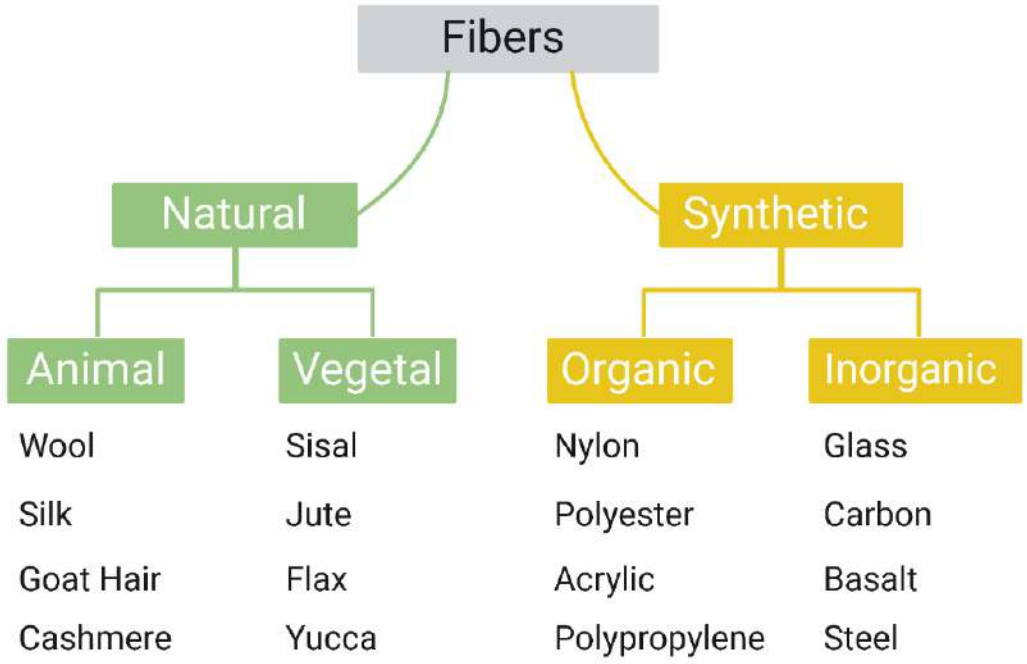

Figure 1.1 - Fiber reinforcement types for composite applications.

1.2.4.1 Carbon fiber

Renowned for their exceptional properties, carbon fibers are high-performance reinforcements that stand out for their lightness, unrivalled rigidity and exceptional tensile strength. Manufactured by the controlled pyrolysis of organic precursors such as polyacrylonitrile (PAN), these fibers offer not only remarkable thermal and chemical stability, but also enhanced resistance to fatigue and corrosion, making them the preferred choice in cutting-edge sectors such as aeronautics, the automotive industry and renewable energies, where the search for materials that combine lightness, robustness and longevity is essential[36].

However, carbon fiber production is energetically intensive, generating significant environmental impacts, particularly in terms of CO_2 emissions and production waste. In addition, their non-biodegradable nature and the complexity of their recycling pose major challenges at the end of their life. To address these concerns, efforts are underway to optimize manufacturing processes, reduce their environmental footprint and develop efficient recycling technologies, particularly chemical and mechanical recycling [37, 38].

1.2.4.2 Glass fiber

Glass fibers, the mainstay of composite materials, are synthetic reinforcements that are prized for their remarkable performance, affordability and versatility. Derived from the fusion of silica, they have a unique combination of lightness, tensile strength and exceptional thermal and chemical resistance [39]. These characteristics make glass fibers an essential choice in sectors such as the automotive, construction, aeronautical and protective equipment industries, in which durability and reliability are paramount. Their ability to enhance dimensional stability while limiting thermal conductivity also makes them a material of choice for a variety of applications [40, 41].

Although energy-intensive to produce, with a relatively smaller carbon footprint than other reinforcement materials such as carbon fiber, glass fiber offers a more environmentally friendly solution. However, it is not without its ecological challenges, particularly when it comes to recyclability [42].

1.3 Natural fibers: a renewable resource

1.3.1 Definition and classification

Natural fibers are filamentous materials derived from biological sources, including plants, animals, and minerals. These fibers are characterized by their ability to be processed into yarns or directly used as

reinforcements in composite materials [43]. Their composition is primarily based on natural polymers, such as cellulose in plant (vegetal) fibers, proteins like keratin or fibroin in animal fibers, and other organic or mineral constituents. This unique composition provides natural fibers with a balance of desirable mechanical properties, such as tensile strength, flexibility, and low density, making them highly versatile materials for a broad spectrum of applications [44].

A key advantage of natural fibers lies in their renewable origin and biodegradability, which align with the global emphasis on sustainability and environmental responsibility. Their ability to replace synthetic fibers in various industries, including textiles, automotive, construction, and packaging, highlights their potential as eco-friendly alternatives [45]. Moreover, their compatibility with bio-based and synthetic matrices enhances their utility in composite materials, where they provide reinforcement while reducing the environmental footprint of the final product [46].

Natural fibers are classified according to their biological origin into two main categories: vegetal fibers and animal fibers, as shown in **Figure 1.2**.

Figure 1.2 - Natural fiber classification.

1.3.1.1 Vegetal natural fibers

Vegetal fibers are filamentous lignocellulosic materials of biological origin, extracted from various parts of plants. These fibers are distinguished by their intricate hierarchical structure, primarily composed of cellulose, hemicellulose, and lignin. Additionally, they contain secondary components such as pectins, waxes, and mineral ashes, which contribute to their unique properties and functional versatility [47].

One of the defining characteristics of vegetal fibers is their specific set of properties, including low density (ranging from 1.2 to 1.5 g/cm^3), high mechanical strength (reaching up to 1000 MPa), limited elasticity, and biodegradability. These attributes make them particularly attractive for sustainable applications, where lightweight and environmentally friendly materials are required [48].

Structurally, vegetal fibers are composed of longitudinally aligned fibrous cells encased in an intercellular matrix of pectin and lignin, which ensures cohesion between the cells. Each fiber cell is further organized into a primary cell wall and multiple secondary walls. Within these walls, cellulose microfibrils are arranged at precise angles to the longitudinal axis, creating anisotropic mechanical properties that vary along different directions of the fiber [49]. The structural complexity of vegetal fibers is influenced by several factors, including their botanical origin, growth conditions, extraction techniques, and post-extraction treatments, all of which affect their mechanical, thermal, and chemical behavior [50, 51].

From a functional perspective, vegetal fibers find widespread use in various industrial sectors, particularly in the development of bio-composites. Their natural compatibility with both bio-based and synthetic polymer matrices enhances their utility in lightweight, high-performance, and sustainable materials. The integration of vegetal fibers into composite materials not only improves mechanical properties such as stiffness and strength but also reduces the environmental footprint of the final products [52].

1.3.1.2 Animal natural fibers

Animal fibers are filamentous materials of biological origin obtained from various animal sources, such as hair, wool, silk, or secretions. They are primarily composed of structural proteins, including keratin (found in wool and hair), fibroin (the main component of silk), and collagen (in materials like animal hides) [53]. These proteins give animal fibers their distinct properties, such as high elasticity, excellent thermal insulation, and softness, which make them highly valued in diverse applications, particularly in the textile industry. Secondary components, such as lipids, pigments, and mineral residues, may also be present and contribute to the fiber's unique characteristics [54].

1.3.2 Properties of natural fibers

Natural fibers, primarily sourced from biological materials, exhibit a unique set of physical, chemical, and mechanical properties that render them highly suitable for various industrial applications, particularly in the reinforcement of composite materials [55]. These fibers are inherently diverse, with their properties being heavily influenced by factors such as their botanical or animal origin, cellular structure, and the methods of harvesting and extraction. The effectiveness of natural fibers as reinforcements is largely governed by their chemical composition, molecular organization, and the processing treatments they undergo [56]. Therefore, a detailed and systematic evaluation of these properties is essential to determine the feasibility and potential of natural fibers in the development of high-performance, environmentally sustainable composite materials.

1.3.2.1 Morphological and structural properties

The morphology of natural fibers is a fundamental parameter that governs their behavior and performance in composite materials. These fibers possess a highly organized hierarchical structure, with cellulose microfibrils forming helical arrangements embedded within a matrix of hemicellulose and lignin [57]. This architectural complexity provides the fibers with their intrinsic tensile strength, rigidity, and mechanical stability. The dimensions of natural fibers, including their length, diameter, and aspect ratio, are highly variable and depend on the specific plant source and fiber type. For example, flax and jute fibers are characterized by their long, continuous lengths, making them particularly advantageous in applications requiring high tensile strength and toughness [58]. Conversely, shorter fibers such as hemp and ramie, while less continuous, still exhibit commendable mechanical properties suitable for various composite applications [59].

1.3.2.2 Mechanical properties

The mechanical properties of natural fibers are primarily governed by their chemical composition and structural morphology. Cellulose, with its high crystallinity, provides significant tensile strength and stiffness, while hemicellulose and lignin influence flexibility and compressive strength [60, 61]. Factors such as moisture content**,** water absorption, and extraction methods further affect their mechanical performance. Although natural fibers often exhibit lower tensile properties than synthetic fibers, they offer advantages in cost, sustainability, and moderate mechanical performance. To illustrate these variations, the **Table 1.1** summarizes key mechanical properties of commonly used natural fibers.

Table 1.1- Mechanical properties of different natural fibers reported in the literature.

Fiber type	Tensile strength (MPa)	Elongation (%)	Youngs modulus (GPa)	References
Sisal	415	6.43	9.89	[62]
Alfa	862.62	3.77	22.8	[63]
Pineapple	413	1.6	6.5	[64]
Palm date	117	3.13	4.3	[65]
Flax	300	1.1	30	[66]

1.3.2.3 Thermal properties

The thermal properties of natural fibers are mainly dictated by their chemical composition and complex structure. These fibers, rich in cellulose, hemicellulose and lignin, exhibit distinct thermal degradation behaviors attributable to the individual characteristics of each constituent. Cellulose, a crystalline polymer, demonstrates relatively high thermal stability, with degradation initiating around 300°C, driven by the cleavage of glycosidic bonds between glucose units [67]. In contrast, hemicellulose is an amorphous polymer that exhibits lower thermal stability, with degradation typically commencing between 200°C and 250°C. This process is associated with the breakdown of polysaccharide chains and the release of volatile decomposition products. Lignin, a complex aromatic polymer, degrades over a broader temperature range, typically between 250°C and 500°C, undergoing a series of intricate thermal processes that generate both carbonaceous residues and volatile compounds. Due to the combined presence of these components, natural fibers exhibit limited thermal stability when compared to synthetic materials [68].

1.3.3 Chemical composition

The chemical composition of natural fibers encompasses both organic and inorganic constituents, which collectively determine their physical, mechanical, and chemical properties. The principal components of biofiber, as illustrated in **Figure 1.3**, include cellulose, hemicellulose, and lignin, with additional minor constituents such as waxes, pectin, and other extractable compounds. Each of these components contributes to the distinct characteristics of the fiber, influencing key properties such as stiffness, tensile strength, and durability. Moreover, the chemical composition plays a significant role in the fiber's ability to interact with various polymer matrices in composite materials, thereby affecting the performance, stability, and sustainability of the resulting bio-composites [61].

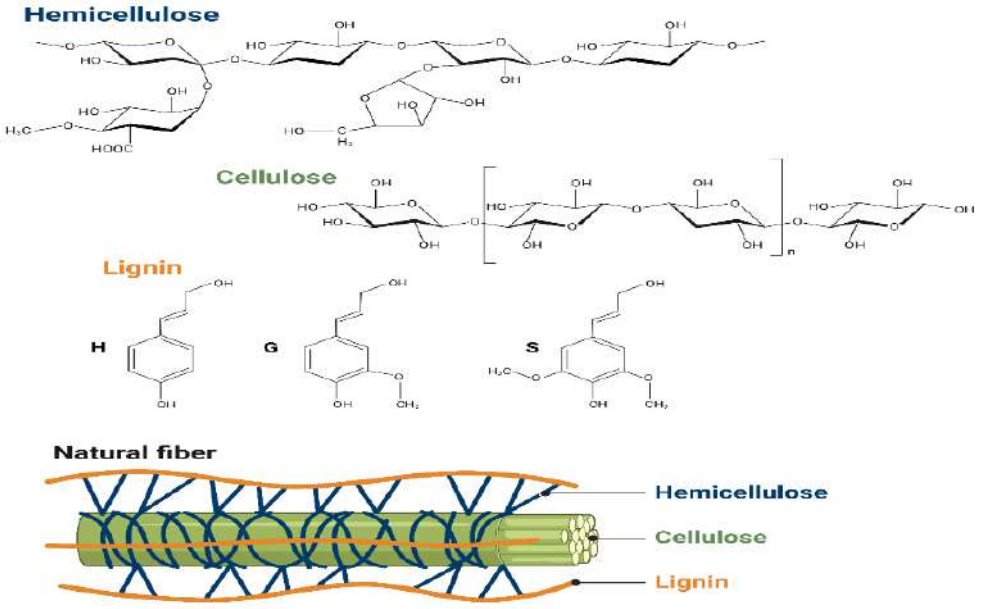

Figure 1.3 – Natural fiber chemical composition with details of the three essential components.

1.3.3.1 Cellulose

Cellulose is a linear polysaccharide consisting of repeating β-D-glucose units linked by β(1→4) glycosidic bonds. It is the primary structural component of plant cell walls, accounting for approximately 40% to 80% of the total weight of natural fibers [69]. This abundance, coupled with its exceptional structural properties, underscores cellulose's pivotal role in natural fibers performance. Its highly ordered crystalline structure, reinforced by a dense network of strong intra- and intermolecular hydrogen bonds, imparts outstanding mechanical properties, including remarkable tensile strength, stiffness, and dimensional stability. These features are critical for the ability of natural fibers to withstand mechanical stresses and maintain their structural integrity.

1.3.3.2 Hemicellulose

Hemicellulose is a heterogeneous polysaccharide composed of various sugar monomers, including glucose, xylose, mannose, arabinose, and galactose, linked through a combination of β(1→4) and β(1→3) glycosidic bonds. Unlike cellulose, hemicellulose exhibits a branched, amorphous structure, which makes it less crystalline and more soluble in water. It constitutes between 20% and 30% of the dry weight of natural fibers and plays an essential role in the architecture of plant cell walls. Acting as a matrix that binds cellulose microfibrils and lignin, hemicellulose contributes to the cohesive structure of the cell wall and facilitates load distribution across its components [69].

From a mechanical perspective, hemicellulose enhances the flexibility and elasticity of natural fibers, complementing the rigidity provided by cellulose. This synergy is vital for maintaining the balance between strength and pliability in plant tissues. Furthermore, its partial hydrophilicity allows it to interact with polar matrix materials, making it a potentially valuable component in bio-composites. However, its lower thermal stability and susceptibility to hydrolysis under certain conditions can limit its application in high-temperature or high-moisture environments [70].

1.3.3.3 Lignin

Lignin is a complex three-dimensional polymer composed mainly of phenolic monomers such as p-coumaryl alcohol, coniferyl alcohol and sinapyl alcohol. Representing approximately 10% to 30% of the total weight of natural fibers, lignin plays an essential role in the structural reinforcement of plant cell walls. Its hydrophobic nature and remarkable chemical stability enable lignin to function as a natural adhesive, surrounding cellulose microfibrils and stabilizing hemicellulose chains. This unique structural arrangement contributes significantly to the mechanical properties of plant tissues, including enhanced rigidity, resistance to compressive forces, and protection against biological degradation, as well as environmental factors [71].

However, in the field of bio-composites, lignin presents certain drawbacks that must be addressed. Its hydrophobicity and low chemical reactivity often inhibit strong fiber-matrix adhesion, reducing the efficiency of load transfer at the interface and, consequently, the mechanical performance of the composite. Furthermore, its presence may result in reduced compatibility with polar matrices, thereby necessitating chemical modifications or pre-treatments to enhance interfacial bonding. As a result, a deeper understanding of lignin's influence on fiber-matrix interactions is essential for developing bio-composites with improved structural and functional properties [72].

1.3.4 Natural fibers advantages

As reinforcements for composite materials, natural fibers offer numerous ecological, economic and technical advantages. These properties are making them increasingly attractive in a range of industrial sectors, including automotive, construction, packaging and biomedical. The main advantages of using composite materials are:

- Renewable and biodegradable character: natural fibers come from renewable resources, and their ability to biodegrade naturally at the end of their life helps to reduce environmental waste.

- Reduction of the ecological footprint: the production of natural fibers emits less CO_2 and consumes less energy than synthetic fibers such as glass or carbon.
- Lightweight and low density: Natural fibers exhibit low density, which makes it possible to produce lighter composites. This characteristic is essential in sectors such as the automotive and aerospace industries, where weight reduction improves energy efficiency and reduces fuel consumption.
- Competitive mechanical properties: Some natural fibers, such as flax and hemp, have mechanical properties comparable to glass fibers, particularly in terms of tensile strength and rigidity.
- Good thermal and acoustic insulation properties: Natural fibers offer excellent thermal and acoustic insulation properties, making them ideal for applications in the construction industry.
- Compatibility with various types of matrices: Natural fibers can be incorporated into thermoplastic or thermosetting polymer matrices to form high-performance composites.
- Reasonable cost: The extraction and production processes are less complex and less costly, which reduces the total cost of the final materials.
- Abundant availability: Natural fibers are widely available throughout the world

1.4 Natural fibers extraction methods

The extraction of natural fibers is a critical process in their transformation into effective reinforcements for composite materials. This step is fundamental for isolating fibers from the complex matrix of plant tissues and significantly influences their mechanical, physical, and chemical properties. The efficiency and quality of the extracted fibers are highly dependent on the extraction method employed, making this stage essential for optimizing their structural integrity and ensuring their suitability for advanced composite applications [73].

1.4.1 Role and importance

The primary objective of fiber extraction is to isolate fiber bundles from the plant matrix, which consists of pectin, lignin, and hemicellulose. This separation is essential for unveiling the intrinsic characteristics of the fibers, such as their cellular architecture, length, diameter, and crystalline cellulose content, all of which play a pivotal role in their mechanical performance [73]. Furthermore, the success of the extraction process directly influences the purity and quality of the fibers, which are key parameters for their effective utilization in composite materials [14].

By removing unwanted components, such as non-cellulosic materials, fiber extraction enhances the compatibility and adhesion between the fibers and polymer matrices. This improved interfacial adhesion facilitates the efficient transfer of mechanical stresses from the matrix to the fibers, thereby optimizing the overall mechanical properties of the resulting composite. Moreover, advanced extraction techniques have been shown to reduce fiber variability, improve surface roughness, and increase fiber wettability, which are critical for ensuring uniform stress distribution within the composite. Thus, fiber extraction is not only a process of mechanical or chemical separation but also a decisive factor that dictates the final performance and durability of natural fiber-reinforced composites [74].

1.4.2 Vegetal fiber extraction location

The extraction of vegetal fibers involves a range of technical processes designed to obtain fibers that are sufficiently long, strong, and pure for various industrial applications, including bio-composite reinforcement. The extraction process is highly dependent on the specific type of plant, the structure of the fiber, and the particular requirements of the intended application. Vegetal fibers can be extracted from different parts of the plant, such as roots, stems, leaves, or seeds, as illustrated in **Figure 1.4**. In addition, the method chosen for extraction and the quality of the fibers obtained can vary significantly depending on which part of the plant is used [74, 75].

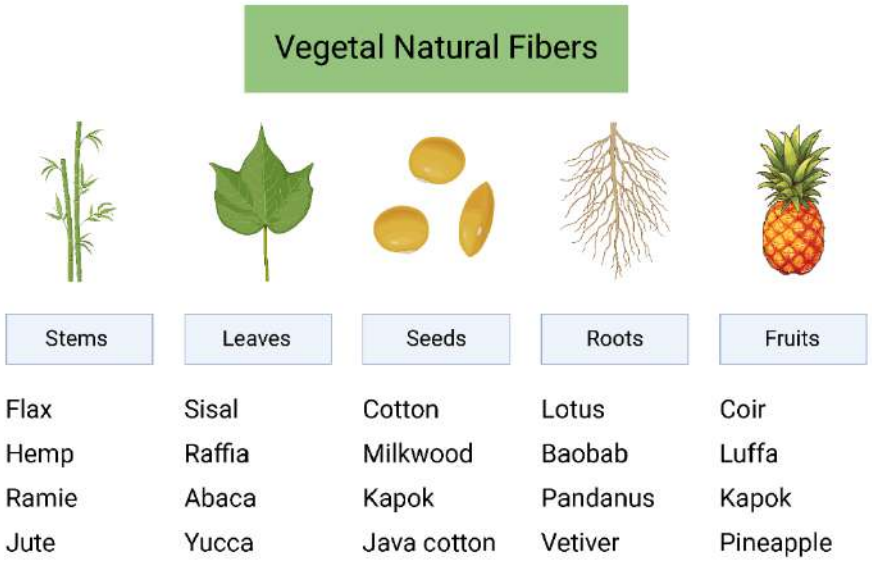

Figure 1.4 - Fiber location in different natural plants.

- **Stem fibers:** The most common plant fibers come from the stems of plants such as flax, jute, hemp and kenaf. These fibers are extracted mainly from the phloem, the vascular layer on the outside

of the stem, which contains long, strong fibers. The stalks are harvested and then subjected to an extraction method to separate the fibers from the straw.

- **Leaf fibers:** Some plants, such as sisal, yucca and aloe, produce fibers that are extracted from the leaves. These fibers, which are generally stiffer, are extracted after the leaves have been harvested.
- **Root fibers:** Less common but nonetheless important in certain applications, root fibers can be extracted from plants such as manioc and certain types of palms. These fibers are generally shorter and less solid than those extracted from stems or leaves.
- **Fruit fibers:** The fibers extracted from fruit generally come from the wall of the fruit or its internal tissues. These fibers are finer and often more flexible than those from stems and leaves.
- **Seed fibers:** The seeds contain fibers that can be extracted for specific industrial applications. These fibers are often fine, but they can be used in blends or as reinforcement in composite materials.

1.4.3 Vegetal extraction process

1.4.3.1 Mechanical methods

Mechanical extraction of vegetal fibers is a physical process that utilizes specific techniques and equipment to separate fibers from the plant matrix without involving chemical or biological treatments. This method relies on the application of mechanical forces to detach the fibers from non-fibrous components, such as hemicellulose, lignin, and other associated materials. It is widely employed due to its simplicity, rapidity, and lower environmental impact [76]. Depending on the plant part, equipment availability, and industrial needs, various techniques are utilized, including:

- **Combing**: Fibers are detangled and impurities removed using manual or mechanical combs.
- **Decortication**: Fiber bundles are isolated by removing outer layers (e.g., husks or cuticles) using defibration machines. This technique is particularly common for coconut and banana fibers, as illustrated in **Figure 1.5**.
- **Crushing**: Plant stalks or leaves are passed through rollers or crushers to break tissues and release the fibers.
- **Teasing**: Machines equipped with rollers or rotating claws gently separate fibers while preserving their length and structural integrity.

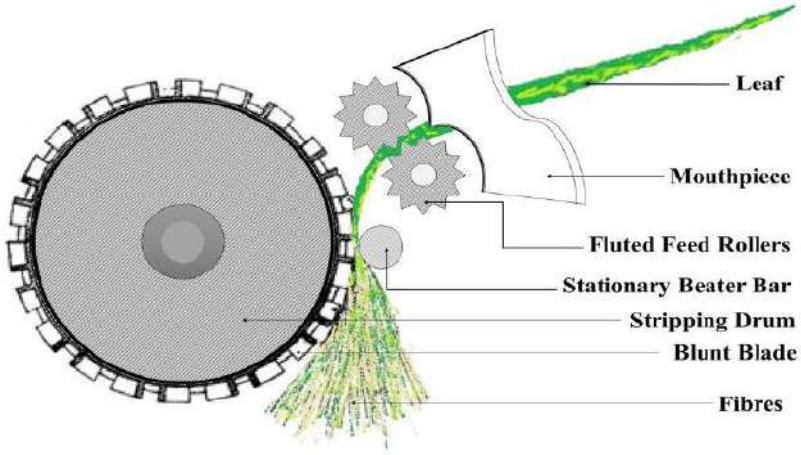

Figure 1.5- Decortication techniques to extract the natural fibers [77].

This family of methods is particularly effective for fibers derived from stems, leaves, or fruit, as it preserves fiber length and mechanical properties. However, like any extraction approach, it has its specific advantages and limitations (detailed in **Table 1.2**) that must be evaluated based on production objectives and the desired fiber characteristics.

Table 1.2- Advantages and limitation of natural fiber mechanical extraction method.

Advantages	Disadvantages
Fast method, suitable for large-scale production.	May cause physical damage to fibers if mishandled
No chemicals, reducing environmental impact and health risks for workers.	Requires specific machinery, involving high initial costs for equipment acquisition and maintenance.
Preserves fiber length and integrity, essential for textile and composite applications.	Extraction efficiency can vary depending on the plant and the quality of the equipment employed.
Suitable for many types of fiber.	Some fibers that adhere strongly to tissues require preliminary treatment to facilitate extraction.

1.4.3.2 Chemical methods

Chemical extraction of plant fibers relies on chemical agents such as strong bases, acids, or oxidizing agents to dissolve non-fibrous components like lignin, hemicellulose, and pectin, which hold the fibers within the plant matrix. By selectively breaking the chemical bonds in the plant structure, this process efficiently releases purified and homogeneous fibers, making it particularly suitable for advanced

applications in bio-composites and high-performance textiles. However, despite its effectiveness in producing high-quality fibers, the method poses environmental challenges due to the generation of toxic effluents, safety risks associated with handling hazardous chemicals, and economic considerations linked to the process's overall feasibility [78].

Chemical extraction advantages

- High Purification: Chemical extraction efficiently removes non-cellulosic and non-fibrous components, resulting in a higher crystallinity index for the fibers. This improves mechanical properties such as tensile strength and stiffness.
- Fiber Uniformity: The process produces fibers with consistent size and chemical composition, essential for technical applications in composites and textiles.
- Versatility: Chemical extraction methods can be tailored to different plant types and fiber structures, making them adaptable to a wide range of raw materials.

Chemical extraction disadvantages

- Environmental Impact: The use of strong chemicals, such as sodium hydroxide (NaOH), strong acids, or oxidizing agents, generates toxic effluents that can contaminate soil and water if not properly managed.
- Health and Safety Risks: The handling of corrosive and hazardous chemicals poses risks to workers, necessitating strict adherence to safety protocols and protective measures.
- Fiber Degradation: Poorly controlled parameters (e.g., concentration, duration, and temperature) can lead to excessive degradation of fibers, compromising their mechanical properties and durability.

1.4.3.3 Biological methods

Biological extraction is a sustainable and environmentally friendly method that relies on microorganisms or enzymes to degrade non-fibrous plant components, such as lignin, hemicellulose, and pectin, which bind the fibers within the plant matrix. Unlike chemical methods, it preserves the structural integrity and mechanical properties of fibers, making it suitable for eco-conscious applications. This approach can be divided into two main categories: retting and enzymatic extraction [73].

1.4.3.4 Extraction family comparison

The extraction of plant fibers represents a fundamental step in preparing them for integration into composite materials. This process aims to isolate the fibers from the plant matrix while preserving their essential mechanical, chemical, and physical properties. Commonly, three main categories of extraction methods are employed: mechanical, chemical, and biological. Each of these methods offers distinct advantages and limitations, making the choice of method highly dependent on specific application requirements. Factors such as the desired fiber properties, environmental sustainability, economic feasibility, and processing time play a crucial role in determining the optimal extraction approach. A detailed comparative analysis of these methods is summarized in **Table 1.3**, highlighting their performance, efficiency, and environmental considerations.

Table 1.3- Comparison of natural fiber extraction families [15, 79].

Criteria	Mechanical family	Chemical family	Biological family
Method	Use of physical force.	Use of chemical products.	Use of micro-organisms or enzymes.
Efficiency	Moderate	Very high	Good
Fiber quality	Fibers obtained are generally short, with residual impurities.	Fibers obtained are generally clean and uniform, although they may be weakened by chemical products.	High-quality fibers with preserved mechanical properties.
Environmental impact	Low.	High.	Moderate.
Cost	Moderate: depends on the equipment used.	High: chemical products increase costs.	Moderate to low: depends on the method used.
Complexity	Low: simple process using mechanical equipment.	Medium to high: requires precise control of chemical reactions.	Moderate: depends on biological conditions and method used.
Duration	Fast: quick process.	Variable: depends on the chemical concentration and the method used.	Slow: usually slower, unless controlled enzymes are used.
Fiber category	Stems, Leaves, Fruits, Seeds.	Stems, Leaves, Seeds.	Stems, Leaves.

1.5 Biocomposites: a sustainable solution

1.5.1 Definition

Bio-composites are an emerging class of advanced materials composed of a polymer matrix and natural reinforcements such as plant-based fibers, animal-derived materials, or bio-minerals. Designed to address the increasing demand for environmentally sustainable alternatives, these materials uniquely integrate the renewable and lightweight characteristics of natural fibers with the mechanical robustness and processing versatility of the matrix. As a result, bio-composites are highly adaptable and find use in a wide range of industrial applications [80].

In a typical bio-composite, the polymer matrix serves as the binding phase, ensuring structural cohesion and protecting the reinforcements from environmental degradation. Additionally, it facilitates the transfer of mechanical stresses throughout the material. These matrices can be sourced from renewable biopolymers such as polylactic acid (PLA) or polyhydroxyalkanoates (PHA) or may involve partially biodegradable synthetic polymers. The reinforcement phase, often comprising lignocellulosic fibers such as hemp, sisal, jute, or yucca, contributes significantly to the material's rigidity, tensile strength, and thermal stability, enhancing its overall performance. Bio-composites stand out from traditional composites not only due to the inclusion of natural, bio-based components but also through their significantly lower environmental impact. These materials help to mitigate the challenges associated with climate change by actively reducing carbon emissions, minimizing the reliance on non-renewable resources, and fostering the principles of the circular economy. Unlike conventional composites, bio-composites are often designed with end-of-life strategies, such as biodegradability or recyclability, in mind. This focus on sustainability ensures that bio-composites have a reduced ecological footprint throughout their lifecycle, from production to disposal, making them a more environmentally responsible choice in material engineering [81].

1.5.2 Characteristics

Bio-composites exhibit a unique combination of physical, mechanical, thermal and environmental properties, which result from the interaction between the polymer matrix and the natural reinforcement. These characteristics are predominantly influenced by the type of fiber used, the characteristics of the polymer matrix, and the manufacturing conditions.

1.5.2.1 Lightness and Density

Bio-composites offer a significant advantage in terms of density, being lighter than traditional composites reinforced with synthetic fibers. The density of natural fibers typically ranges from 1.2 to 1.5 g/cm³, while synthetic fibers tend to have a density greater than 2.5 g/cm³. This lower density imparts bio-composites with a remarkable advantage in weight-critical applications, particularly in the automotive, aerospace, and transportation sectors, where reducing weight is essential for enhancing fuel efficiency, performance, and reducing environmental impact. Therefore, the use of bio-composites in these applications aligns with the growing demand for lightweight, sustainable materials [82].

1.5.2.2 Mechanical proprieties

The mechanical properties of bio-composites are competitive with those of synthetic composites, though they can vary significantly depending on the types of fibers and matrices used. These properties play a critical role in determining the suitability of bio-composites for various applications.

- **Tensile strength:** Natural fibers can achieve tensile strengths of up to 800 MPa, enabling bio-composites to support high loads.
- **Modulus of elasticity:** These composites offer adequate stiffness, although often less than glass fiber composites, making them a preferred choice for applications requiring flexibility and resilience.

These mechanical characteristics ensure that bio-composites can serve as reliable alternatives to synthetic composites, providing both performance and sustainability [70].

1.5.2.3 Thermal stability

Bio-composites typically offer good thermal properties, which can be enhanced depending on the specific combination of natural fibers and the polymer matrix. While natural fibers themselves generally have low thermal conductivity, the overall thermal insulation performance of bio-composites can be optimized through careful selection of materials and processing techniques. However, the thermal conductivity of bio-composites tends to be lower than that of conventional synthetic composites, making them suitable for applications where thermal insulation is desired. In addition, it is important to note that their thermal performance may not always match that of high-performance synthetic composites, particularly in applications requiring very low thermal conductivity or extreme temperature resistance [83].

1.5.2.4 Production price

The cost of producing bio-composites presents an essential role in their competitiveness with traditional composite materials. The cost of natural fibers is influenced by several factors, including the availability of resources, the type of fiber, and the harvesting or extraction processes used. Several bio-fibers are often less expensive when compared to synthetic fibers, primarily due to the lower costs of raw materials and simpler harvesting techniques. In contrast, bio-based polymer matrices, such as those derived from renewable resources (polylactic acid or bio-polyethylene), tend to be more expensive than conventional synthetic resins like epoxy or polyester. This is due to the higher costs associated with the production and processing of bio-sourced monomers, as well as the relatively small-scale production of these materials [84].

Despite these cost considerations, the overall economic viability of bio-composites can be enhanced by automating production processes, optimizing manufacturing techniques, and improving material sourcing efficiency. Automation in fiber extraction, composite molding, and curing processes can significantly reduce labor costs and increase production consistency, leading to economies of scale. Additionally, further advancements in bio-based resin production could lower costs and increase the competitiveness of bio-composites in the market [85].

1.5.2.5 Environmental compatibility

Bio-composites are increasingly valued for their sustainability, durability, and minimal environmental impact. One of the primary benefits of using natural fibers, derived from renewable resources such as plants, is their ability to reduce the carbon footprint of composite materials. This is in contrast to synthetic fibers, which often require energy-intensive processes and contribute to greater environmental degradation. The use of natural fibers not only lowers the environmental impact during production but also helps mitigate climate change by capturing carbon dioxide during the growth of the plants. Furthermore, bio-composites offer significant advantages in terms of waste management at the end of their life cycle. Many natural fibers are biodegradable, meaning that once the bio-composite material reaches the end of its useful life, it can decompose naturally without contributing to long-term waste accumulation. This biodegradability reduces the reliance on landfills or incineration, further promoting a circular economy. As industries move toward more eco-conscious production practices, bio-composites stand out as a key material in reducing the environmental burden associated with traditional composites, offering both environmental and functional benefits [86, 87].

1.5.3 Fiber reinforcement format

The format of natural fibers embedded in a polymer matrix is a key factor that influences the overall performance of bio-composites. Fibers can be incorporated in various forms as illustrated in **Figure 1.6**, including short fibers, long fibers, and powdered fibers, each contributing differently to the mechanical, thermal, and structural characteristics of the composites. The impact of these formats is determined by factors such as fiber length, alignment, and distribution within the matrix. Moreover, the choice of fiber format is guided by the specific requirements of the intended application, the manufacturing process, and industrial constraints, ensuring the composites meet both functional and practical demands [88].

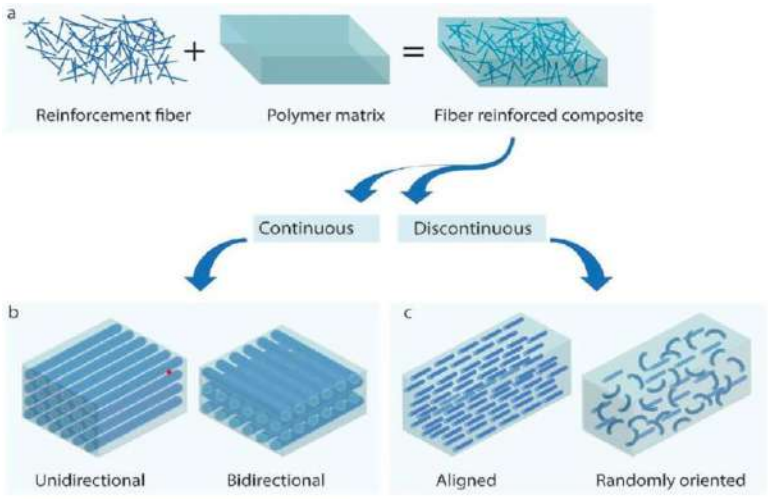

Figure 1.6- Structure of a fiber-reinforced biocomposite: (a) Fundamental components; (b) Continuous fiber-reinforced biocomposites (unidirectional and bidirectional); (c) Discontinuous fiber-reinforced biocomposites (aligned and randomly oriented) [89].

1.5.3.1 Short fibers

Short fibers are fiber segments of relatively short length, often between 0.1 mm and 10 mm. These fibers are incorporated into the matrix in a random or slightly oriented manner, resulting in bio-composites with isotropic properties. Short fibers are extensively utilized due to their compatibility with high-speed manufacturing techniques such as injection molding, extrusion, and compression molding. Bio-composites reinforced with short fibers exhibit moderate enhancements in mechanical performance, including increased stiffness and intermediate tensile strength. However, the limited length of the fibers restricts load transfer efficiency between the matrix and the fibers, leading to lower overall strength compared to composites reinforced with long fibers [90].

1.5.3.2 Long fibers

Long fibers, exceeding 10 mm in length and potentially reaching several tens of centimeters or even continuous lengths, enable superior load transfer, especially when aligned in a specific direction. This alignment significantly enhances the mechanical properties of bio-composites, notably tensile strength, bending resistance, and impact durability. Long fibers exhibit enhanced adhesion with the matrix, reducing localized stress concentrations. However, their integration requires specialized processes such as vacuum molding or resin transfer molding (RTM) to ensure proper orientation and minimize damage during manufacturing [91].

1.5.3.3 Powder fibers

Powder fibers, are obtained by grinding natural fibers to a size of less than 500 µm. This format, often referred to as 'lignocellulosic fillers', while they do not contribute significantly to mechanical strength, they enhance other properties, such as dimensional stability, shrinkage resistance, and weight reduction. Powder fibers are easily dispersed within the polymer matrix and are well-suited for high-speed manufacturing processes like extrusion and injection molding. This format is particularly advantageous for applications prioritizing cost-efficiency and environmental sustainability [91, 88].

1.5.4 Elaboration methods

The production of bio-composites involves the strategic integration of natural fibers into a polymer matrix, whether it is thermoplastic, thermosetting, or bio-based polymers. The selection of both materials and manufacturing processes is critical to achieving the desired performance attributes in the final bio-composite, including mechanical strength, thermal stability, and environmental sustainability. Understanding the interaction between the fibers and the matrix, as well as optimizing processing conditions, is essential to ensuring the bio-composite meets the performance specifications for its intended application. Various methods are employed in the manufacturing of bio-composites, each offering distinct advantages depending on factors such as material properties, production scale, and application requirements [92]. These manufacturing techniques include:

1.5.4.1 Injection molding technique

Injection molding is a widely used technique, particularly for producing complex parts from bio-composites. In this process, natural fibers are blended with a molten resin, and the mixture is then injected into a mold under high pressure, as illustrated in **Figure 1.7**. This method allows for the production of parts

with intricate geometries and high precision, making it ideal for a variety of industrial applications [93]. The process involves several key steps:

1- Preparation of the composite material: Natural fibers are mixed with a molten thermoplastic resin. The resin is typically heated to a temperature that allows it to flow easily, ensuring uniform dispersion of the fibers within the matrix.

2- Injection into the mold: The fiber-resin mixture is injected into a closed mold cavity under high pressure. The pressure ensures that the composite material fills every part of the mold, even in areas with complex shapes or thin walls.

3- Cooling and solidification: After injection, the mold is cooled, causing the resin to harden and the composite material to take the shape of the mold. Cooling rates and temperature control are critical to achieving desired properties and preventing defects.

4- Demolding: Once the material has cooled and solidified, the part is removed from the mold. The final product can then undergo additional processes such as trimming, polishing, or painting to meet specific requirements.

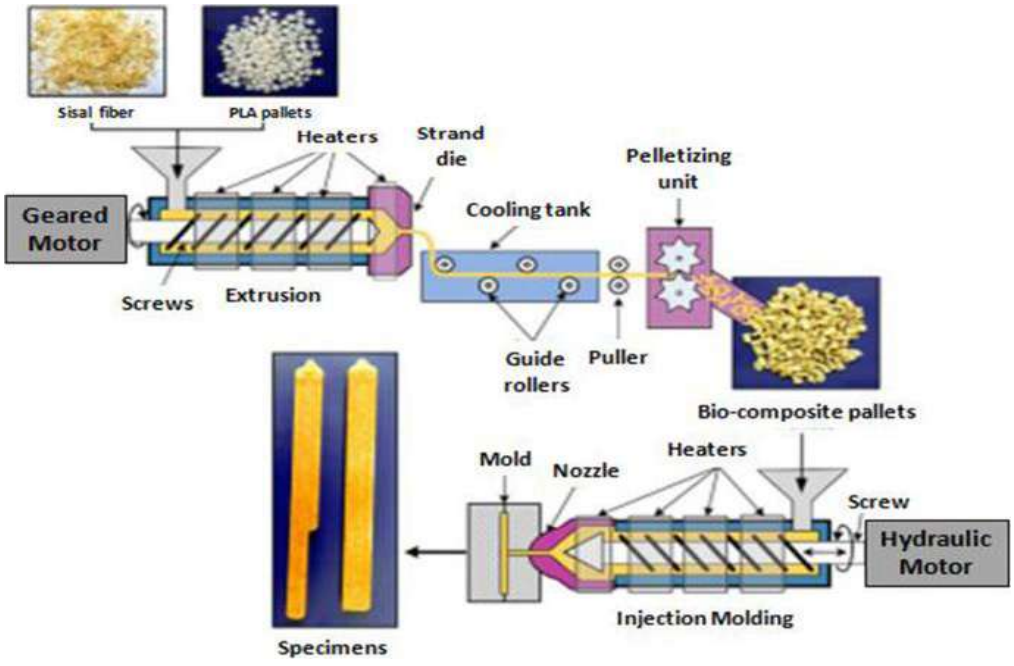

Figure 1.7 - Bio-composite injection molding technique [94].

Advantages of this technique: High output, ability to produce complex parts with precise quality control, fast process.

Disadvantages of this technique: Requires specialized equipment, uniform fibers distribution can be a challenge, high temperatures may affect fiber properties.

1.5.4.2 Casting molding technique

Cast molding is a simple and cost-effective manufacturing process used to produce bio-composites by mixing natural fibers with a resin and then pouring the mixture into a mold to form the desired shape. Unlike injection molding, which requires high pressure, casting relies on gravity and the natural curing properties of the resin, as indicated in **Figure 1.8**. This makes cast molding a suitable method for producing larger or less intricate bio-composite parts where high precision is not required [95]. Steps in the cast molding process are:

1- Preparation of the Composite Material: Natural fibers are first mixed with a resin, which can be either thermoplastic or thermosetting depending on the application. The resin is usually mixed with a hardener or catalyst if needed, especially in the case of thermosetting resins, which cure upon chemical reaction.

2- Pouring into the Mold: Once the fiber-resin mixture is ready, it is poured into a pre-prepared mold. The mold can be made of various materials, including metal, silicone, or plastic, depending on the requirements of the final product. The mixture is poured into the mold at room temperature.

3- Curing: After pouring, the resin cures and hardens either at room temperature or under controlled conditions, depending on the type of resin used. Thermosetting resins typically require longer curing times, while thermoplastics can solidify more quickly as they cool down.

4- Demolding: Once the resin has completely cured and solidified, the part is removed from the mold. The final product may undergo finishing processes such as trimming, polishing, or painting to achieve the desired appearance and functionality.

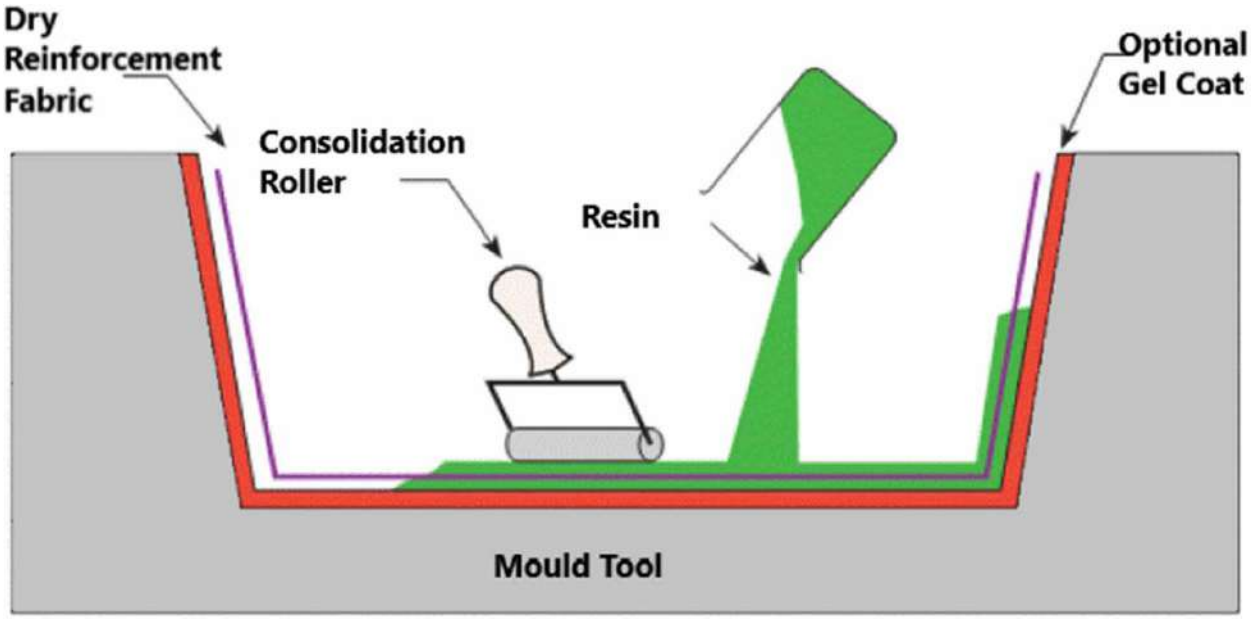

Figure 1.8- Casting molding technique elaboration [96].

Advantages of this technique: Relatively low manufacturing cost, simple process, suitable for larger or less complex geometries, possibility of using several types of matrices.

Disadvantages of this technique: Limited in terms of shape accuracy and mechanical strength, slower production speed than injection or compression molding, requires careful control of resin viscosity to avoid defects.

1.5.4.3 Additive Manufacturing technique

3D printing of bio-composites represents a breakthrough in sustainable manufacturing, allowing for the production of intricate parts and structures with minimal waste and enhanced customization. By combining natural fibers with bio-based polymers, 3D printing provides a flexible approach to designing materials that are both functional and eco-friendly. The use of natural fibers, such as hemp, flax, or yucca, within a polymer matrix (typically PLA) is particularly beneficial for applications where reduced environmental impact and resource efficiency are important [97]. Key steps in 3D printing of bio-composite materials are:

1- Filament Production:

- The first step in 3D printing with bio-composites is to create a high-quality printable filament that can be used in a 3D printer. This involves mixing natural fibers with a polymer matrix, such as PLA or another biodegradable polymer, in a controlled manner.
- The fibers must be finely ground and carefully blended with the polymer to ensure uniform distribution and consistency. The filament must then be extruded and cooled into solid form, creating a spool of filament ready for printing. The quality of this filament is critical for successful 3D printing, as illustrated in **Figure 1.9**.
- The formulation of the filament is crucial. The viscosity, flexibility, and extrusion temperature need to be carefully adjusted to ensure that the filament flows smoothly through the 3D printer's extruder without clogging, while also ensuring the filament retains the desired mechanical properties.

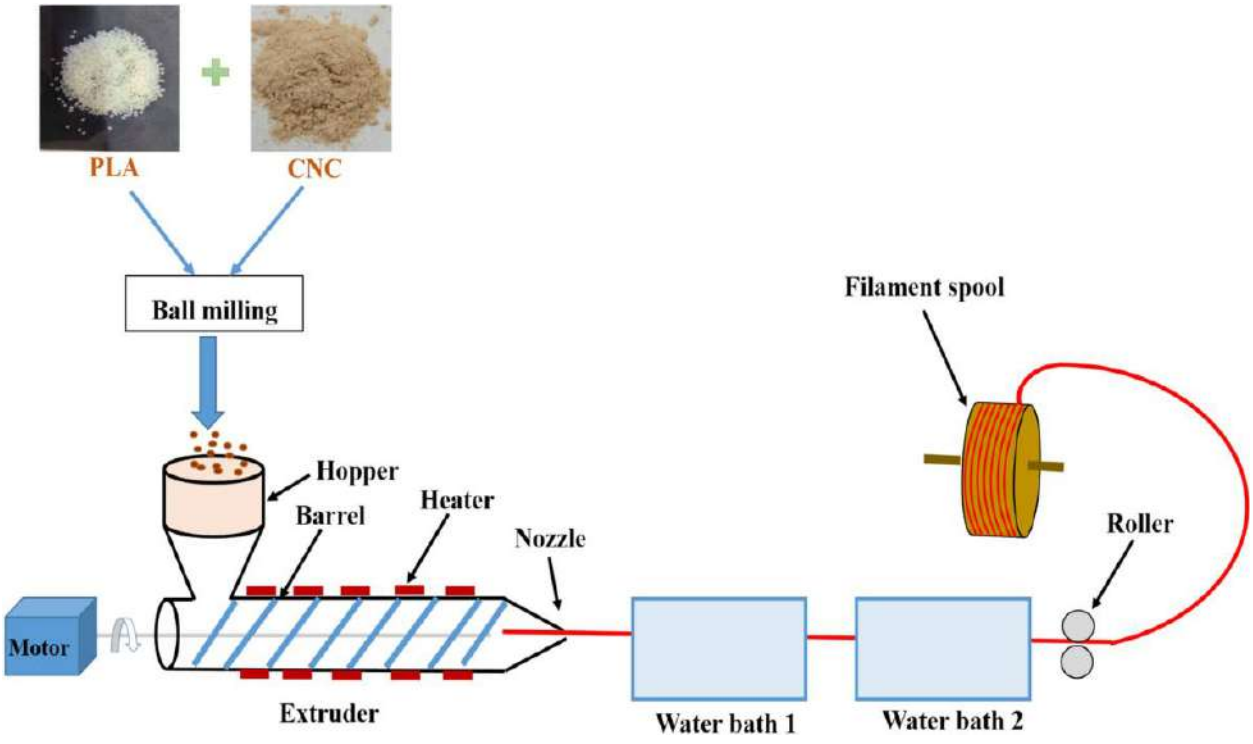

Figure 1.9 - Production of printable bio-composite filament [98]. (CNC: Cellulose Nanocrystals)

2- Filament Characteristics:

- Viscosity and Flexibility: The filament needs to maintain an optimal balance of viscosity and flexibility, allowing it to pass through the extruder and nozzle without difficulty, while also being

strong enough to maintain the desired properties once printed. Too much viscosity can result in clogging, while too little can result in weak or inconsistent prints.

- Fiber concentration and particle size: The fiber concentration and the size of the natural fiber particles significantly impact the mechanical properties of the final 3D printed object. The fiber concentration affects the reinforcement of the matrix, which in turn determines the strength, flexibility, and impact resistance of the printed part. The particle size of the fibers needs to be fine enough to avoid clogging the printer but large enough to provide reinforcement within the polymer matrix.
- Uniformity: A key factor for the success of 3D printing bio-composites is ensuring uniform dispersion of fibers within the filament. Inconsistent fiber distribution can lead to weak points in the printed parts, reducing their mechanical integrity.

3- 3D Printing Process:

- After preparing the bio-composite filament, the printing process begins. The 3D printer heats the filament to a specific extrusion temperature, melting it so that it can be deposited in thin layers on a build platform. Each layer fuses with the layer beneath it, gradually building up the structure, as demonstrated in **Figure 1.10.**
- The ability to print in complex geometries makes 3D printing an ideal technique for creating customized components and parts. The bio-composites used in 3D printing can be adjusted to meet specific strength, flexibility, and durability requirements for different applications.

4- Post-Processing:

- After printing, post-processing steps such as curing, sanding, or coating may be required to improve the appearance and mechanical properties of the bio-composite parts. These processes help to refine the final product and ensure its quality and longevity.
- The printed part may need to undergo additional treatments, such as heat curing or surface coatings, to further enhance its performance.

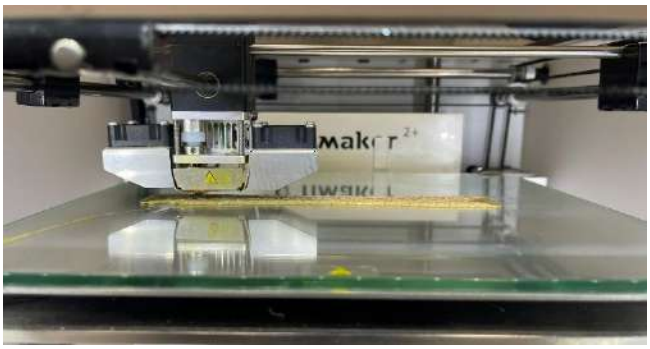

Figure 1. 10- Biocomposite printing process.

Technique advantages: Fast, personalized manufacturing, reduced waste, ability to manufacture complex geometries.

Technique disadvantages: Need to produce a filament suitable for 3D printing, production speed limitations, mechanical properties often inferior to those of traditional materials for structural applications.

1.5.4.4 Other techniques

Several other manufacturing methods are employed to produce bio-composites, including pultrusion, which is a continuous process where fibers are pulled through a resin bath and then passed through a heated mold to allow the resin to polymerize. Another method is laminating, where successive layers of natural fibers and resin are stacked to create multi-layered composites. Additionally, the compression-injection molding (CIM) technique is used, where molten resin is injected under high pressure into a matrix containing natural fibers [99].

1.5.5 Applications

As sustainable and high-performance materials, bio-composites are increasingly contributing to various industrial sectors, owing to their exceptional mechanical, thermal, and environmental properties. Comprising polymer matrices reinforced with biodegradable fibers, these materials present a promising alternative to traditional composites, fulfilling the rising demand for enhanced durability and performance [55]. Bio-composites are finding applications across a range of industries, including:

1.5.5.1 Automotive industry

Bio-composites are widely utilized in the automotive industry for the production of lightweight and sustainable components, as illustrated in **Figure 1.11**. These materials are applied in both interior and

exterior parts, including door panels, dashboards, seatbacks, headliners, bumpers, and underbody shields. The integration of natural fibers into bio-based polymer matrices effectively reduces the overall weight of the vehicle, leading to enhanced fuel efficiency and a decrease in greenhouse gas emissions. Moreover, the acoustic damping properties of bio-composites contribute to increased interior comfort by minimizing noise and vibration [100].

Figure 1.11- Application of biocomposite in automobile application [100].

1.5.5.2 Construction sector

The construction industry has increasingly adopted bio-composites for a wide range of structural and non-structural applications. These materials are commonly used in decking, cladding, insulation panels, partition walls, and furniture due to their favorable attributes, including a high strength-to-weight ratio, excellent thermal insulation properties, resistance to UV degradation, and durability against weathering. Bio-composites, composed of bio-fibers combined with thermoset or thermoplastic matrices, present sustainable alternatives to conventional materials such as concrete and wood. Furthermore, their biodegradability aligns with the growing emphasis on eco-friendly and green building practices [101].

1.5.5.3 Aerospace industry

In the aerospace sector, bio-composites are increasingly utilized in secondary structures, such as interior panels, storage compartments, and flooring systems, where weight reduction is crucial for enhancing fuel efficiency and optimizing payload capacity. The combination of natural fibers with high-performance bio-based resins imparts excellent specific strength and stiffness, while simultaneously

contributing to a reduction in the carbon footprint of aerospace manufacturing. Nevertheless, the stringent regulatory and safety standards governing this industry necessitate comprehensive testing to ensure that these materials satisfy the demanding mechanical and thermal performance criteria required for their application in aerospace systems [102].

1.5.5.4 Biomedical industry

Bio-composites are emerging as innovative materials in medical applications, owing to their biocompatibility, adaptability, and potential for controlled degradation. They are being explored in a variety of applications, including orthopedic implants, prosthetic devices, dental implants, and scaffolds for tissue engineering. The incorporation of bio-fibers into bio-based matrices imparts the necessary mechanical strength while also promoting favorable biological responses. For example, scaffolds made from bio-composites can support cell adhesion and proliferation, thus aiding in tissue regeneration. However, despite these benefits, challenges such as controlling degradation rates and ensuring sterility must be addressed to enable their widespread use in clinical settings [103].

1.5.5.5 General industry

Bio-composites are increasingly being adopted in the packaging industry as sustainable alternatives to conventional petroleum-based plastics, primarily due to their biodegradability and composability. These materials are particularly utilized in food packaging and kitchen utensils applications, including trays, containers, and films, where the use of starch-based or polylactic acid (PLA) matrices, reinforced with natural fibers ensures the necessary mechanical strength, flexibility, and shelf-life. The incorporation of natural fibers not only enhances the mechanical properties of the bio-composites but also contributes to reducing their environmental impact by reducing reliance on fossil-derived plastics. Additionally, bio-composites offer promising potential in addressing the growing concern of plastic pollution, as they can be naturally degraded through microbial activity, thereby minimizing waste accumulation [104].

1.5.6 Bio-composite material advantages

Bio-composites represent a significant advancement in the development of sustainable materials, offering a balance between high mechanical performance and a minimal environmental impact, regrouped in **Figure 1.13**. Composed of natural fibers and polymer matrices, bio-composites are sourced from renewable resources, providing a clear advantage in terms of sustainability. A key benefit of bio-composites lies in their reduced environmental footprint. Natural fibers, are derived from plants that

actively sequester carbon dioxide (CO_2) during their growth, which effectively mitigates the overall carbon footprint of these materials. This characteristic positions bio-composites as an eco-friendlier alternative to traditional composites, which are often reliant on fossil-based resources [87].

Another significant advantage of bio-composites lies in their mechanical properties. The inclusion of natural fibers imparts high strength to these materials while maintaining a lightweight structure. This makes bio-composites especially suitable for industries where weight reduction is a critical factor, such as aerospace and automotive. For instance, bio-composites are increasingly used in the production of structural components in automobiles, contributing to reductions in fuel consumption and CO_2 emissions. Additionally, the combination of high specific strength and excellent vibration-damping properties makes bio-composites ideal for applications in thermal and acoustic insulation, particularly within the construction sector, where their performance can enhance energy efficiency and occupant comfort [105].

From an economic perspective, bio-composites present significant viability. Natural fibers, often sourced from agricultural by-products, are typically more cost-effective than their synthetic counterparts, resulting in reduced material costs. This reduction makes bio-composites increasingly competitive with traditional composite materials. Furthermore, the production of bio-composites contributes to the rural economy by generating employment opportunities within the agricultural sector and the fiber processing industry. By leveraging the accessibility of raw materials and enhancing performance, bio-composites emerge not only as innovative materials but also as economically viable solutions for a broad spectrum of industrial applications, offering both environmental and financial benefits [84].

The versatility of bio-composites is a key driver behind their increasing adoption across various industries. These materials can be customized for a wide range of applications, including automotive components, construction panels, eco-friendly packaging, and technical textiles. By combining natural fibers with bio-based polymer matrices or synthetic resins, the properties of bio-composites can be precisely tailored to meet the specific performance requirements of each application. This adaptability offers significant potential in diverse sectors such as sustainable mobility, renewable energy, and construction. Additionally, hybrid bio-composites, which integrate natural fibers with recycled plastics, represent a promising avenue for advancing the circular economy by reducing waste and promoting resource efficiency [106].

Finally, bio-composites are often produced using more environmentally responsible processes compared to traditional composite materials. Fiber extraction methods, such as biological retting or

enzymatic extraction, significantly reduce the need for aggressive chemicals, thereby minimizing environmental impact. These sustainable extraction techniques preserve the mechanical and chemical properties of the fibers while ensuring minimal effects on worker health and environmental quality. Furthermore, bio-composites contribute to the transition of industries towards more circular and sustainable production models, where materials are recycled or reused at the end of their life cycle, effectively closing the loop in the value chain and promoting long-term environmental benefits [107].

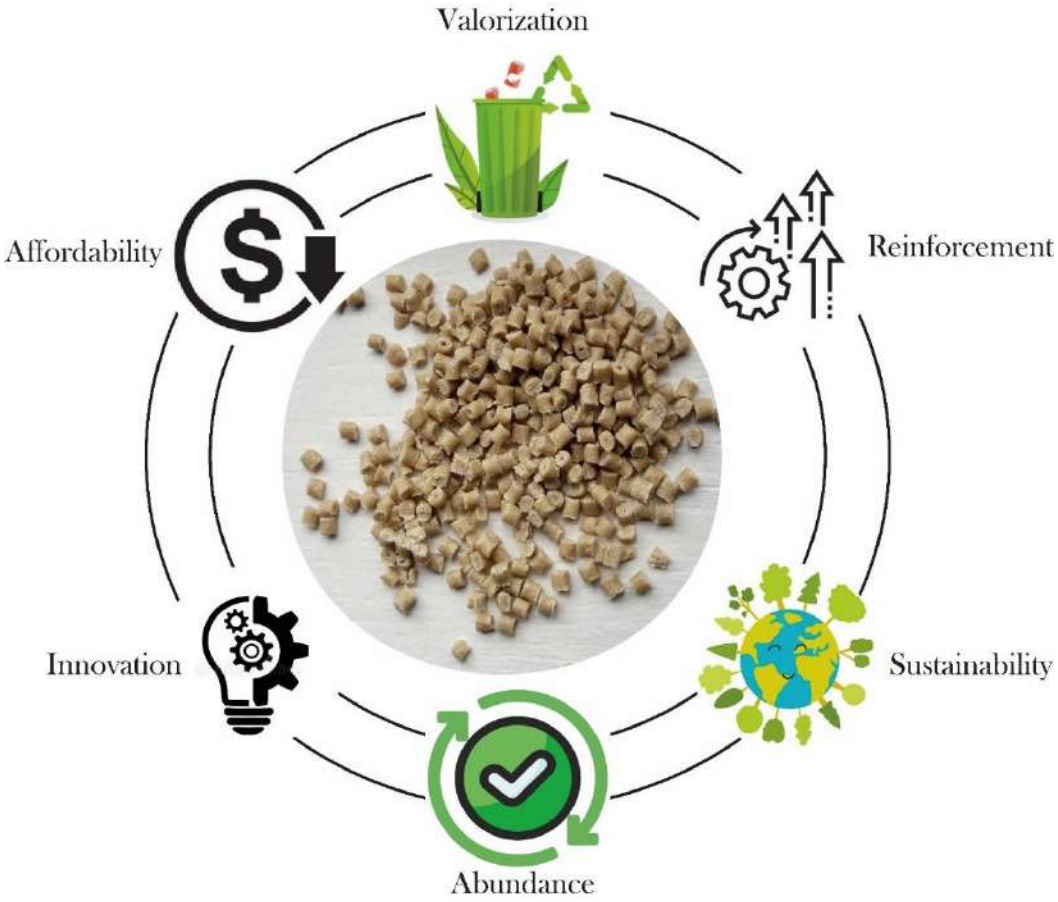

Figure 1.12- Advantages of applications for bio-composite materials.

1.6 Influence of extraction methods on the fiber and bio-composite properties

1.6.1 Impact of extraction processes on fibers

The extraction processes applied to plant fibers significantly impact their properties, which in turn affect their performance in bio-composite applications. These properties include chemical purity, crystallinity, mechanical strength, thermal stability, moisture absorption, surface structure, and interfacial

adhesion [51]. The manner in which extraction methods influence these key attributes is examined in detail:

1.6.1.1 Chemical Purity

Chemical extraction methods, are designed to remove non-cellulosic components such as lignin, hemicelluloses, and pectin, which bind the fibers together within the plant tissue. By increasing the cellulose content, chemical extraction enhances the chemical purity of the fibers. This purification process not only improves the adhesion between the fiber and polymer matrix in bio-composites but also strengthens the fibers, particularly in terms of tensile strength and rigidity. The removal of impurities increases the accessibility of hydroxyl groups on the fiber surface, facilitating better interactions with the matrix and improving overall bio-composite performance [15].

1.6.1.2 Crystallinity

Crystallinity refers to the ordered structure of the cellulose within the fibers, which directly influences their mechanical properties. The extraction process can affect the crystallinity of fibers; an increase in crystallinity leads to greater rigidity and mechanical strength, particularly enhancing the tensile strength of the fibers. However, poorly controlled extraction processes can result in fiber degradation, reducing crystallinity and diminishing the fiber's mechanical performance. Optimizing the extraction conditions is crucial to achieving the desired balance between crystallinity and fiber integrity [51].

1.6.1.3 Mechanical proprieties

The mechanical properties of fibers, including tensile strength, stiffness, flexibility, and toughness, are heavily influenced by the extraction process. Controlled chemical extractions can remove impurities and increase crystallinity, thereby enhancing the mechanical strength of the fibers. However, excessive chemical treatment can lead to fiber degradation, negatively affecting their strength and durability. In contrast, mechanical extraction processes, which preserve the structural integrity of the fibers, generally lead to more consistent mechanical properties. These processes, however, may be less effective at removing non-cellulosic impurities compared to chemical treatments. A balance between fiber enhancement and preservation of structural integrity is essential for optimizing the mechanical properties of bio-composites [75].

1.6.1.4 Thermal properties

The thermal stability of fibers, including their degradation temperature, is influenced by the extraction process. Chemical extraction methods, which remove components such as lignin, can increase

the thermal stability of fibers, making them more suitable for applications that require resistance to high temperatures. However, excessive chemical extraction can lead to premature thermal degradation of the fibers, reducing their performance in high-temperature applications. Mechanical and biological extraction methods tend to have a more moderate impact on the thermal stability of fibers, but they are more effective at preserving the fiber structure under elevated temperatures [108].

1.6.1.5 Morphology and surface structure

The surface morphology of the fibers plays a critical role in their interaction with the composite matrix. Mechanical methods, such as defibration or decortication, can create a rough, irregular surface that enhances adhesion to the polymer matrix, improving the bonding between the fiber and matrix in the final bio-composite. In contrast, chemical extraction processes, which remove non-cellulosic components, tend to smooth the fiber surface, potentially reducing adhesion with the matrix. Biological methods, which are gentler on the fibers, generally preserve their natural surface structure, but they may not achieve the same level of surface modification as mechanical or chemical treatments. The surface structure of the fibers is crucial in determining the efficiency of the fiber-matrix interface, impacting the overall performance of the bio-composite [109].

1.6.1.6 Interfacial adhesion and compatibility

The adhesion between the fiber and the polymer matrix is primarily determined by the surface structure of the fibers. Chemically extracted fibers, although exhibiting smoother and more homogeneous surfaces, may demonstrate poor adhesion to certain polymer matrices due to reduced surface roughness. In contrast, mechanically extracted fibers, with their rougher surfaces, tend to have better adhesion to the matrix, which results in stronger bio-composite performance. Biological extraction methods typically preserve the natural structure of the fibers, maintaining good adhesion without compromising the mechanical properties of the fibers. Ensuring good interfacial adhesion is essential for achieving high-performance bio-composites, and the choice of extraction method plays a significant role in this regard [109, 110].

1.6.2 Impact on bio-composite performance

The natural fiber extraction process directly influences the performance of bio-composites, as it modifies the fibers' structure, surface chemistry, and morphology [111]. The following properties of bio-composites are particularly impacted by the extraction methods used:

1.6.2.1 Mechanical properties

The mechanical properties of bio-composites, such as tensile strength, flexibility, stiffness, and compressive strength, are highly dependent on the characteristics of the extracted fibers. These properties are influenced by the extraction methods employed, which can alter the fiber's chemical composition, crystallinity, and surface morphology [112].

1.6.2.2 Thermal properties

The thermal properties of bio-composites, such as thermal resistance and thermal conductivity, can be influenced by the extraction process [113]. Particularly with regard to the crystallinity of the fibers:

- Degradation temperature: Increasing the crystallinity of fibers can improve the degradation temperature of bio-composites. This enhancement makes the bio-composites more resistant to high temperatures, a valuable trait for applications requiring thermal stability.
- Thermal conductivity: Fibers that retain a structure closer to that of the original natural fiber may offer lower thermal conductivity, making bio-composites more suitable for thermal insulation applications. The impact of extraction methods on thermal conductivity depends on how the fiber structure is modified during processing.

1.6.2.3 Dimensional stability

The dimensional stability of bio-composites, which is critical to their long-term performance, can also be affected by extraction methods. Mechanically or biologically extracted fibers can have improved dimensional stability, as they are less sensitive to variations in moisture or temperature than chemically extracted fibers, which can be stiffer and less sensitive to moisture [114].

1.7 Conclusion:

In this chapter, we have explored natural fibers in detail, with an emphasis on vegetal fibers, their advantages, limitations, and their integral role in the development of bio-composites. Renowned for their lightweight, renewable nature, and cost-effectiveness, these fibers pose certain technical challenges, including their susceptibility to moisture and limited compatibility with polymer matrices.

The discussion has placed particular focus on the extraction methods of natural fibers, which play a key role in preserving and optimizing their properties. These methods significantly influence the structural, morphological, and mechanical characteristics of the fibers, thereby impacting the overall performance of

the resulting bio-composites. The choice of an appropriate extraction method was underscored as essential to meet the specific demands of various applications.

In addition, the chapter explored the various techniques for manufacturing bio-composites and emphasized the role of surface treatments for natural fibers as indispensable strategies to enhance interfacial interactions between fibers and polymer matrices. These processes enable the optimization of mechanical and physicochemical properties, making bio-composites competitive across a wide array of applications while addressing the challenges of sustainability.

This chapter provides the theoretical groundwork necessary to understand the influence of extraction methods on the properties of natural fibers and bio-composites. These foundational concepts will inform the experimental investigations detailed in the following chapters, which will assess the performance of fibers and bio-composites in relation to the processes explored.

Chapiter 2: Material characteristics and experimental strategies

2.1 Introduction:

This chapter outlines the experimental methodologies and protocols implemented to investigate the influence of fiber extraction methods, their incorporation into bio-composites, and the impact of chemical treatments on the resulting material properties. The objective of this methodological approach is to elucidate how different processing stages, from fiber selection to chemical modification, affect the structural, mechanical, and physicochemical characteristics of natural fibers and, consequently, the performance of bio-composites.

The selection of natural fibers, particularly yucca fibers, is first justified based on their intrinsic properties, which make them suitable reinforcements for polymer matrices. Their high mechanical strength and durability render them promising candidates for bio-composite applications. The extraction methodologies, encompassing biological, mechanical, and chemical techniques, are then detailed, with an emphasis on how each method influences fiber quality and properties. These extraction processes not only alter the physical structure of the fibers but also modify their chemical composition, particularly in terms of lignin, cellulose, and hemicellulose content, which directly affects their compatibility with polymer matrices.

Following extraction, various characterization techniques are employed to analyze the structural and compositional modifications induced by the different methods. Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and scanning electron microscopy (SEM) are utilized to assess changes in the chemical functionalities, crystalline structure, and morphological features of the fibers.

The subsequent phase involves the fabrication of bio-composites using different polymer matrices, both with and without fiber reinforcements. The prepared samples undergo a series of mechanical tests to evaluate their strength, durability, and overall performance. Comparative analyses are conducted to assess the influence of fiber type and extraction method on the mechanical behavior of the resulting bio-composites.

Finally, the chapter details the chemical treatments applied to the extracted fibers, including alkaline and acidic modifications. These treatments are designed to enhance fiber properties, improve interfacial adhesion with the polymer matrix, and optimize the mechanical performance of the bio-composites. The effects of these chemical modifications on the final properties of the materials are

systematically analyzed to identify optimal processing conditions for high-performance bio-composite development.

2.2 Natural vegetal fibers

2.2.1 Choice criteria

The utilization of natural fibers in the fabrication of bio-composites is governed by several fundamental criteria that directly impact their mechanical performance, durability, and compatibility with the polymer matrix. The plant sources for fiber extraction must satisfy specific requirements to ensure the structural integrity and long-term viability of the resulting composites [115]. The principal selection criteria are as follows:

2.2.1.1 Fiber mechanical properties

The mechanical performance of natural fibers is a critical determinant of their suitability as reinforcement in bio-composites, where they serve to enhance structural integrity. Key parameters such as tensile strength, stiffness, and flexibility are essential to ensure the mechanical stability and longevity of bio-composites under diverse operational conditions. Furthermore, fatigue resistance is a crucial property, as it dictates the fiber's ability to withstand cyclic loading without significant degradation, thereby extending the service life of the composite material [116].

2.2.1.2 Fiber structural characteristics

The structural attributes of natural fibers exert a direct influence on their compatibility with polymer matrices. Fibers of appropriate length and diameter facilitate effective load distribution, thereby optimizing the reinforcement potential within the composite. Additionally, a homogeneous microstructure characterized by minimal impurity content and a high cellulose fraction is imperative for ensuring superior mechanical performance. Conversely, an excessive lignin content can adversely affect fiber flexibility and processing efficiency, potentially compromising composite integrity [117].

2.2.1.3 Accessibility and availability

The large-scale application of natural fibers in bio-composite manufacturing necessitates a reliable and sustainable supply. The selected plant species should be widely accessible and available in sufficient quantities to support industrial-scale production. Additionally, economic considerations play a

pivotal role, as the cost of fiber procurement must remain competitive with alternative reinforcement materials to ensure the financial feasibility of bio-composite production [115].

2.2.1.4 Environmental considerations

A primary advantage of natural fibers is their renewable nature. Therefore, the selection process must prioritize fibers derived from sustainably cultivated plant sources with minimal environmental impact. The biodegradability of fibers post-service life is another critical factor, as it contributes to the reduction of ecological footprints and aligns with sustainable material development initiatives. The capacity for fiber recycling or composting at the end of the composite's lifecycle is essential for promoting environmentally responsible solutions [117].

2.2.1.5 Nutritional considerations

Certain natural fibers possess inherent nutritional value, such as dietary fibers beneficial to human digestion. When fibers are intended for pharmaceutical or food-related applications, stringent purity and non-toxicity requirements must be met. In such cases, their use in bio-composites may be inappropriate, as it could compromise their suitability for human consumption due to potential contamination risks. Consequently, the allocation of these fibers should be carefully evaluated to prevent conflicts between industrial and alimentary applications.

2.2.1.6 Efficiency of fiber extraction

The fiber extraction process must be both efficient and cost-effective. Various extraction techniques, including mechanical, chemical, or biological, should be employed to obtain high-quality fibers while preserving their intrinsic mechanical and chemical properties. Post-extraction characteristics such as tensile strength, surface texture, and impurity levels must be optimized to enhance fiber performance in bio-composite applications [116].

2.2.1.7 Economic viability

The economic feasibility of bio-composite production is strongly influenced by the cost of fiber extraction and processing. The cultivation, harvesting, and treatment of natural fibers must remain economically viable to ensure competitiveness with conventional composite materials. Additionally, the

integration of circular economy principles, such as fiber recycling and end-of-life reutilization can contribute to cost reduction and further enhance the sustainability of bio-composites [116].

2.3 Yucca plant

Yucca (**Figure 2.1**) is a genus of perennial plants belonging to the Asparagaceae family, subfamily Agavoideae, widely distributed across arid and subtropical regions of North, Central, and South America. The plant is characterized by its long, rigid, linear leaves (up to 120 cm in length and approximately 5 cm in width) arranged in a basal rosette. Additionally, yucca produces terminal inflorescences comprising clusters of white flowers. Certain species, such as *Yucca schidigera* and *Yucca filamentosa*, are particularly valued for their fibers, which exhibit superior mechanical properties, including high tensile strength, flexibility, and resilience. These attributes make yucca fibers highly suitable for bio-composite applications.

Given their desirable mechanical performance and renewable nature, yucca fibers are increasingly investigated as a sustainable alternative to synthetic reinforcements in composite materials. Their potential for enhancing the mechanical integrity of eco-friendly composites aligns with the increasing demand for high-performance materials with a reduced environmental impact.

The structural composition of yucca fibers contributes significantly to their mechanical performance. These fibers are primarily composed of cellulose, hemicellulose, lignin, and a small proportion of waxes and pectins. The high cellulose content is largely responsible for their strength and stiffness, while the presence of hemicellulose and lignin influences fiber flexibility and thermal stability. Furthermore, the hierarchical microstructure of yucca fibers, featuring a dense arrangement of fibrils and a well-organized lamellar structure, enhances load transfer when embedded in a polymeric matrix. This natural architecture, combined with the fiber's low density, makes yucca a promising reinforcement candidate for the development of lightweight and high-strength bio-composites.

Figure 2.1- Yucca plant

2.3.1 Yucca fiber

Yucca fiber, primarily extracted from the leaves, represents a promising natural reinforcement for various applications, particularly in textiles and bio-composites. The fiber's composition consists predominantly of cellulose, hemicellulose, and lignin, with a high cellulose content conferring excellent mechanical properties, including remarkable tensile strength and enhanced flexibility. Moreover, its low density and intrinsic porosity facilitate efficient integration into polymer matrices, while its surface chemistry allows for improved adhesion through targeted chemical modifications.

Due to their exceptional mechanical strength, cost-effectiveness, and compatibility with polymer matrices, yucca fibers present a sustainable and innovative alternative for the development of high-performance bio-composite materials. Their integration into bio-based composites contributes to the advancement of green materials, meeting the growing global demand for environmentally responsible engineering solutions.

2.3.2 Selection of yucca

In order to study the impact of the extraction methodology on the properties of the final materials and the development of bio-composites, the choice of the yucca plant for the extraction of natural fibers was based on several specific criteria.

- Untapped research potential: Despite its prevalence in arid regions, yucca remains underexplored compared to other natural fiber sources. This offers significant opportunities for novel research in fiber-reinforced bio-composites and sustainable material development.
- Non-competition with food and pharmaceutical industries: Unlike many other fibrous plants, yucca is not commonly utilized in human food or pharmaceutical applications. This eliminates stringent regulatory constraints associated with food safety, enabling greater flexibility in its use for composite materials.
- Abundance in Algeria: Yucca is widely available in Algeria, particularly in arid and semi-arid regions, providing an abundant and locally sourced raw material for fiber extraction. This availability enhances both the economic feasibility and sustainability of large-scale bio-composite production.
- Ease of fiber extraction: The extraction of yucca fibers is relatively straightforward and cost-effective compared to other natural fibers, optimizing production costs and facilitating industrial-scale manufacturing.
- Superior physicochemical and mechanical properties: Yucca fibers exhibit high tensile strength, flexibility, and resilience, making them excellent candidates for reinforcement in composite matrices. Their favorable physicochemical attributes enhance their compatibility with various eco-friendly polymer systems.
- Minimal environmental impact: As a natural fiber, yucca represents a sustainable alternative to synthetic reinforcements, contributing to the development of bio-composites with a significantly reduced ecological footprint. This aligns with global sustainability objectives and the promotion of renewable materials in engineering applications.

These attributes position yucca as an optimal candidate for bio-composite reinforcement, offering an environmentally sustainable and high-performance alternative to conventional synthetic fibers.

2.4 Extraction methods

The extraction of natural fibers is an essential process that directly influences the quality, structural integrity, and performance of fibers intended for industrial applications. Effective extraction methods facilitate the separation of fibers from the non-cellulosic components of the plant while preserving their mechanical and physicochemical properties [118].

In this study, various extraction techniques are employed with aims to obtain and isolate yucca fibers from the leaves, as shown in **Figure 2.2**. The selection of an appropriate extraction method is essential to optimize fiber quality and ensure its suitability for bio-composite applications.

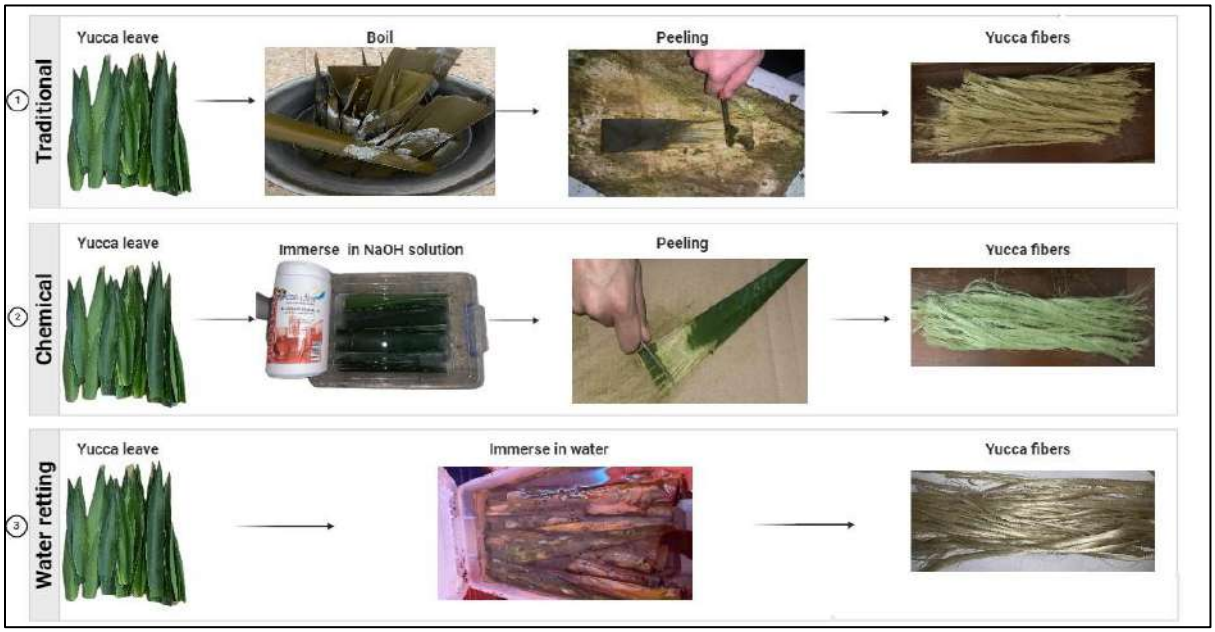

Figure 2.2- The different extraction processes used for yucca fiber.

2.4.1 Algerian yucca leaves harvest

The yucca leaves used in this study were harvested from specific regions in Algeria where the plant is abundantly available, particularly in the Mohamed Khemisti Garden, located in Hadjout town within the Tipaza province, as shown in **Figure 2.3**. The exact geographical coordinates of the collection site are 36°30'42"N 2°24'50"E, as determined via Google Earth. The climatic conditions of this region provide a favorable environment for yucca growth, ensuring the availability of mature leaves suitable for fiber extraction.

The harvesting process was conducted manually, with careful selection of mature, healthy leaves exhibiting optimal dimensions, ranging from 70 to 110 cm in length and 3 to 6 cm in width. These parameters were chosen to ensure the extracted fibers possess sufficient length and homogeneity for bio-composite applications. Leaves showing visible defects such as cracks, discoloration, or fungal contamination were excluded to maintain the integrity and quality of the extracted fibers.

This approach not only preserves the mechanical and physicochemical properties of the fibers but also underscores the sustainable utilization of an abundant, renewable local resource. Furthermore, the manual harvesting method minimizes environmental impact while ensuring that the leaves are adequately prepared for subsequent fiber extraction processes.

Figure 2.3- Yucca leaves harvest location.

2.4.2 Water retting

Water retting is a natural fiber extraction process that relies on microbial activity to degrade the non-cellulosic components binding the fibers within the leaf matrix [119]. In this study, multiple yucca leaves were submerged in a basin of river water for a period of 35 to 40 days at ambient temperature. This prolonged immersion facilitates the breakdown of hemicellulose, pectin, and other binding substances through the action of anaerobic bacteria naturally present in the aquatic environment, as illustrated in **Figure 2.4**. The enzymatic activity of these microorganisms accelerates the degradation process, thereby facilitating the separation of the fibers with minimal mechanical intervention.

Figure 2.4- Yucca leaves in a water bath, and attacked by micro-organisms.

Following the retting period, the extracted fibers were thoroughly rinsed with distilled water to remove any residual organic matter and microbial contaminants. The fibers were then air-dried at room temperature for at least seven (7) days to ensure complete removal of moisture while preserving their structural integrity.

2.4.3 Traditional extraction

Traditional fiber extraction methods have been employed for centuries and rely on manual techniques to separate fibers from the non-cellulosic components of the plant. In this technique, yucca leaves were cut into segments of approximately 30 cm and subjected to boiling in water at 80°C for one hour. This thermal treatment softens the plant tissue, facilitating the breakdown of lignocellulosic components and rendering the organic material more pliable and easier to manipulate.

After boiling, the leaves segments were allowed to cool at ambient temperature for 24 hours. During this period, the softened non-cellulosic material further degraded, simplifying fiber separation. The fibers were then manually extracted through a peeling process to remove all residual non-fibrous material (**Figure 2.5**). Finally, the fibers were washed with distilled water to eliminate any remaining organic residues and air-dried at room temperature for seven days to achieve optimal moisture content.

Figure 2.5- Yucca fibers being extracted using the traditional, peeling process.

2.4.4 Chemical extraction

Chemical extraction is an efficient technique for obtaining high-quality fibers while optimizing their physicochemical and mechanical properties. In this method, yucca leaves were immersed in sodium hydroxide (NaOH) solutions at varying concentrations (3%, 5%, and 10%) for a duration of five hours at 20°C. This alkaline extraction method effectively dissolves hemicellulose, pectin, and other amorphous constituents, thereby facilitating fiber separation with minimal mechanical intervention.

However, careful process control is necessary to prevent excessive degradation of the fibers and to mitigate the environmental impact associated with chemical extraction technique. To neutralize residual NaOH and halt the chemical reaction, the extracted fibers were thoroughly rinsed with distilled water. The fibers were subsequently air-dried at room temperature to ensure proper stabilization and preservation of their structural integrity.

2.4.5 Mechanical extraction

Mechanical extraction is a widely used method for obtaining natural fibers. In this study, the fiberizing method was employed to extract fibers from yucca leaves. This technique involves the application of controlled mechanical forces to break down the non-fibrous components and isolate the fibers while preserving their structural integrity.

As a first step, the yucca leaves were cut into uniform segments and subjected to a crisping process using mechanical rollers. This procedure facilitated the removal of excess moisture and partially loosened the non-cellulosic material. The crisped leaves were subsequently manually processed through a peeling procedure, which applied controlled mechanical forces to separate the fibers from the remaining plant matrix.

Following fiber separation, the extracted fibers were thoroughly washed with distilled water to eliminate residual plant matter and impurities. The fibers were then air-dried at room temperature for at least seven days to ensure optimal moisture content and structural stabilization.

2.5 Natural fiber characterization

Characterization of natural fibers is essential for assessing their chemical, morphological, and structural properties, which directly influence their performance in bio-composites. In this study, various analytical methods were employed to evaluate the properties of yucca fibers, including chemical composition, morphological structure, functional group analysis, crystallinity proprieties, and mechanical properties**.**

2.5.1 Chemical composition calculation

The chemical composition of the fibers was analyzed to quantify their main constituents, namely cellulose, hemicellulose, and lignin**.** These analyses were conducted following standardized protocols**,** ensuring the reproducibility and accuracy of the results [120]. The relative distribution of these components is a key indicator of the mechanical performance, chemical stability, and compatibility of the fibers with polymer matrices**,** directly influencing their suitability for bio-composite applications.

In this part of study, the mass fractions of the chemical constituents of yucca fibers were determined using the following analytical procedures:

2.5.1.1 Determination of Cellulose Content:

A mass of 2 grams of yucca fibers (Ws) was subjected to a chemical treatment using a solution composed of 40 mL of 80% acetic acid and 2 mL of concentrated nitric acid. The mixture was heated under reflux to initiate and sustain the reaction for a duration of 30 minutes. Following this treatment, the fibers were separated by centrifugation at 15,000 rpm for 5 minutes in hot ethanol (95%), then filtered under

vacuum. The washing protocol was carried out sequentially using hot benzene, followed by ethanol (95%), and finally petroleum ether to ensure the removal of residual impurities. The treated fibers were subsequently dried in an oven at 105°C for 1 hour. At this stage, their weight was recorded in a hermetically sealed container, yielding the mass ($W2$).

In the final stage, the dried sample was placed in a muffle furnace at 500°C for 3 hours to eliminate all organic matter, leaving only the inorganic ash content. The weight of the remaining ashes was measured and recorded as ($W1$).

The cellulose content of the treated fibers was determined using the following equation (**2.1**)

$$\%Cellulose = \frac{W2 - W1}{Ws} \times 100 \quad \textbf{(2.1)}$$

Where:

- Ws: Represents the initial dry mass of the yucca fibers (2 g),
- W2: Corresponds to the mass of the sample after chemical treatment and drying,
- W1: Denotes the residual ash content after combustion at 500°C.

This methodology enables an accurate quantification of cellulose content by eliminating non-cellulosic components and inorganic residues, thereby providing a reliable assessment of the fiber's chemical composition.

2.5.1.2 Determination of hemicellulose content

A mass of 2 g of yucca fibers was subjected to an alkaline treatment using a 5% (w/v) potassium hydroxide (KOH) solution for 2 hours. Following this treatment, the mixtures were filtered to separate the solid residue from the liquid phase. Hemicellulose was subsequently precipitated from the filtrate by the addition of ethanol (95%). The resulting precipitate was isolated by centrifugation at 15,000 rpm for 15 minutes.

The recovered hemicellulose was then dried in a convection oven at 105°C for 1 hour. Upon completion of the drying process, its mass was recorded in a hermetically sealed container, yielding the measurement denoted as (W1). To determine the ash content, the dried sample was subjected to calcination in a muffle furnace at 500°C for 3 hours. After cooling, its final mass was measured within a sealed vessel, providing the weight of the residual ash (Wa).

The hemicellulose content (%) was calculated using the following equation (**2.2**):

$$\%Hemicellulose = \frac{WA}{W1} \times 100 \quad \textbf{(2.2)}$$

In this context, W1 is the mass of the precipitated hemicellulose after drying, Wa represents the ash content after calcination at 500°C.

This methodology enables an accurate quantification of the hemicellulose fraction by eliminating non-hemi-cellulosic components and inorganic residues, thereby providing a reliable assessment of the fiber's biochemical composition.

2.5.1.3 Determination of lignin content

With aim to determine the lignin content, 2 g of fibers (Wa) were analyzed following the ASTM D1106 standard. The biofiber samples were initially treated with 72% sulfuric acid (H_2SO_4) for 2 hours to induce partial hydrolysis. Subsequently, the mixture was subjected to reflux conditions for an additional 3 hours to ensure complete lignin isolation.

After the reaction, the insoluble residue was separated by filtration and thoroughly washed with hot water to remove any residual acid. The recovered solid fraction was then dried in a convection oven at 105°C for 1 hour, after which its mass was recorded (W1).

The dried residue was subsequently subjected to calcination in a muffle furnace at 500°C for 3 hours. After cooling under ambient conditions, the final weight of the ash content was measured in a sealed container (W2).

The lignin content (%) was calculated using the following equation (**2.3**):

$$\%Lignin = \frac{W1 - W2}{Wa} \times 100 \quad \textbf{(2.3)}$$

Where:

- Wa: Represents the initial dry mass of the fiber sample (2 g),
- W1: Is the mass of the lignin residue after drying,
- W2: Denotes the ash content after calcination at 500°C.

This methodology ensures an accurate determination of the lignin fraction by effectively eliminating non-lignin components and inorganic residues, providing critical insights into the chemical composition of the fibers.

2.5.2 Scanning electron microscopy (SEM) analysis

Scanning Electron Microscopy (SEM) was utilized to examine the morphology and surface structure of the extracted natural fibers. This technique provides valuable insights into the surface characteristics following extraction and chemical treatments, including roughness, impurities, cracks, and the potential adhesion between the fibers and the polymer matrix. Comparative analysis between untreated and treated fibers allows for a better understanding of the structural modifications induced by different extraction methods [121].

2.5.2.1 Morphology analysis of yucca fiber

The surface morphology of yucca fibers was analyzed using a JEOL JSM-7100F scanning electron microscope (Figure 2.6 A)**.** Prior to observation, the fibers were carefully prepared to ensure high-quality imaging. To enhance conductivity and improve image resolution, the samples were coated with a thin layer of carbon using a vacuum sputtering system (Figure 2.6B)**.** This step is crucial for preventing electrostatic charge accumulation during SEM analysis.

Observations were performed at different magnification levels to capture both macrostructural and microstructural features. Special attention was given to parameters such as fiber diameter, surface roughness, and the presence of residual non-cellulosic material, as these factors influence the fiber's interfacial adhesion with polymer matrices in bio-composites.

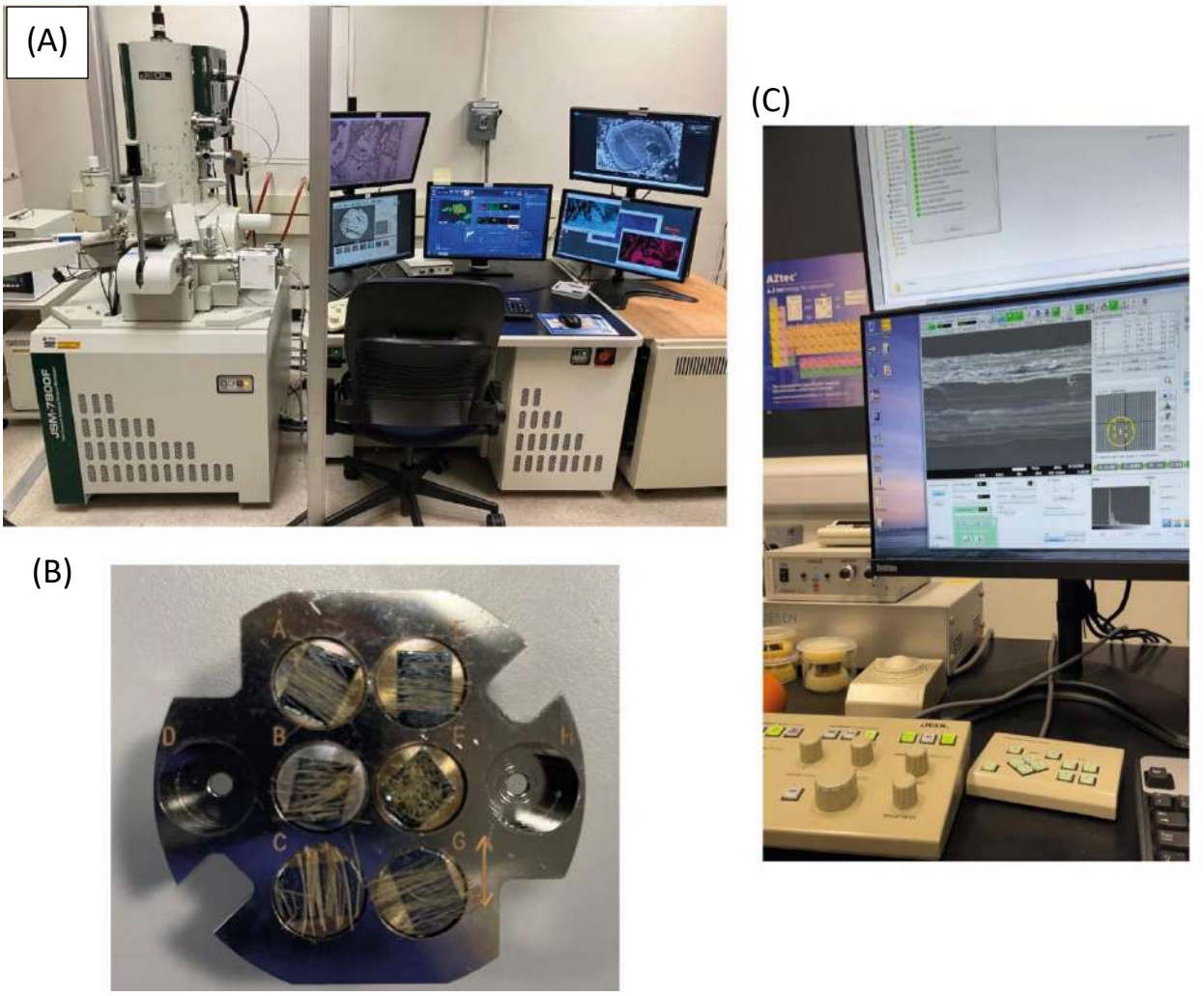

Figure 2.6- SEM analysis was conducted on yucca fibers. (A) Represents the JEOL scanning electron microscope used for the analysis, (B) shows the prepared yucca fiber sample prior to SEM examination, and (C) illustrates the JEOL machine in operation during the test.

2.5.3 Fourier transform infrared spectroscopy (FTIR) analysis

FTIR spectroscopy was employed to characterize the functional groups present in yucca fibers extracted using different methods. This analytical technique allows for the identification of characteristic absorption bands associated with cellulose, hemicellulose, and lignin, thus providing insights into the chemical composition and structural modifications of the fibers after extraction. Furthermore, the presence or absence of specific functional groups helps determine the efficiency of the extraction process in removing non-cellulosic components [122].

2.5.3.1 Functional group analysis of yucca fiber

The chemical bonds and functional groups within the yucca fibers were examined through Fourier Transform infrared spectroscopy (FTIR)**,** as illustrated in **Figure 2.7**. FTIR spectra were recorded over a wavenumber range of 4000 to 500 cm^{-1} using a high-resolution infrared spectrometer. Prior to analysis, fiber samples were dried at 105°C for 24 hours to eliminate residual moisture, which could interfere with the spectral measurements. The resulting spectra were analyzed to identify key functional groups indicative of structural modifications, degradation, or enhancements induced by different extraction techniques.

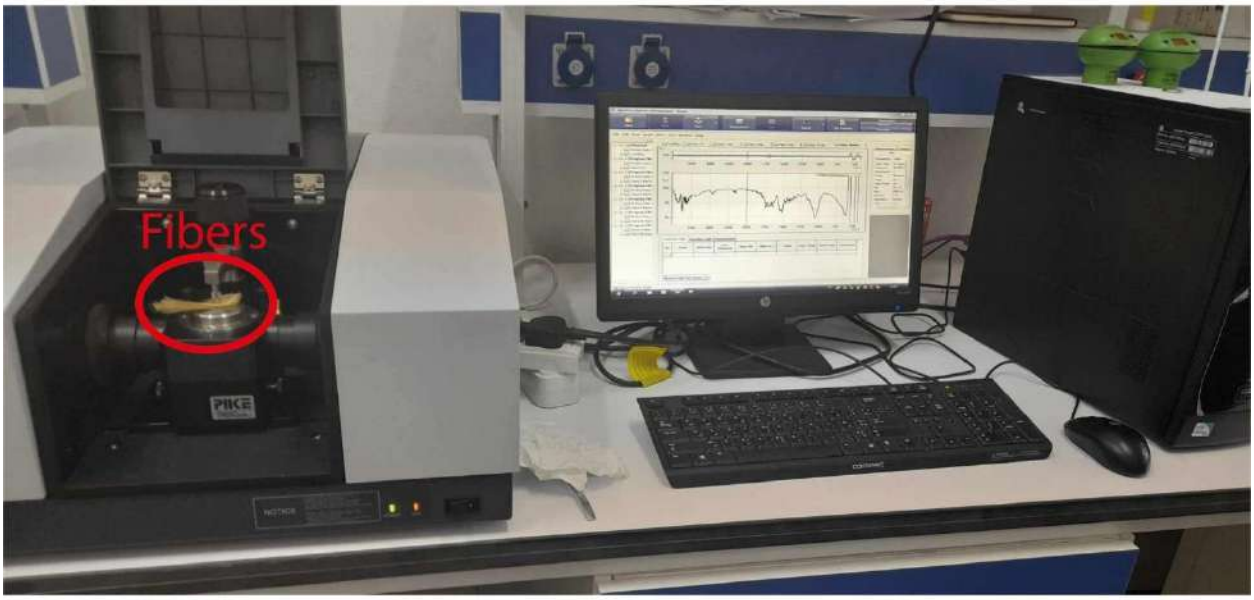

Figure 2.7 - Yucca fibers undergoing analysis in the FTIR spectrometer during the test.

2.5.4 X-ray diffraction (XRD) analysis

X-ray diffraction (XRD) was employed to evaluate the crystallinity index and crystallite size of yucca fibers. This analysis provides critical insights into the molecular organization of cellulose, enabling the distinction between crystalline and amorphous domains. The degree of crystallinity is a key parameter influencing the mechanical properties of natural fibers, particularly their stiffness, strength, and thermal stability [123].

2.5.4.1 Crystallinity analysis of yucca fiber

The crystalline structure of yucca fibers was characterized using X-ray diffraction (XRD) to assess the impact of extraction methods on their crystallinity index and crystallite size. XRD is an essential technique for biofiber characterization, as it differentiates crystalline regions, predominantly composed of well-ordered cellulose chains, from amorphous regions, primarily associated with lignin and hemicellulose.

The diffraction analyses were performed using a PANalytical X'Pert PRO diffractometer (**Figure 2.8A**), operating at 40 kV and 30 mA with CuKα radiation (λ = 1.5406 Å). The fiber samples were ground into fine powder and compacted to ensure homogeneous diffraction measurements, as illustrated in **Figure 2.8B**. Data acquisition was conducted over a 2θ range of 5° to 90°, with a step size of 0.02° and a counting time of 2 seconds per step.

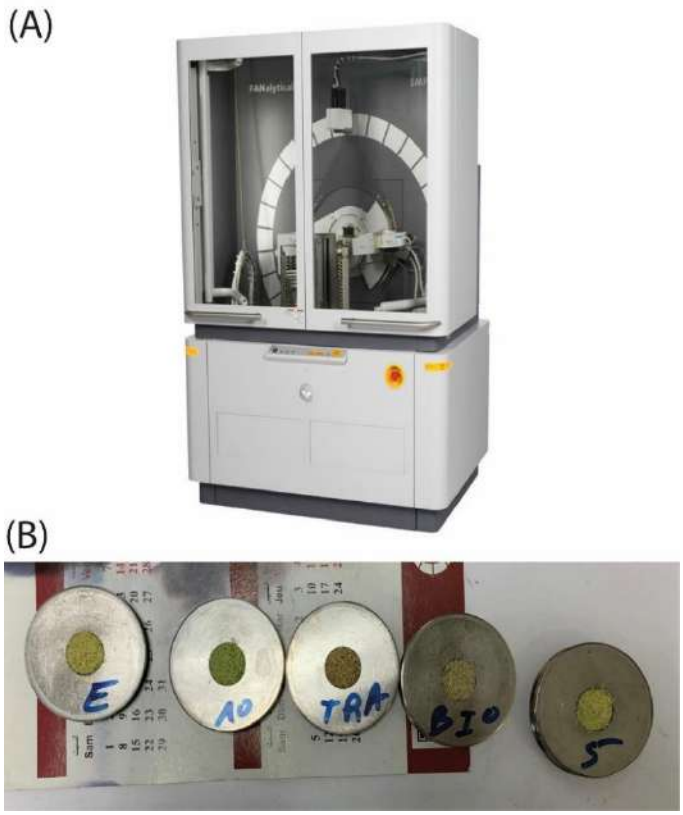

Figure 2.8 - DRX analysis was performed on yucca fibers. (A) Represents the PANalytical X'Pert PRO diffractometer used for the analysis, (B) shows the prepared yucca fiber powder sample prior to SRX examination.

The crystallinity index (CI) was determined using the Segal equation [124] (**2.4**), given by:

$$CI = \frac{I_{200} - I_{am}}{I_{200}} \times 100 \quad \textbf{(2.4)}$$

With:

CI: is the crystallinity index (%)

I_{200}: is the maximum intensity of the crystalline peak at about 2θ=22° (associated with the (200) plane of crystalline cellulose). I_{am}: is the maximum intensity of the amorphous peak 2θ=16°and 34°

In addition, the average crystallite size (L) was calculated using the Scherrer equation [125] (**2.5**):

$$L = \frac{K \times \lambda}{\beta \times cos\,\theta} \quad \textbf{(2.5)}$$

In this context, L is crystal size in nanometers (nm). K is the form factor (generally K=0.9 for cellulose). λ is the wavelength of the X-ray radiation (1.5406 for CuKα). β is the width at half maximum (FWHM) of the diffraction peak (in radians). θ is diffraction angle corresponding to the peak (2θ/2).

2.5.5 Mechanical analyses

Tensile testing of natural fibers is a critical evaluation method for determining their intrinsic mechanical properties, including tensile strength, Young's modulus, and elongation at break. These parameters are fundamental to assessing the reinforcing potential of fibers within bio-composite materials, as they directly influence the mechanical integrity and load-bearing capacity of the final composite structure [126, 127].

2.5.5.1 Yucca single fiber tensile test

The mechanical behavior of yucca fibers was evaluated through single-fiber tensile testing, conducted in accordance with ASTM D3822, a standardized method for assessing the tensile properties of individual fibers [128]. This analysis aimed to quantify the tensile strength, Young's modulus, and elongation at break, which are essential for determining the suitability of these fibers as reinforcement in polymer-based composites.

With aim to ensure statistical reliability, a total of 20 individual fiber specimens were tested for each extraction method. The selected fibers were cut to a standardized length of 50 mm, with precise measurements taken prior to testing. To facilitate specimen handling and minimize slippage or damage,

each fiber was carefully mounted onto a paper frame using a mild adhesive. This frame, ensuring fiber alignment, was subsequently clamped within the grips of the tensile testing machine. Prior to testing, the edges of the paper frame were carefully trimmed, leaving only the fiber exposed for mechanical loading, as illustrated in **Figure 2.9**.

Tensile tests were performed under controlled laboratory conditions at room temperature using a Zwick universal testing machine equipped with a 2.5 kN load cell. A constant displacement rate, as recommended by ASTM D3822, was applied to obtain precise stress-strain curves. The recorded data were subsequently analyzed to determine the mechanical performance of the fibers and to compare the effects of different extraction methods on their tensile properties.

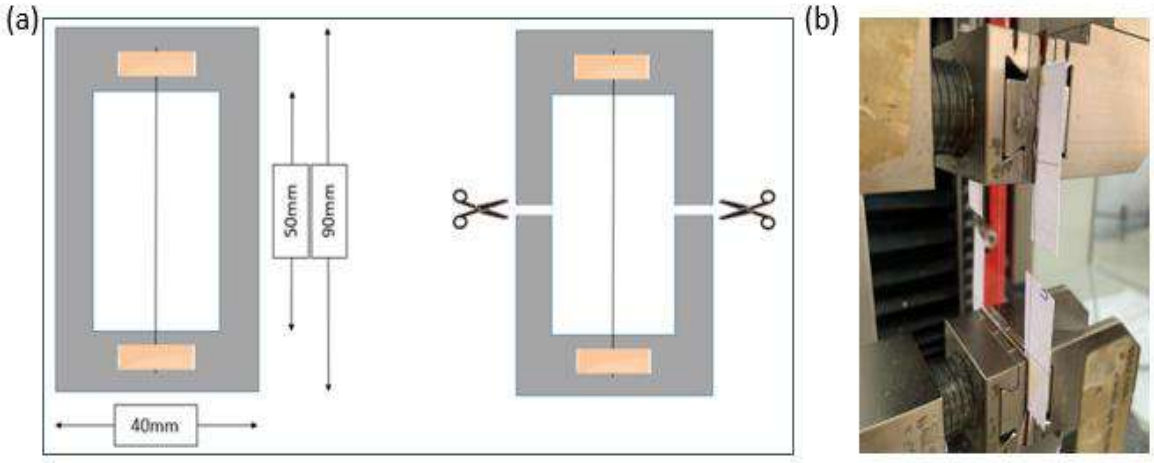

Figure 2.9- Tensile tests on a single natural fiber. (a) represent the fiber fixing scheme and (b) is the fiber during the tensile test.

2.5.5.2 Data analysis methodology

At the conclusion of the tensile tests, the mechanical properties of the fibers, including tensile strength, Young's modulus, and elongation at break were analyzed using statistical methods. Specifically, the arithmetic mean was calculated for each parameter based on the 20 tested samples per extraction method, ensuring a representative evaluation of the mechanical behavior of the fibers. This statistical approach enables the identification of variations associated with different extraction techniques and provides insights into their influence on fiber performance [129].

The adopted methodology guarantees that the fibers are tested under optimal conditions, minimizing the risk of pre-test damage while ensuring high measurement accuracy. The results obtained

from these analyses are crucial for assessing the impact of fiber extraction methods on mechanical performance and determining their suitability as reinforcements in high-performance bio-composites.

2.6 Bio-composite elaboration

This section outlines the fabrication processes employed for the development of bio-composites reinforced with yucca fibers. Two distinct manufacturing techniques were utilized: casting molding and 3D printing, both aimed at producing samples with enhanced mechanical and physical properties for subsequent characterization.

2.6.1 Matrix

In bio-composites, the matrix serves as a critical component, binding the reinforcing fibers and ensuring efficient stress transfer throughout the material. The selection of an appropriate matrix significantly influences the overall mechanical performance, thermal stability, and durability of the composite. Thermoplastic polymers such as polylactic acid (PLA) and thermosetting resins such as epoxy are widely employed due to their superior mechanical properties, ease of processing, and compatibility with natural fibers [130].

2.6.1.1 Thermoplastic matrix:

Polylactic acid (PLA) is a bio-based thermoplastic polymer synthesized from renewable resources such as corn starch and sugarcane. PLA is widely recognized for its biodegradability, reduced environmental impact, and suitability for sustainable applications [131]. In this study, PLA (type 4043D, NatureWorks Ingeo Biopolymer, USA) was selected as the matrix material (**Figure 2.10**). The polymer exhibits a density of approximately 1.24 g/cm³ and a melting temperature ranging from 145°C to 160°C, making it an appropriate choice for bio-composite processing.

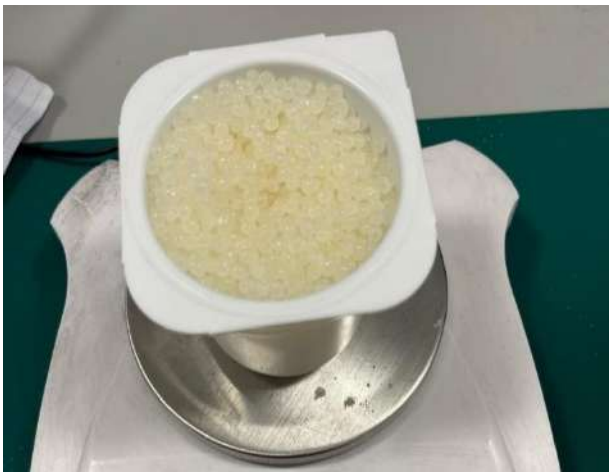

Figure 2.10 - Pure PLA pellets used in this study.

2.6.1.2 Thermosetting matrix

Epoxy resin is a thermosetting polymer extensively used in composite manufacturing due to its high mechanical strength, superior chemical resistance, and excellent adhesion to reinforcement materials [132]**.** In this study, epoxy was selected for composite formulations requiring enhanced stiffness and load-bearing capacity. The Epoxy matrix was prepared by mixing resin and hardener in a 70:30 ratio, following the manufacturer's specifications (Genc, Turkey), the resin used are presented in **Figure 2.11**.

Figure 2.11- Epoxy used in this study from GENC company.

2.6.2 Yucca fiber type

In this study, yucca fibers were utilized in the form of a fine powder with a particle size of less than 500 µm, as illustrated in **Figure 2.12**. This specific morphology was selected due to its ability to achieve homogeneous dispersion within the polymer matrix**,** thereby enhancing fiber-matrix interactions and contributing to the overall mechanical and physicochemical performance of the bio-composite.

Figure 2.12 - Yucca fiber in powder form.

The fiber preparation process involved a controlled drying step at 105°C for seven days, followed by mechanical grinding using a high-speed rotary blade mill to ensure uniform fragmentation. The resulting fiber powder was then sieved using a calibrated 500 µm mesh, ensuring a consistent particle size distribution by eliminating larger fragments. This standardized sieving step is critical to achieving uniform fiber dispersion within the matrix, minimizing local agglomerations, and enhancing the reproducibility of the composite's properties.

The powdered form of yucca fiber was particularly advantageous for casting molding and 3D printing techniques, as it facilitates efficient processing and improves the material's overall structural integrity. This methodological approach was strategically chosen to maximize composite performance while ensuring experimental consistency and reproducibility.

2.6.3 Samples preparation

A rigorous and standardized protocol was followed for the preparation of bio-composite samples to ensure uniformity, reproducibility, and reliability of the results. The yucca fibers, in the form of a fine powder (<500 µm), were precisely weighed using an analytical balance (accuracy: 0.001 g) to achieve the exact fiber-to-matrix mass ratios required for the study. The polymer matrices, PLA and epoxy**,** were prepared according to the technical specifications provided by the manufacturers**.**

2.6.4 Compliance with ASTM standards

The sample preparation strictly adhered to the standards established by the American Society for Testing and Materials (ASTM) to ensure comparability with existing literature and industrial applicability. These standards provide precise specifications regarding the dimensions, geometry, and preparation protocols of samples intended for mechanical and physicochemical characterization **(Figure 2.13).**

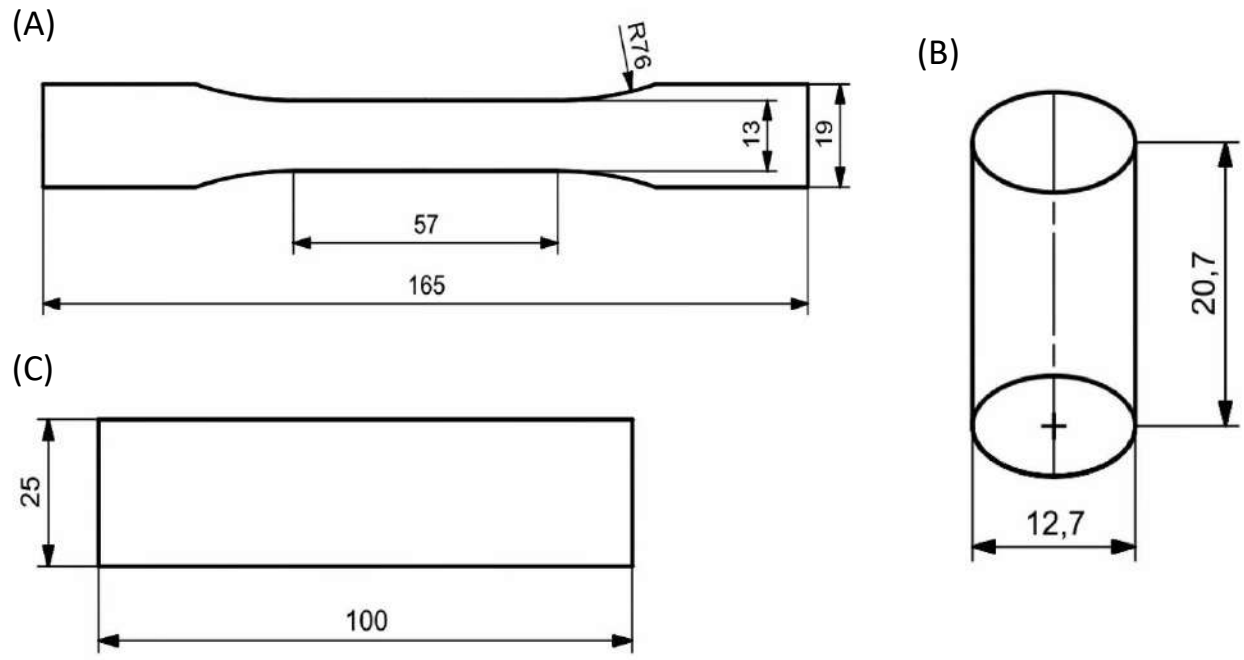

Figure 2.13 - Samples design for mechanical testing. (A) Sample design for tensile test – ASTM D638. (B) Compression sample design – ASTM D695, and (C) Bending sample design – ASTM D790. (All dimensions expressed in mm).

In this part of the study, ASTM D638 was adopted for the tensile tests [133]. This standard defines the specific dimensions of the specimens, including a uniform gauge zone to minimize variations during testing. Specimens for bending tests were prepared in accordance with ASTM D790, while those for compression tests followed the requirements of ASTM D695 [134, 135]. In addition, other test samples, such as mechanical fatigue analysis, and even Charpy test, have been prepared in accordance with the ASTM standard, as summarized in **Table 2.1**.

Table 2.1- The standards used in the other different tests in this study

Test	Standard ASTM	Ref.
Fatigue	ASTM D7791	[136]
Charpy impact	ASTM D6110	[137]

To ensure compliance with these standards, 3D-printed and cast samples were meticulously adjusted when necessary. Dimensional tolerances were verified using a high-precision digital caliper (±0.01 mm accuracy), ensuring the accuracy and repeatability of the measurements.

By following ASTM standards, this study ensures that the mechanical properties of bio-composites can be directly compared with other materials studied in the literature, while also meeting academic and industrial quality criteria.

2.6.5 Bio-composite filament preparation

In the case of PLA-based biocomposite, the PLA granules were mixed with the yucca fibers powder using a mechanical mixer, resulting in a uniform pre-dispersion to obtain a fiber powder concentration of 1% and 3% in the mixture. The homogeneous mixture was then fed into a 3devo filament extrusion system (**Figure 2.14**).

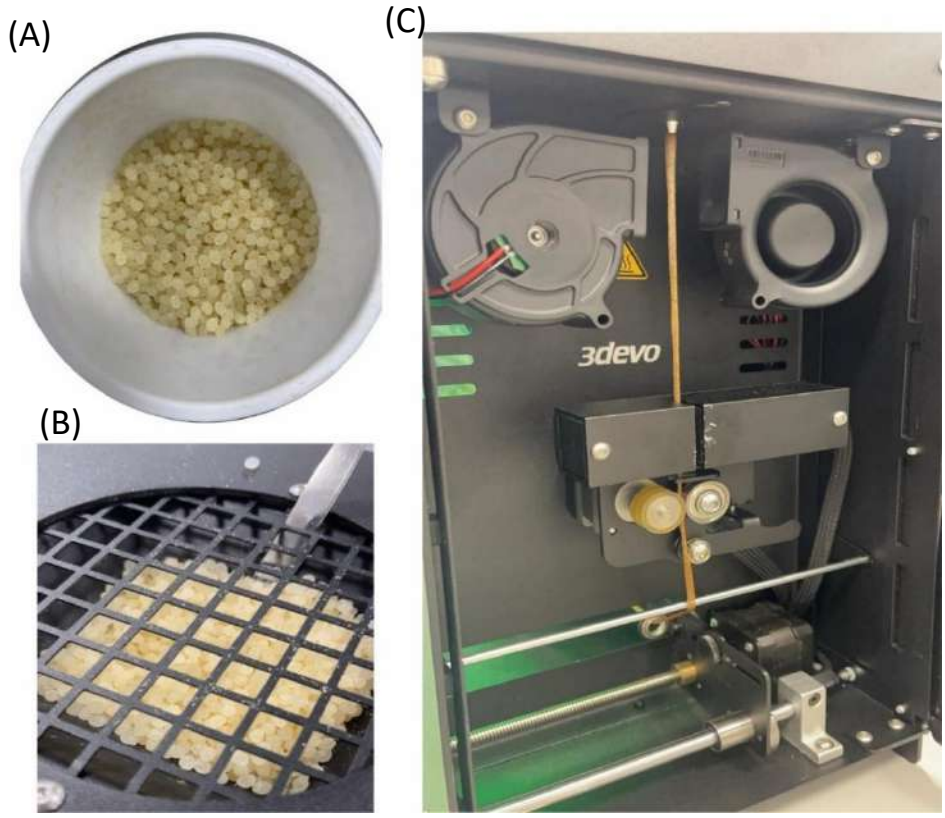

Figure 2.14 - Biocomposite filament fabrication. (A) PLA/yucca powder mixture, (B) the mixture in the extrudes, (C) the extrusion process.

The extrusion process was carried out at controlled temperatures specific to PLA, ensuring uniform fiber distribution within the polymer matrix. The resulting bio-composite filaments (Ø = 2.85 mm) were characterized to confirm their structural integrity and suitability for 3D printing. The extrusion parameters, including temperature zones and screw speed, are summarized in **Table 2.2**.

Table 2.2 - The parameters required for correct extrusion.

Parameters (unit)	Settings
Temperature (◦C) (Z1, Z2, Z3, Z4)	170, 185, 190, 180
Screw speed (rpm)	5
Filament fan speed (%)	60
Spooling speed (rpm)	Auto
Filament diameter (mm)	2.85

2.6.6 3D printed of bio-composites

The fabrication of bio-composite samples via 3D printing followed a highly controlled methodology to ensure dimensional precision and mechanical integrity. The Fused Deposition Modeling (FDM) Ultimaker S3 was used (**Figure 2.15**), where the pre-extruded bio-composite filaments were processed under optimized printing conditions.

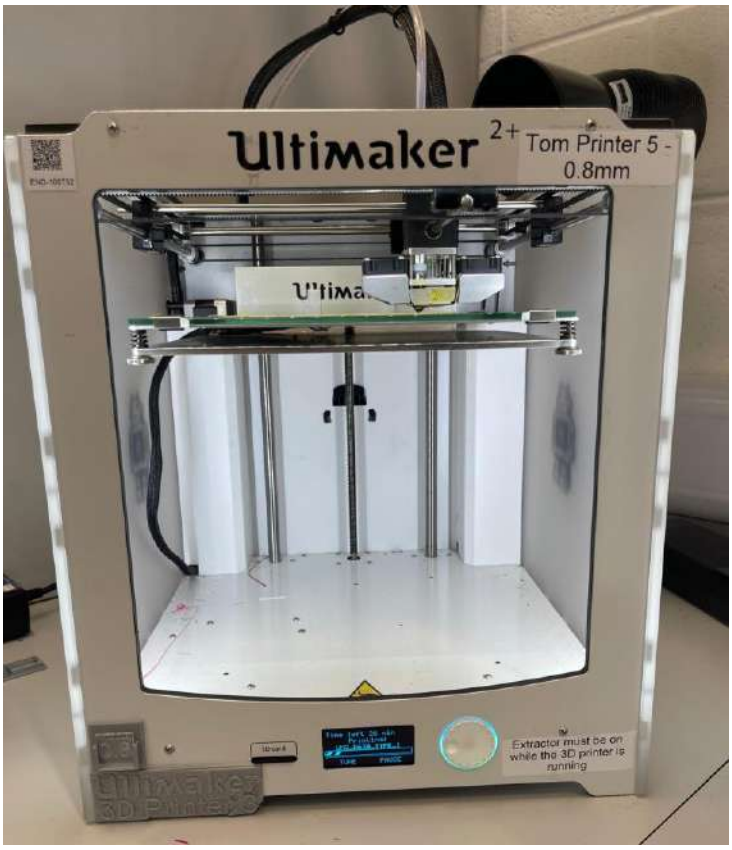

Figure 2.15- FDM 3D printer model Ultimaker S3 used in this study.

The biocomposite filaments were then fed into an FDM-type 3D printer equipped with a 0.8 mm nozzle, which was carefully calibrated to optimize the printing parameters. The samples were designed using CAD software, with dimensions meeting the normative requirements of mechanical testing. The nozzle temperature (200-220°C), heating plate temperature (60°C), printing speed (50 mm/s) and layer height (0.2 mm) were precisely adjusted using *CURA* software. These parameters were determined to ensure smooth extrusion, satisfactory inter-layer adhesion and to prevent any alteration in the properties of the bio-composite during the printing process.

After printing, the samples were carefully inspected visually for any defects, such as under-extruded areas, air bubbles or surface irregularities. To stabilize their physico-mechanical properties, the

printed samples were conditioned in an air-conditioned chamber at a controlled temperature (25°C) for a minimum of 48 hours. This conditioning guarantees the homogeneous properties of the samples before they are subjected to mechanical, thermal and physico-chemical analysis.

2.6.7 Casting mold elaboration

For the epoxy-based biocomposites, the resin and hardener were mixed according to the manufacturer's specifications in a precise ratio of 70:30. Yucca fibers powder were then gradually incorporated into the mixture under constant mechanical stirring to ensure homogeneous dispersion, targeting a fiber content of 10% by weight. This percentage was selected based on findings from the literature, aiming to achieve an optimal balance between mechanical properties and overall performance [138]. Once fully blended, the composite mixture was poured into pre-prepared silicone molds (**Figure 2.16**), designed to match ASTM standard specimen dimensions.

- Mold Preparation: The silicone molds were pre-cleaned and coated with a release agent (demolding wax) to facilitate easy sample extraction.
- Curing Process: The polymerization was carried out at ambient conditions (25°C) for 24–36 hours to allow complete crosslinking of the epoxy matrix.

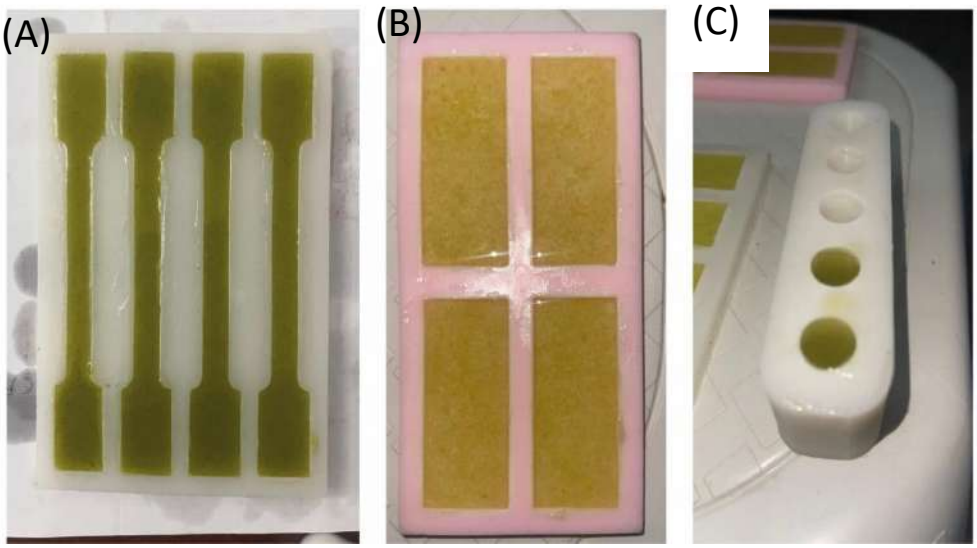

Figure 2.16 - The silicone molds used in this part of the study. (A) the composite tensile test mold, (B) the mold for bending test, and (C) the mold for the compression test.

After curing, samples were carefully demolded and visually inspected for cracks, porosities, or air inclusions. Before experimental testing, they were conditioned under controlled temperature and humidity to minimize variations due to environmental factors. This systematic approach ensures that all samples meet ASTM specifications**,** improving reliability, reproducibility, and comparability with other bio-composites studied in the field.

2.7 Bio-composite characterization

This section presents the methodologies employed to evaluate the performance of the developed bio-composites. The characterization techniques encompass mechanical, thermal, and physicochemical analyses, along with water absorption, dynamic behavior, and fatigue testing. These assessments provide a comprehensive understanding of the material's properties, facilitating its validation for various industrial applications.

2.7.1 Mechanical analyses definition

Mechanical characterization is essential for assessing the structural and functional performance of materials under various loading conditions. Key mechanical parameters such as tensile strength, bending strength, compressive strength, and impact resistance are evaluated to determine critical properties, including elastic modulus, yield strength, ultimate strength, and elongation at break. However, mechanical tests were conducted following internationally recognized standards, such as ASTM, to ensure consistency and reliability. These analyses are particularly significant for bio-composites, as they quantify the effects of natural fiber reinforcements and polymeric matrices (PLA or epoxy) on overall mechanical behavior.

The calculation of mechanical properties is based on rigid body mechanics (RDM) formulations, summarized in **Table 2.3:**

Table 2.3 - Rigid body mechanics formulations employed in this section of study.

Analysis	Measured property	Equation	Symbols
Tensile	Tensile strength	$\sigma_t = \frac{F}{A}$	F: Force applied (N) A: Cross-sectional area (mm^2)
	Young's modulus	$E = \frac{\sigma_t}{\varepsilon_t}$	σ_t: Tensile stress (MPa) ε_t: Tensile strain
	Elongation at break	$\varepsilon_t = \frac{\Delta L}{L_0}$	ΔL: Variation in length L_0: Initial length
Bending	Bending strength	$\sigma_f = \frac{3 \times F_f \times L}{2 \times b \times d^2}$	F_f: Force applied (N) L: Span length (50mm) b: Sample width (mm) d: Sample thickness (mm)
	Bending modulus	$E_f = \frac{L^3 \times F_f}{4 \times b \times d^3 \times D}$	F_f: Force applied (N) L: Span length (50mm) b: Sample width (mm) d: Sample thickness (mm) D: Proportional limit point deviation
Compression	Compressive strength	$\sigma_c = \frac{F_c}{A}$	F_c: Force applied (N) A: Cross-sectional area (mm^2)

These equations enable the accurate determination of mechanical properties based on applied forces and sample dimensions, ensuring rigorous and precise analysis.

2.7.1.1 Mechanical testing for bio-composites

In this study, mechanical testing was conducted on PLA- and epoxy-based bio-composites reinforced with treated and untreated yucca fibers powder.

2.7.1.1.1 Tensile test

Tensile tests were performed according to ASTM D638 on molded or 3D-printed specimens. Five samples per category were tested using a Zwick 8306 universal testing machine (10 kN load cell) for epoxy-based composites and a Shimadzu AG-X Plus machine (50 kN load cell) for PLA-based composites, both operating at a crosshead speed of 2 mm/min.

Testing Procedure:

- Samples were securely clamped to ensure proper alignment, minimizing off-center stresses.
- A constant displacement rate was applied, adhering to ASTM standards for repeatability.
- Force (F) and elongation (ΔL) were recorded throughout the test until failure.

Data Analysis:

- Stress-strain curves were generated to identify mechanical behavior phases (elastic, plastic, and fracture regions).
- Statistical analysis (mean and standard deviation) was performed for each category to ensure data reliability.

This analysis is essential for comparing the performance of bio-composites reinforced with yucca fibers with non-reinforced composites, identifying the effect of the fibers on the overall mechanical properties of the material, as well as the influence of the fiber extraction and treatment method.

2.7.1.1.2 Bending test

Five samples in each category were manufactured and tested in accordance with ASTM D790, to assess the mechanical properties of the bio-composites, including their bending strength and bending modulus of elasticity. In the case of epoxy specimens reinforced with yucca powder, whether treated or not, the analysis was performed using the Zwick-Line machine, which has a force cell of 2.5 kN and a span of 50 mm, the speed was programmed at 2 mm/min, in accordance with the international standard. In another hand, the yucca powder-reinforced PLA samples were analyzed on the Shimadzu AG-X Plus machine, which has a force cell of 50 kN and a span of 50 mm, at the same speed.

Testing Procedure:

- Samples were subjected to a three-point bending test with a central load application.
- Load was increased progressively until failure or a predetermined deformation limit.

Data Analysis:

- Statistical evaluation ensured consistency and reproducibility, with mean and standard deviation calculations providing a reliable assessment of mechanical properties.

This analysis provides a better understanding of the bending behavior of bio-composites made from natural fibers, offering crucial information for their use in industrial and composite materials applications.

2.7.1.1.3 Compression test

Compression tests were carried out in accordance with ASTM D695. Five samples of cylindrical bio-composites (*12.4 mm* in diameter and *25 mm* in height) were subjected to uniaxial compression testing using a universal testing machine, including Zwick 8306 (10 kN in force cell) and Shimadzu AG-X Plus (50 kN in force cell). The samples were placed between two parallel platforms, and a load was applied progressively (at a rate of *2 mm/min*) until the sample underwent plastic deformation or fracture.

Data Analysis:

- Statistical analysis was performed to evaluate consistency and reproducibility.
- The compressive strength of the bio-composites was compared to assess the impact of fiber reinforcement and treatment.

The compression test thus provides an overview of the resistance of yucca fiber-reinforced bio-composites to compressive forces, providing important information for the development of composite materials adapted to specific applications.

2.7.2 TGA analyses definition

Thermogravimetric analysis is essential for characterizing the thermal properties of bio-composites, as it enables to assess their thermal stability, their behavior under different temperatures and their ability to maintain their mechanical properties at high temperatures. Common thermal tests include thermogravimetric analysis (TGA), differential temperature analysis (DTA) and differential scanning calorimetry (DSC). These analyses are carried out to study mass changes, thermal transitions and degradation processes in materials [139].

2.7.2.1 Thermogravimetric analysis

Thermogravimetric analysis (TGA) measures the variation in the mass of a sample as a function of temperature or time, in a controlled environment (generally under a nitrogen or air atmosphere). This technique can be used to analyze a material's thermal stability and degradation behavior. It is particularly useful for determining degradation temperature, composition in terms of volatile and non-volatile phases, and degradation rates.

The curve obtained in the TGA test represents the residual mass as a function of temperature, making it possible to identify degradation stages, such as the loss of volatiles at moderate temperatures or the degradation of polymer components at higher temperatures. The results are used to determine the maximum temperature at which the material can be used and to study the thermal interactions between polymer matrices and natural fibers [140].

2.7.2.2 Differential temperature analysis

Differential temperature analysis (DTA) is often combined with TGA, but it specifically measures the temperature differences between the sample and a reference when heated. This makes it possible to detect melting points, phase transitions or other thermal phenomena. DTA is particularly useful for composite materials where several phases are present, making it possible to identify the temperature at which each phase undergoes thermal changes [141].

2.7.2.3 Experimental procedure

In this study, thermal analysis was conducted on bio-composite samples, specifically PLA filaments reinforced with yucca fiber powder. The samples, weighing approximately 20 mg, were subjected to thermal testing using the SDT-Q600 TA machine over a temperature range of 25°C to 650°C, at a heating rate of 10°C/min. This approach enables the assessment of the bio-composite's thermal stability by monitoring mass degradation and thermal transitions during heating. The results obtained will provide detailed insights into the thermal resistance of the bio-composite and the influence of yucca fiber extraction methods on its thermal behavior in the final material.

Analytical Procedure

- Approximately 20 mg of pure PLA and PLA-based bio-composite filaments reinforced with various concentrations of yucca fiber powder were prepared.

- The samples were placed in the machine's cell under a nitrogen atmosphere to prevent oxidation during testing.
- The temperature was gradually increased from 25°C to 650°C at a heating rate of 10°C/min. This range allows for the evaluation of the samples' thermal behavior at different temperatures, including the degradation temperatures of the organic components.

Data Collection and Analysis

- The machine continuously recorded the variation in sample mass as the temperature increased, allowing observation of mass loss due to the thermal degradation of bio-composite components.
- The mass loss curve was analyzed to identify degradation temperatures, melting points, and other relevant thermal transitions.
- The obtained results were processed using Origin software to evaluate the thermal stability of the samples by comparing the thermal behavior of pure PLA and PLA-yucca composites.

This analysis helps determine the maximum operating temperatures and assess potential improvements in thermal performance resulting from the addition of yucca fiber.

2.7.3 Physicochemical analyses

Physicochemical analyses of yucca fiber-reinforced bio-composites are essential for understanding the interactions between their components and assessing the effects of chemical treatments and material processing on their final properties. These analyses include morphological inspection using scanning electron microscopy (SEM) and spectroscopic techniques (FTIR) to identify the functional groups present on the surface of the fibers and bio-composites. Such characterizations provide fundamental insights into structural and chemical modifications as well as the interfacial properties between natural fibers and the polymer matrix.

2.7.3.1 Morphological inspection

To gain a detailed understanding of the bio-composite microstructure, morphological inspection was performed exclusively on bio-composite samples to qualitatively and quantitatively assess the dispersion homogeneity of yucca fibers within the polymer matrix and the fiber-matrix interfacial quality.

The examined samples were taken from the fractured regions of specimens previously subjected to tensile testing.

This characterization was conducted using a GEMINI SEM300 scanning electron microscope, a high-resolution instrument designed for detailed structural and topographical analysis of surfaces at the nanometric scale. Prior to imaging, the samples underwent a rigorous preparation protocol, including precise sectioning and metallization via the deposition of an ultra-thin carbon layer. This step was essential for enhancing surface conductivity and minimizing artifacts caused by electrostatic charge accumulation.

The SEM analysis enabled the spatial distribution of fibers within the matrix (PLA or epoxy) to be precisely identified, interfacial adhesion to be assessed, and the presence of structural defects such as microcracks, inclusions, or air bubbles potentially affecting the composite's mechanical performance to be detected. These observations, when correlated with mechanical and thermal testing data, provide a comprehensive understanding of the bio-composite's behavior and serve as a solid scientific foundation for optimizing manufacturing processes to enhance durability and functional performance.

2.7.3.2 Functional groups analyses

Fourier transform infrared spectroscopy (FTIR) is a key analytical technique for the chemical characterization of bio-composites. In this study, FTIR analysis was performed to identify and quantify the functional groups present in the polymer matrix and yucca fibers, as well as to evaluate the interfacial interactions between these components. The analysis was conducted using a Perkin-Elmer spectrometer. This technique is based on measuring molecular vibrations specific to chemical bonds, providing a characteristic spectral "fingerprint" of the material.

Sample preparation involved fine grinding and mixing with potassium bromide (KBr) to form transparent pellets. Spectra were recorded in the range of 4000 to 500 cm^{-1} with a resolution of 2 cm^{-1}. This analysis allows for a comparative evaluation of the chemical composition of bio-composites before and after treatment, facilitating the identification of structural modifications induced by the incorporation of yucca fibers into the polymer matrix.

2.7.4 Water absorption analyses

Water absorption analysis is an essential experimental method for evaluating the ability of bio-composites to absorb water when exposed to specific environments. This characterization provides insights into the material's dimensional stability and structural integrity by identifying the mechanisms through which water diffuses and interacts with both the polymer matrix and reinforcements. Understanding this behavior is crucial for predicting the durability of composite materials, particularly regarding hydrostatic resistance and the potential degradation of mechanical properties [142].

2.7.4.1 Experimental procedure

Water absorption testing is a key assessment of the dimensional stability and durability of bio-composites under water conditions. In accordance with the ASTM D570 standard, bio-composite samples were immersed in water at room temperature for predefined durations to quantify the amount of water absorbed over time. The experimental protocol consists of the following steps:

Sample preparation

- Fabricate or 3D-print the samples in accordance with ASTM D570 specifications.
- Accurately weigh each dry sample (M_o) using an analytical balance.
- Record the initial mass of each sample to ensure consistency in comparison.

Water immersion

- Submerge the samples in a container filled with spring water or seawater at room temperature.
- Ensure complete immersion to guarantee uniform exposure.

Periodic measurements

- Remove the samples at regular intervals (every 24 hours).
- Carefully wipe the surface with absorbent paper to remove excess water without disturbing the absorbed moisture.
- Weigh each sample immediately to determine the mass (M_t) at time t.

Water absorption calculation:

- Calculate the percentage of water absorption using the following equation (**2.6**)

$$WA(\%) = (\frac{M_t - M_0}{M_0}) \times 100 \quad \textbf{(2.6)}$$

- Measurements were repeated until the sample mass stabilized, indicating saturation (typically after 96 hours).

Data analysis

- A water absorption versus time curve was plotted for each sample.
- The trends were analyzed to determine the initial absorption rate, saturation points, and variations between samples.

By following these experimental steps in compliance with ASTM D570 standards, this study provides a precise evaluation of the water absorption capacity of bio-composites. These findings contribute to optimizing material design for enhanced durability and resistance in humid environments.

2.7.5 Dynamic mechanical analyses

Dynamic mechanical analysis (DMA) is an advanced experimental technique employed to evaluate the viscoelastic behavior of materials, particularly polymers and composites. This method enables the measurement of storage modulus (E'), which represents elastically stored energy, and loss modulus (E''), which corresponds to energy dissipated as heat. Additionally, DMA provides the dissipation factor (tan δ), defined as the ratio of E'' to E', which serves as an indicator of damping behavior. These parameters are critical for characterizing thermal transitions, such as the glass transition temperature (Tg), and for understanding the response of materials subjected to oscillatory stresses. This technique offers a comprehensive insight into the mechanical behavior of bio-composites under dynamic loading, facilitating the optimization of their formulation and long-term durability [143, 144].

2.7.5.1 Experimental procedure

For this study, bio-composite samples were prepared according to standardized dimensions (30mm × 10mm × 2mm) and subjected to DMA tests using a TA Instruments Q800 analyzer (**Figure 2.17**). The samples were positioned in a tension mode configuration and exposed to a low-amplitude oscillatory load to ensure linear viscoelastic behavior.

Figure 2.17- TA Instruments Q800 machine employed in this study.

The experimental protocol consisted of a temperature sweep conducted over a controlled range of 25°C to 80°C**,** with a heating rate of 5°C/min**,** to capture thermal transitions such as Tg and to evaluate the material's dimensional stability under thermal fluctuations. In addition, measurements were carried out at a constant frequency of 2 Hz to observe the frequency-dependent variations in E'**,** E"**,** and tanδ**,** providing a detailed assessment of the viscoelastic behavior of the composite.

The results were presented as graphical representations of storage modulus, and tanδ as functions of temperature or frequency. These data enable the identification of key dynamic mechanical characteristics, including the influence of yucca fiber reinforcement on material stiffness and energy dissipation. Moreover, the correlation of DMA findings with overall mechanical performance allows for a deeper understanding of the deformation mechanisms under cyclic loading. This knowledge is essential for optimizing the formulation of bio-composites, particularly for demanding industrial applications requiring enhanced mechanical stability and durability.

2.7.6 Charpy impact test

The Charpy impact test is a standardized method used to evaluate the energy absorption capacity of a material when subjected to sudden impact loading. Governed by ASTM D6110, this test measures the

energy absorbed by a notched specimen when struck by a pendulum hammer. The absorbed energy, expressed in joules (J), serves as a key indicator of material toughness, reflecting its resistance to crack initiation and propagation under dynamic conditions. The Charpy test is widely employed to assess the strength and ductility of materials, providing essential insights into their behavior under impact loading [145].

2.7.6.1 Experimental procedure

In this study, bio-composite specimens were prepared following the standardized dimensions prescribed by ASTM D6110, ensuring consistency in sample preparation. A notch of predefined depth and angle was introduced in each specimen to act as a stress concentrator, facilitating controlled crack propagation.

Each sample was then positioned horizontally on two fixed supports within the Charpy test apparatus. The pendulum was raised to a specified height, accumulating potential energy, before being released to strike the specimen precisely at the notch, as depicted in **Figure 2.18**. The difference between the initial kinetic energy of the pendulum before impact and the remaining energy after impact was recorded, allowing for the calculation of absorbed energy.

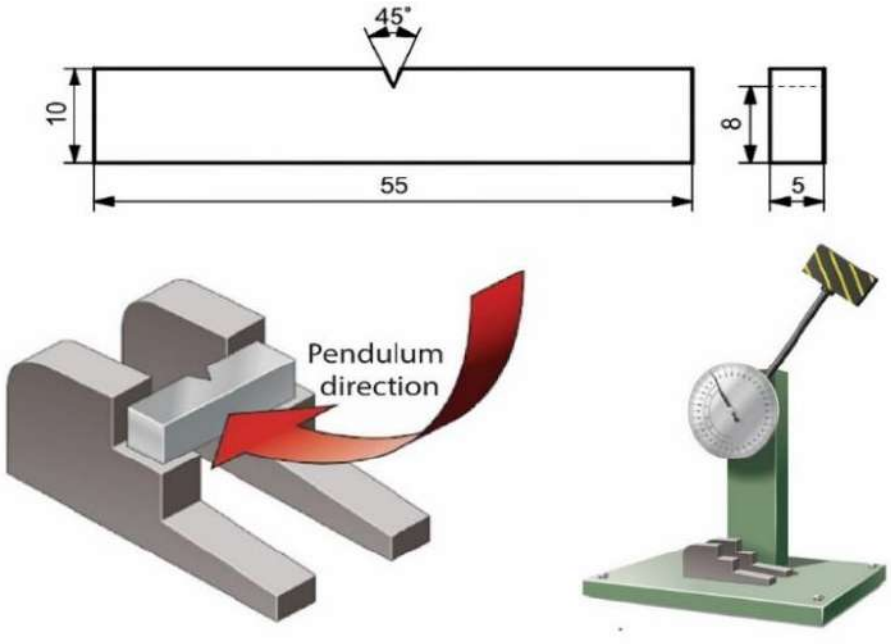

Figure 2.18- Charpy test presentation, (A) is the test sample design according to ASTM D6110, (B) is the virtual presentation of the machine and its function

To ensure the reproducibility and statistical significance of the results, three (3) specimens were tested, and the data were analyzed using appropriate statistical methods. This rigorous experimental approach provides a quantitative assessment of the impact toughness of bio-composites, facilitating a correlation with their overall mechanical properties.

The resilience (Kcv) of the bio-composites was determined using the following equation (**2.7**):

$$K_{cv} = \frac{U}{S} \quad \textbf{(2.7)}$$

With:

Kcv: is the material's resilience (in J/cm²),

U: is the energy absorbed during rupture (in joules),

S: is the cross-sectional area of the sample at the notch (in cm²).

This equation allows for a precise evaluation of the material's ability to withstand impact forces, contributing to the optimization of bio-composites for applications requiring enhanced toughness and durability.

2.7.7 Fatigue test

The fatigue test is a mechanical evaluation method used to analyze the behavior of materials under cyclic loading conditions**.** This test assesses the fatigue resistance of a material, which refers to its ability to withstand repeated stresses over time without experiencing structural failure. Unlike static mechanical tests, fatigue tests focus on progressive material degradation, where cracks may initiate and propagate, eventually leading to failure**,** even when the applied stresses remain below the material's elastic limit [146]**.**

In addition, fatigue testing is crucial for predicting the service life of components subjected to cyclic stresses, ensuring the structural integrity and durability of mechanical systems. The results from fatigue tests are typically represented as S-N curves (Stress vs. Number of cycles), which illustrate the relationship between the applied stress amplitude and the fatigue life of the material. These curves provide key insight**s** for engineers and material scientists in designing safe and long-lasting structures subjected to repeated mechanical loads [147].

2.7.7.1 Wöhler (S-N) curve:

The Wöhler curve, commonly referred to as the S-N curve (Stress vs. Number of cycles), is a fundamental tool in fatigue analysis. This curve characterizes the fatigue performance of a material by correlating the applied stress amplitude (σ) with the corresponding number of cycles to failure (N). To construct an S-N curve, fatigue tests are conducted at different stress levels, and the corresponding fatigue life (N) is recorded. The results are typically plotted on a semi-logarithmic or logarithmic scale, where the stress amplitude (σ) is represented on the y-axis and the number of cycles to failure (N) on the x-axis [147, 148]. This graphical representation allows for:

- Determining the fatigue life of a material under varying stress conditions.
- Identifying the fatigue limit (σf), which is the stress level below which the material can theoretically endure an infinite number of cycles without failure (for certain metals and composites).
- Understanding the influence of stress amplitude on failure behavior, which is essential for designing high-performance bio-composites.

The S-N curve serves as a predictive tool in structural applications, aiding in the optimization of materials and components exposed to dynamic loading conditions. **Figure 2.19** illustrates a representative Wöhler curve, highlighting the typical fatigue behavior of bio-composites under cyclic stress.

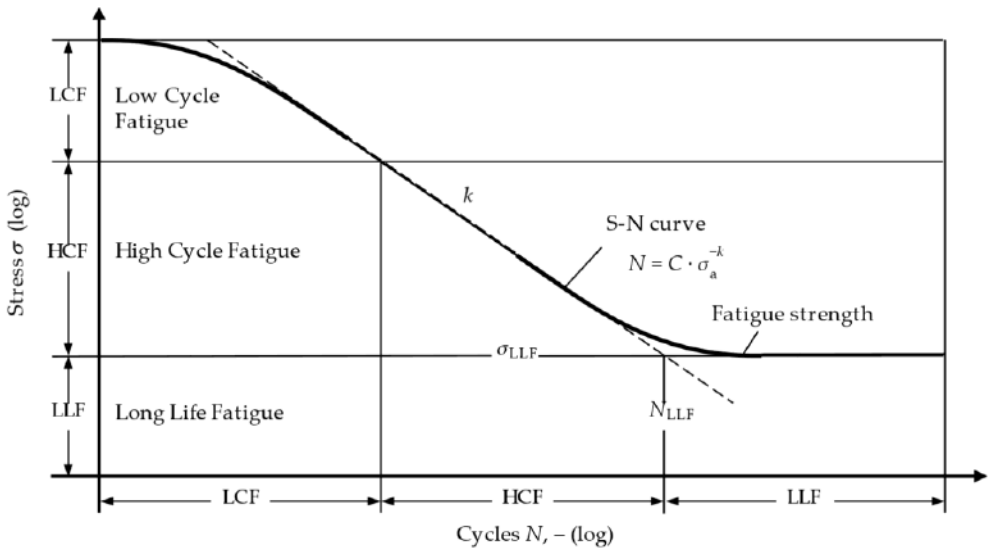

Figure 2.19- SN curve [149].

2.7.7.2 Experimental methodology

The fatigue performance of 3D-printed samples manufactured using Fused Deposition Modeling (FDM) technology was evaluated experimentally. The study focused on two types of materials: pure PLA composites and PLA reinforced with natural yucca fiber powder bio-composites, as illustrated in **Figure 2.20**.

The fatigue test was conducted using an MTS SILENTFLO 515 universal testing machine, equipped with a 100 kN load cell and an MPE (Machine Performance Evaluation) system to ensure accurate force application and measurement. The testing procedure was designed in accordance with ASTM D7791, a standard dedicated to fatigue characterization under tension-compression loading conditions.

The experimental setup involved:

- Loading conditions: The samples were subjected to a tension-compression stress regime.
- Stress ratio (R): A ratio of R = 0.1 was selected, indicating that the minimum stress applied was 10% of the maximum stress during the loading cycle.
- Waveform and frequency: A sinusoidal wave was applied at a frequency of 2 Hz, simulating cyclic loading under real-world conditions.
- Stress levels: The fatigue tests were performed at three different load levels, corresponding to 70%, 40%, and 20% of the ultimate tensile strength (UTS) of each material.

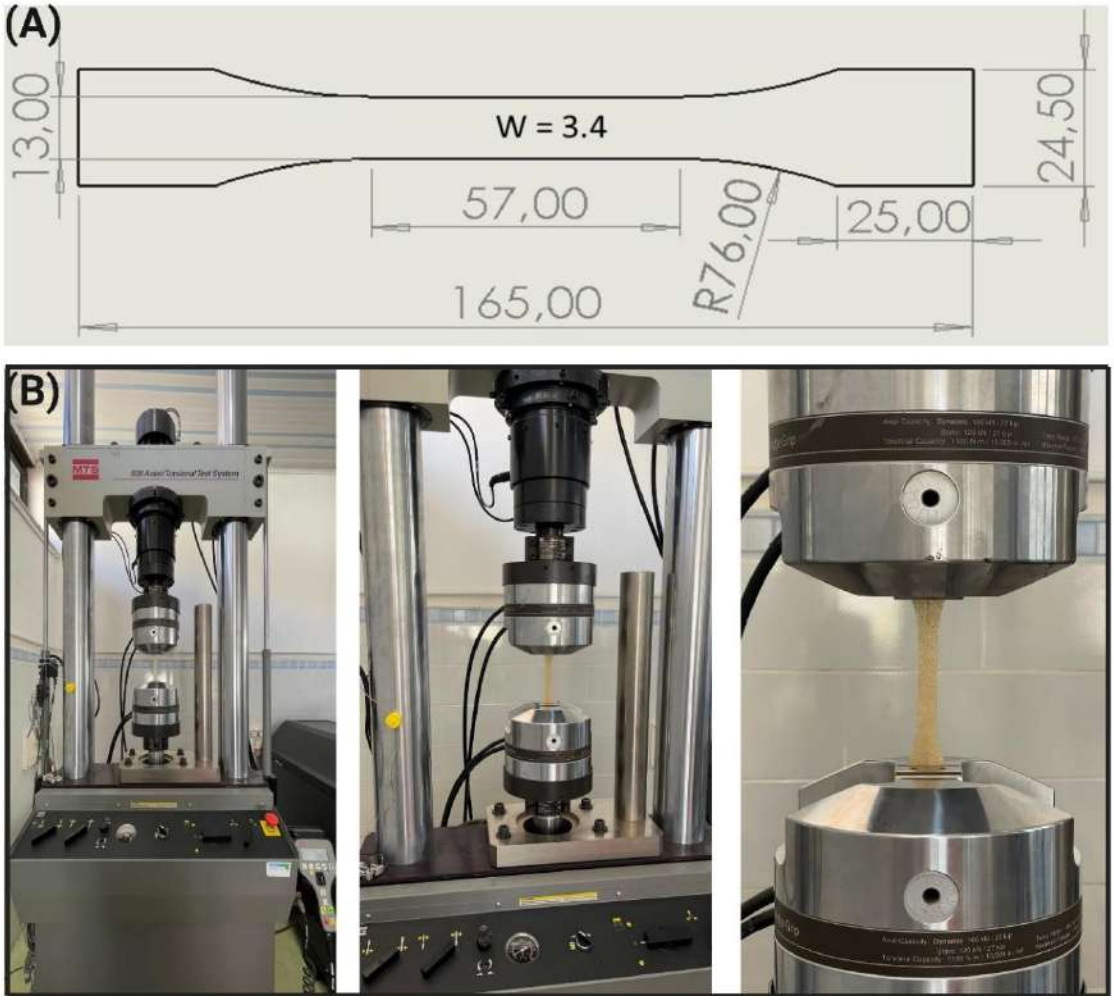

Figure 2.20 - Fatigue test on 3D printed bio-composites, (A) is the test sample design, (B) is the position of the samples in the machine.

This experimental approach aimed to assess fatigue failure mechanisms**,** which predominantly result from repeated cyclic loading at stress amplitudes below the ultimate static strength of the material. The collected data contributes to understanding the long-term durability and mechanical reliability of bio-composites reinforced with natural yucca fiber powder.

The input parameters required for the fatigue test software were determined using mathematical equations, which are presented in the following equations (**2.8** to **2.12**).

$$F = \sigma_u \times A \quad (2.8)$$

$$F_{max} = r \times F \quad (2.9)$$

$$F_{min} = R \times F_{max} \quad (2.10)$$

$$F_A = (F_{max} - F_{min})/2 \quad (2.11)$$

$$F_M = (F_{max} + F_{min})/2 \quad (2.12)$$

In this analysis, F represents the applied force based on the ultimate tensile strength (σ_u), where A denotes the sample's cross-sectional area. The parameter r corresponds to the level of ultimate tensile strength (20%, 40%, and 70%). The maximum and minimum forces applied to the sample during the fatigue test are denoted as Fmax and Fmin, respectively. The fatigue stress ratio is defined as R = (Fmin/Fmax) = 0.1. Additionally, FA represents the force amplitude, while FM corresponds to the mean force, which characterizes the average force value during cyclic loading.

2.8 Surface modification

The surface modification of natural fibers is a crucial strategy for enhancing their properties and broadening their applications across various industries, including composites, textiles, and biomedical materials. By altering the surface characteristics of fibers, it is possible to optimize their performance and compatibility with different matrices.

2.8.1 Objectives of Surface Modification

In this part of study, the surface modification of yucca fibers was pursued to achieve several key improvements:

- Enhancement of mechanical properties: Increasing tensile strength, stiffness, and toughness, thereby making the fibers suitable for applications requiring high mechanical performance.
- Improvement of fiber-matrix adhesion: Strengthening the interfacial bonding between fibers and polymer matrices in bio-composites, leading to improved mechanical properties.
- Reduction of hydrophilicity: Minimizing water absorption, which enhances moisture resistance and long-term durability of the materials.

These objectives are achieved by various surface modification methods, including chemical treatments, which modify the surface chemistry of fibers to improve their performance in specific applications.

2.8.2 Surface Modification Techniques

With goal of meet these objectives, various surface modification methods have been explored, with a focus on chemical treatments that alter the surface chemistry of fibers. These modifications are tailored to enhance fiber-matrix compatibility, improve mechanical reinforcement, and optimize performance in specific applications. The following sections will discuss the chemical treatment approaches used in this study and their effects on the properties of yucca fibers.

2.8.2.1 Experimental methods

In this study, a comprehensive experimental protocol was implemented to enhance the surface properties of yucca fibers, aiming to optimize their integration into a polymer matrix. The process consists of three key steps: (i) a mild pre-treatment with a sodium carbonate solution, (ii) chemical modification using NaOH and sulfuric acid solutions, and (iii) a neutralization phase with sodium bicarbonate, followed by multiple rinses and prolonged drying. This rigorous methodology ensures the removal of unwanted components, leading to fibers with improved surface characteristics, enhanced polymer compatibility, and superior mechanical performance in the final composites.

2.8.2.2 Pre-treatment step

The initial pre-treatment with sodium carbonate serves as a mild approach to eliminate surface impurities such as pectins and waxes, thereby improving the fibers' affinity with the polymer matrix. A 4% (w/v) Na_2CO_3 solution was prepared by dissolving the powder in distilled water. The yucca fibers were immersed in this solution for 60 minutes under moderate agitation. Unlike sodium hydroxide (NaOH), sodium carbonate induces a less aggressive alkaline hydrolysis of non-cellulosic components, following the reaction:

$$Fiber_{COOR} + Na_2CO_3 \rightarrow Fiber_{COO^-Na^+} + CO_2 \uparrow + H_2O$$

This reaction generates mild effervescence due to CO_2 release while preserving the cellulose structure. The fibers were then rinsed with distilled water until a neutral pH (~7) was reached and subsequently air-dried for 24 hours. This step partially removes surface contaminants and exposes hydroxyl groups (-OH), making the fibers more receptive to subsequent chemical modifications.

2.8.2.3 Alkaline treatment

Sodium hydroxide treatment is a critical step in enhancing the surface properties of yucca fibers to improve their adhesion with the polymer matrix. The primary objective was to remove non-cellulosic components such as lignin, pectins, and waxes while increasing fiber surface roughness. NaOH solutions at concentrations of 3% and 8% (w/v) were prepared by dissolving NaOH pellets in distilled water. The yucca fibers were immersed in the alkaline solution at 25°C for 120 minutes under gentle agitation. The hydrolysis reaction facilitates the breakdown of ester bonds and the removal of impurities via saponification, as represented by the equation:

$$Fiber\_OH\ (lignin) + NaOH\ \rightarrow Fiber - O^{-} + Na^{+} + H_2O + Soluble\ compounds.$$

After treatment, the yucca fibers were rinsed with distilled water until neutral pH was achieved and subsequently air-dried for seven days before analysis and composite integration.

2.8.2.4 Sulfuric acid treatment

Sulfuric acid treatment was employed to modify the crystalline structure of yucca fibers, enhancing their mechanical performance. Two different dilute H_2SO_4 solutions (1% and 4% v/v) were prepared by gradually adding concentrated sulfuric acid to distilled water, ensuring strict adherence to safety precautions due to the exothermic nature of the reaction. The yucca fibers were immersed in the acid solution at room temperature (25°C) for 30 minutes. Following the treatment, extensive rinsing with distilled water was performed to remove acid residues before proceeding to the neutralization step, preventing any potential residual acid-induced degradation.

2.8.2.5 neutralization stage

Following sulfuric acid treatment, a neutralization stage is essential to eliminate residual acidity and stabilize the fibers. Sodium bicarbonate ($NaHCO_3$), a weak and non-toxic base, was employed for this purpose. The acid-treated fibers were initially rinsed with distilled water to remove free acid, then immersed in a 1.5% (w/v) $NaHCO_3$ solution for 45 minutes under gentle agitation. The effervescence observed during this step, resulting from CO_2 release, confirmed the progress of the neutralization reaction. The pH was monitored using pH indicator paper or a pH meter until neutralization was achieved (pH ≈ 7). Finally, the fibers underwent three additional rinsing cycles with distilled water to remove residual salts (such as sodium sulfate) before being air-dried for seven days.

This final step ensures the complete elimination of acidic residues, thereby preventing any further fiber degradation and enhancing their compatibility with the polymer matrix.

2.9 Conclusion

This chapter has systematically outlined the key stages involved in the preparation and characterization of natural fibers for their integration into biocomposites. The methodologies employed for the extraction of yucca fibers were examined, encompassing mechanical, chemical, and biological approaches aimed at isolating the fibers from the plant's leaves. Comprehensive characterization techniques were then applied to evaluate their mechanical, chemical, and morphological properties, providing critical insights into their potential applications.

Furthermore, chemical treatments, specifically with sodium hydroxide (NaOH) and sulfuric acid (H_2SO_4), were employed to enhance the structural and interfacial properties of the fibers. These treatments facilitated the removal of non-cellulosic components and induced modifications in the crystalline structure, ultimately improving fiber-matrix adhesion and optimizing the mechanical performance of the resulting biocomposites.

Additionally, the fabrication processes of biocomposites via 3D printing and casting were detailed, along with the analytical techniques used to assess their structural integrity and overall performance. The methodologies presented in this chapter establish a robust foundation for the subsequent discussions, which will focus on the in-depth analysis of natural yucca fibers and their role in biocomposite development.

Chapiter 3: Impact of extraction methodology on natural fiber properties

3.1 Introduction:

The evaluation of extraction methods for natural fibers is crucial for their integration into high-performance composite materials, as their physico-chemical, morphological, and mechanical properties are directly influenced by the processes used. The selection of an appropriate extraction method is therefore essential to optimize their performance and ensure their effective incorporation into composite applications. This chapter aims to analyze the impact of different extraction techniques on the properties of natural fibers through a series of experimental investigations.

In this work, various characterizations were conducted to assess the modifications induced by different extraction methods. The chemical composition of the fibers was analyzed by quantifying the cellulose, hemicellulose, and lignin fractions, which directly affect their reactivity and mechanical performance. Fourier-transform infrared spectroscopy (FTIR) was employed to identify changes in functional groups, while X-ray diffraction (XRD) was used to examine variations in crystallinity. In addition, scanning electron microscopy (SEM) provided detailed insights into fiber morphology, including surface structure and integrity after extraction. Finally, tensile tests were performed to evaluate the mechanical strength and elastic modulus of the fibers as a function of the applied extraction methods.

In addition to the extraction processes, this chapter also presents the results of chemical treatments applied to yucca fibers. These treatments aim to modify fiber composition and structure to improve their mechanical properties. The effects of different chemical agents on fiber morphology, crystallinity, and mechanical performance are analyzed to provide a comprehensive understanding of their impact on fiber functionality.

Through this in-depth analysis, this chapter highlights the effects of extraction methods and chemical treatment process on the properties of natural yucca fibers. The findings contribute to a deeper understanding of the transformations induced by different extraction techniques, ultimately aiding in process optimization to enhance fiber performance for advanced composite material applications.

3.2 Methodology of natural fiber extraction

Yucca fibers have been successfully extracted from their leaves using various methods, each influencing their structural and physico-chemical properties. Unlike other types of natural fibers, yucca fibers can be obtained through several distinct techniques, including mechanical, traditional, water retting, and chemical extraction. The choice of extraction method plays a decisive role in determining fiber

morphology, chemical composition, and mechanical performance, making it a critical step in optimizing fibers for different applications.

Characterization of the extracted yucca fibers reveals variations in diameter, texture, and structural integrity. Microscopic analysis highlights differences in yucca fiber fibrillation and surface roughness depending on the applied process. Additionally, the color of the fibers varies according to the extraction method, serving as an indicator of the extent of non-cellulosic component removal.

3.3 Challenge in fiber extraction

The extraction of natural fibers is a crucial process that directly impacts their physico-chemical and mechanical properties. However, each extraction method presents specific challenges that can affect fiber quality and their suitability for composite applications. The main difficulties encountered in water retting, traditional, mechanical, and chemical methods for yucca fiber extraction are discussed in next sections.

3.3.1 Water retting

Water retting of yucca fibers poses several challenges due to the specific characteristics of this fiber and the process conditions. One major issue is the extended retting time, which can reach up to 40 days due to the high lignin and pectin content in yucca, delaying the degradation of non-fibrous components and prolonging the extraction process. An insufficient retting period results in incomplete fiber separation, whereas excessive retting can weaken the fibers, reducing their mechanical strength. Additionally, this method generates significant water pollution, as organic compounds such as phenols and nitrogenous substances are released, potentially causing eutrophication and necessitating effluent treatment before disposal.

From a health perspective, stagnant water in retting tanks fosters bacterial, fungal, and pathogenic microorganism growth, exposing operators to skin and respiratory infections, particularly in hot and humid environments. Furthermore, the variability of natural conditions (temperature, water pH, microbial activity) complicates process reproducibility, thereby influencing the final fiber properties [150].

3.3.2 Traditional method

Traditional yucca fiber extraction, though faster than water retting, has several limitations that impact process efficiency and fiber quality. First, this method involved manual fiber separation through peeling, leading to significant material loss and inconsistent yield due to the rigid and highly cohesive nature of yucca fiber bundles. The labor-intensive nature of this method exposed operators to

considerable physical fatigue, requiring sustained effort and precision to minimize fiber damage while ensuring efficient extraction [151].

Another major limitation was the heterogeneity of the fibers obtained. The absence of stringent control over extraction conditions results in variations in fiber diameter, length, and structural integrity, affecting their performance in composite applications. Additionally, the incomplete removal of non-cellulosic components such as lignin, pectin, and hemicellulose can hinder fiber-matrix adhesion in bio-composites, limiting improvements in mechanical properties. Despite its simplicity and accessibility, this method remains restrictive and requires optimization to enhance fiber quality while reducing manual labor intensity.

3.3.3 Mechanical method

Mechanical extraction of yucca fibers presented several challenges related to process efficiency and technical constraints. This method necessitates specialized machinery, requiring a relatively high initial investment and substantial energy consumption. Furthermore, accessibility to such equipment can be a limiting factor.

Another challenge was fiber loss during the extraction process. Due to the mechanical stresses applied, some fibers may be damaged or partially torn, reducing overall yield and impacting fiber quality. Additionally, mechanical methods do not completely remove non-cellulosic components, which may affect the physico-chemical and mechanical properties of the extracted fibers. Lastly, operating the machinery requires technical expertise, which may pose difficulties for operators. Improper handling can lead to inefficient extraction or fiber damage, compromising their structural integrity [152].

3.3.4 Chemical methods

Chemical extraction relies on alkaline solutions to selectively dissolve non-cellulosic components, yielding high-purity yucca fibers. However, this method presents several significant challenges. Optimizing extraction parameters, such as chemical concentration and temperature, is essential to prevent excessive fiber degradation. Over-aggressive chemical treatment can alter cellulose structure, shorten fiber length, and weaken internal fiber architecture, ultimately compromising mechanical properties. Additionally, handling chemical agents generates toxic effluents that require appropriate treatment to minimize environmental impact and health risks for operators. Proper waste management strategies must be implemented to mitigate these challenges, making chemical extraction a complex yet effective method for obtaining high-quality yucca fibers.

3.4 Extracted natural fiber characterization

3.4.1 Visual inspection

Visual inspection of yucca fibers extracted by different methods reveals significant differences in appearance, color, and texture, directly influenced by extraction conditions and interactions between the fiber and mechanical, biological, or chemical agents. **Figure 3.1** illustrates the difference between yucca fibers from the same plant but extracted by different methods.

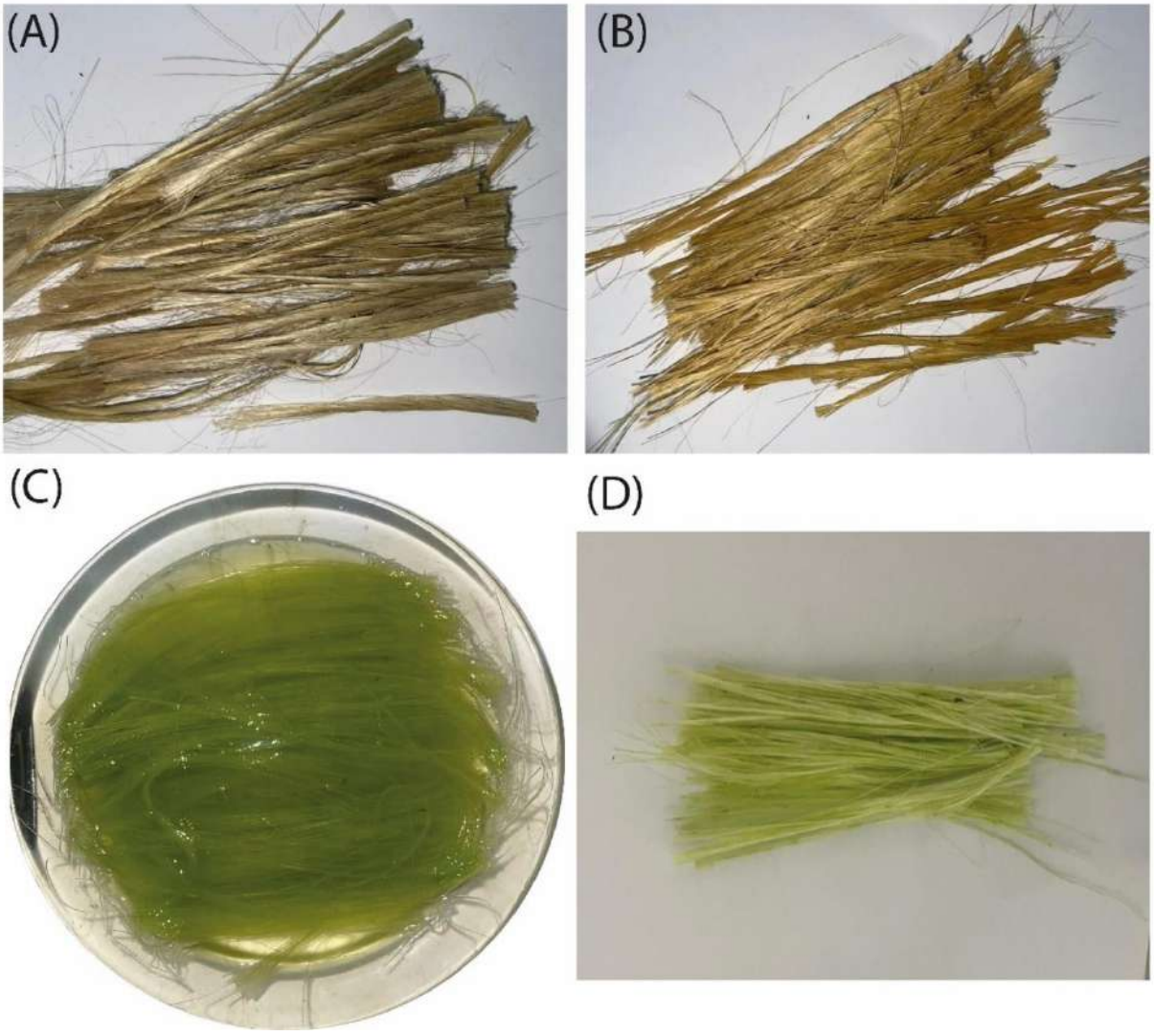

Figure 3.1 – Extracted Algerian yucca fibers via different techniques. (A) Yucca fibers extracted via water retting. (B) Yucca fibers extracted by traditional method. (C) Yucca fibers extracted using mechanical method. (D) Yucca fibers extracted via chemical method.

As a result, yucca fibers obtained by water retting have a white color and a smooth surface, as shown in **Figure 3.1(A)**. This appearance results from the progressive degradation of the non-cellulosic components under the action of the microorganisms present in the water, allowing a natural separation of the fibers. However, this high degree of smoothness can affect adhesion to polymer matrices in composite applications [153]. In addition, yucca fibers extracted using the traditional method were characterized by a yellowish hue and a rough texture **Figure 3.1(B)**. Due to the partial removal of non-fibrous elements, these fibers were often drier, which can influence their flexibility and suitability for impregnation in composites. In contrast, **Figure 3.1(C)** presents the fibers obtained from mechanical extraction retained a green color and have a moderately smooth surface. Although this method allows rapid separation of the fibers, it occasionally results in the retention of plant residues, which can affect the homogeneity of the fibers and require post-extraction treatment to improve their purity [154]. Finally, the yucca fibers obtained through chemical processes were also green in color (**Figure 3.1(D)**), although they do not have a very smooth texture and are very tightly woven together. This characteristic is due to the interactions between the fibers under the effect of the chemical agents, which can cause agglomeration and make it more difficult to separate the fibers individually. These observations confirm that the extraction method has a significant influence on the physical properties of yucca fibers, which will have a direct impact on their behavior in final applications.

3.4.2 Chemical composition results

Analyzing the chemical composition of natural fibers is an essential step in understanding the influence of extraction methods on their structure and physicochemical properties. Cellulose, hemicellulose, and lignin are the main constituents of plant fibers, each playing a fundamental role in their mechanical characteristics and their interaction with a polymer matrix in composites. Cellulose is responsible for the mechanical strength and rigidity of fibers, while hemicellulose and lignin contribute to structural cohesion and flexibility. Any change in their proportions induced by extraction can therefore have an impact on fiber performance in bio-composite applications. **Table 3.1** presents the proportions of these components for different extraction methods, highlighting the effect of the methods applied on the natural yucca fiber.

Table 3.1 – Chemical composition of yucca fibers extracted using different methods.

Fiber extraction method	% cellulose	%lignin	%hemicellulose
Water retting	79.25	10.45	10.75
Traditional	78.06	10.95	10.70
Chemical - 3%NaOH	73.95	07.20	13.55
Chemical - 5%NaOH	73.95	10.10	11.84
Chemical - 10%NaOH	65.85	10.33	12.97

Analysis of the results revealed significant variations in the chemical composition of the natural yucca fibers, depending on the extraction method used. As a result, yucca fibers obtained by water retting had the highest cellulose content (79.25%), followed by those obtained by traditional extraction (78.06%). These values indicated that these two methods significantly preserve the cellulose, limiting its degradation during extraction [47]. On the other hand, extraction by chemical methods leads to a progressive reduction in cellulose content, reaching 73.95% for fibers extracted with 3% and 5% NaOH, and a more marked reduction to 65.85% for the 10% NaOH extraction method. This reduction is explained by the partial dissolution of the amorphous cellulose under the effect of the alkaline solution, a phenomenon that is particularly accentuated at high NaOH concentrations, thus favoring the depolymerization of the cellulose and the extraction of certain parietal constituents [155].

In another context, in terms of lignin content, the yucca fibers obtained by water retting and the traditional method retained relatively similar values at 10.45% and 10.95%, respectively. This similarity suggests that these two methods do not significantly alter the lignin, thus preserving the original structure of the cell walls. However, using an alkaline extraction method with 3% NaOH resulted in a significant reduction in lignin content at a value of 7.20%, reflecting the effect of lignin dissolution in an alkaline environment. A slight increase in lignin content was nevertheless observed for the yucca fibers extracted with 5% and 10% NaOH at 10.10% and 10.33%, respectively, which could be attributed to precipitation of residual lignin or to interactions with the remaining parietal components under more intense alkaline conditions [156].

The trend in hemicellulose content was similar to that of the other constituents. Yucca fibers obtained by water retting exhibited the hemicellulose content at 10.75%, closely followed by those extracted by traditional methods at 10.70%, which confirmed that these two methods limit the

degradation of this polymer. Alkaline extraction methods, on the other hand, led to a progressive reduction in hemicellulose. Yucca fibers extracted with 5% NaOH show the lowest hemicellulose content at around 11.84%, while those extracted with 3% and 10% NaOH show slightly higher values at 13.55% and 12.97%, respectively. This variation suggests that the alkaline extraction method with 5% NaOH favors greater extraction of hemicelluloses, while increasing the concentration to 10% could lead to relative stabilization due to cross-linking phenomena or the formation of residues that are difficult to extract.

These results demonstrate the significant influence of extraction methodology on the chemical composition of natural yucca fibers. Water retting and traditional extraction better preserve the original fiber structure, while alkaline extraction considerably alters the proportion of constituents, leading to a progressive dissolution of cellulose and hemicelluloses and a selective reduction of lignin. These changes can have a direct impact on the physicochemical and mechanical properties of the yucca fibers, influencing their performance as reinforcements for bio-composite applications.

Table 3.2- Comparison of yucca fiber chemical composition with other natural fibers [53].

Natural fibers	Cellulose [%]	Hemicellulose [%]	Lignin [%]
Sisal	67.00	10.00	08.00
Jute	71.50	13.60	13.00
Cotton	82.70	05.70	-
Bamboo	43.00	31.00	31.00
Rice	41.00	33.00	09.00

A comparative analysis with other natural fibers situates yucca fibers extracted by water retting within a broader context (**Table 3.2**). With a cellulose content of approximately 80.25%, these fibers exhibit a composition comparable to that of cotton fibers (82.70%) and superior to jute (71.50%) and sisal fibers (67.00%). This high cellulose content imparts greater rigidity and mechanical strength, attributes that are crucial for composite applications. Furthermore, the lignin content of yucca fibers (10.45%) is lower than that of jute (13.60%) and bamboo fibers (31.00%), which could enhance interfacial adhesion with polymer matrices. In contrast, their hemicellulose content (10.75%) remains relatively similar to that of sisal fibers (10.00%) and jute fibers (13.60%), suggesting a balance between flexibility and processability for chemical treatments. These findings underscore the potential of yucca fibers for bio-composite applications, particularly in contexts requiring an optimal balance between stiffness and flexibility.

3.4.3 Fiber structural analysis

Figure 3.2 presents the FTIR spectra of yucca fibers extracted using different methods, highlighting the chemical transformations induced by these processes. This spectroscopic analysis enables the identification of key functional groups and the assessment of the impact of extraction techniques, including water retting, traditional extraction, mechanical extraction, and chemical extraction using 3%, 5%, and 10% NaOH solutions. The spectral changes observed indicate structural alterations in the lignocellulosic components, particularly cellulose, hemicellulose, and lignin.

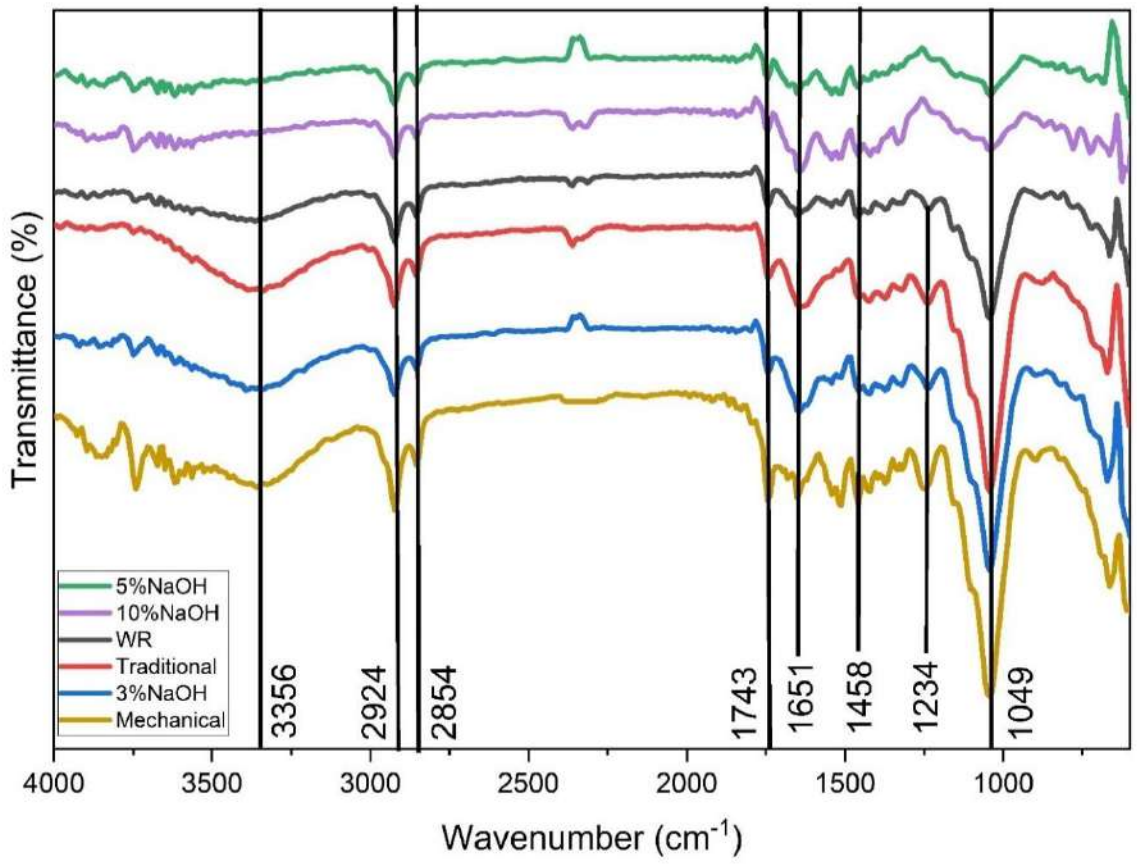

Figure 3.2 - Functional group spectra for each yucca fiber category in this study.

As a result, the FTIR spectra revealed a broad band at 3356 cm^{-1}, characteristic of the stretching vibrations of hydroxyl (-OH) groups in cellulose and lignin, indicating the presence of hydrogen bonds [157]. Additionally, peaks at 2924 cm^{-1} and 2854 cm^{-1} corresponded to asymmetric and symmetric stretching vibrations of C-H bonds, typical of alkyl chains in biopolymers [158]. A band at 1743 cm^{-1}, attributed to the stretching vibrations of C=O bonds in hemicellulose esters and aldehydes, was visible in

untreated yucca fibers in all categories, suggesting a progressive degradation of these constituents [159]. In addition, the peak at 1651 cm^{-1} was associated with C=C bond vibrations in the aromatic structures of lignin, as reported in the literature [160]. The band at 1458 cm^{-1}, corresponded to the deformation vibrations of C-H bonds in lignin and amorphous cellulose, was also affected by chemical treatments. Lastly, a strong band at 1049 cm^{-1} is linked to the stretching vibrations of C-O-C bonds in cellulose, indicating an increase in cellulose content after extraction. However, the impact of chemical extraction was particularly noticeable in fibers treated with 5% and 10% NaOH, where the band at 1234 cm^{-1}, associated with C-O vibrations of hemicellulose and lignin ester bonds, weakens or disappears, confirming their partial removal [161].

These findings demonstrate that water retting, traditional, and mechanical methods effectively purify fibers by eliminating non-cellulosic compounds (lignin, hemicellulose, pectin), whereas chemical treatments, despite being more intensive, preserve a more complex chemical structure with residual lignin and polysaccharides. This structural variation directly influences the mechanical properties of yucca fibers and their adhesion within composite matrices.

3.4.4 Fiber crystallinity analysis

X-ray diffraction (XRD) analysis is used to assess the crystalline structure of yucca fibers as a function of different extraction methods. This technique reveals the proportion of crystalline and amorphous cellulose, which directly impacts the mechanical and physicochemical properties of the fibers. The results are illustrated in **Figure 3.3** and summarized in **Table 3.3**.

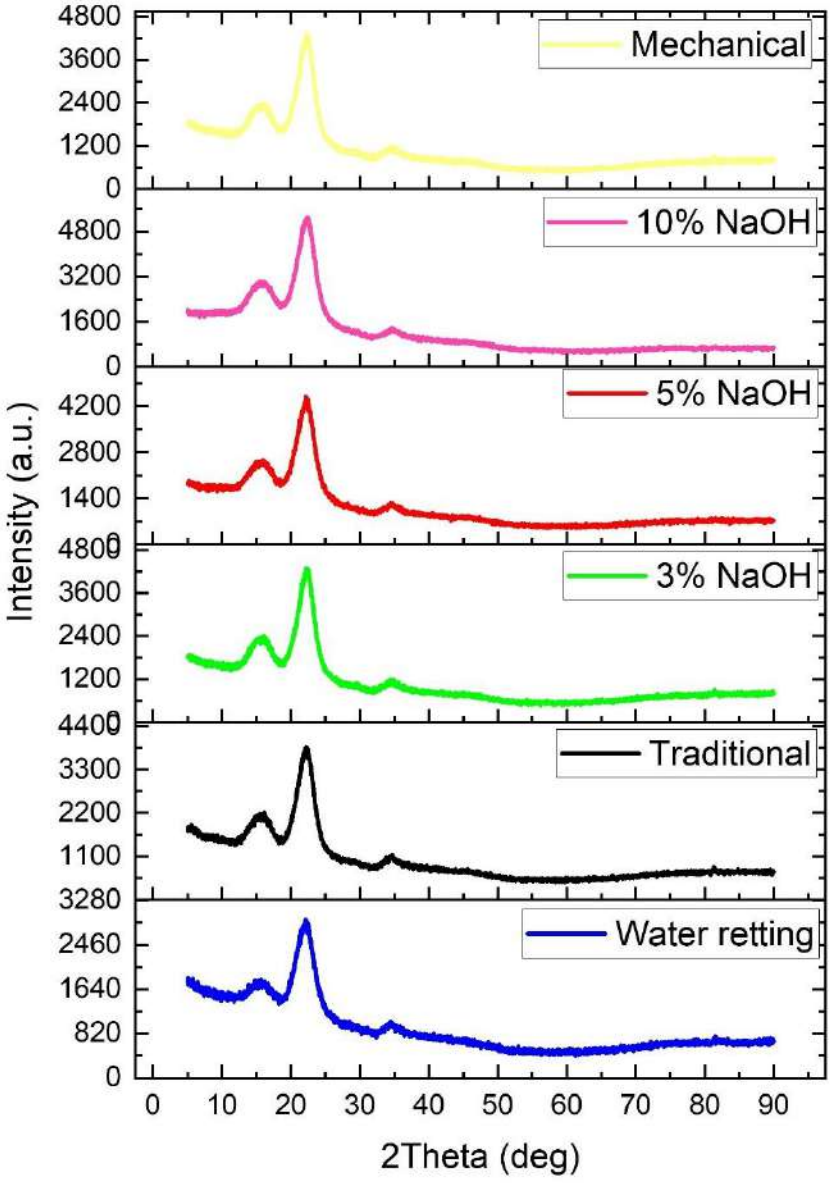

Figure 3.3- XRD spectra of yucca fibers

The spectra exhibited characteristic peaks of cellulose I, with a dominant crystalline peak around 22.3° - 22.49°, attributed to the (002) plane of crystalline cellulose, and two amorphous peaks between 15.86° - 16.14° and 34.62° - 34.81°, corresponding to disordered cellulose regions. The crystallinity index (CI) varies depending on the extraction method, reflecting changes in the structural organization of the fibers [159].

Table 3.3- Extraction method impact on the crystallinity proprieties of yucca fibers.

Extraction method	Amorphous peaks (°)	Crystalline Peaks (°)	Crystallinity Index (%)	Crystallite Size(nm)
Water retting	15.86 / 34.72	22.40	70.77	1.73
Traditional	15.86 / 34.62	22.30	68.12	1.75
Chemical 3%NaOH	16.05 / 34.81	22.30	65.90	1.76
Chemical 5%NaOH	15.86 / 34.81	22.49	65.57	1.84
Chemical 10%NaOH	16.14 / 34.81	22.30	61.75	2.04
Mechanical	15.62 / 34.40	22.10	46.40	1.30

Yucca fibers extracted through water retting showed the highest CI at 70.77%, indicating optimal preservation of their natural crystalline structure. Meanwhile, traditional and chemical extractions using 3% and 5% NaOH resulted in a slight decrease in CI to 68.12%, 65.90%, and 65.57%, respectively, suggesting partial dissolution of amorphous fractions while maintaining a significant proportion of crystalline cellulose. In contrast, chemical extraction with 10% NaOH and mechanical extraction further reduced the CI to 61.75% and 46.40%, respectively, indicating greater disruption of the crystalline structure due to cellulose degradation and the removal of non-cellulosic components [162].

Furthermore, crystallite size ranged from 1.30 to 2.04 nm, as shown in **Table 3.3**, with a gradual increase as NaOH concentration increased. This trend suggested a reorganization of crystalline domains following the partial removal of hemicelluloses and lignin. The largest crystallite size (2.04 nm) was observed in fibers treated with 10% NaOH, which may influence fiber-matrix interactions in bio-composites [162]. However, the primary diffraction peaks were located at 2θ = 15.8°, 16.1°, 22.3°, and 34.8°, corresponding to the Miller indices (101), (10$\bar{1}$), (002), and (040), respectively, as reported in the literature [159].

3.4.5 Morphological inspection

The morphological characterization of natural yucca fibers was performed using scanning electron microscopy (SEM). High-resolution SEM imaging enabled a detailed assessment of the modifications induced by each extraction process, allowing for the identification of structural alterations and surface irregularities. **Figure 3.4** presents the SEM images of yucca fibers obtained through various extraction techniques.

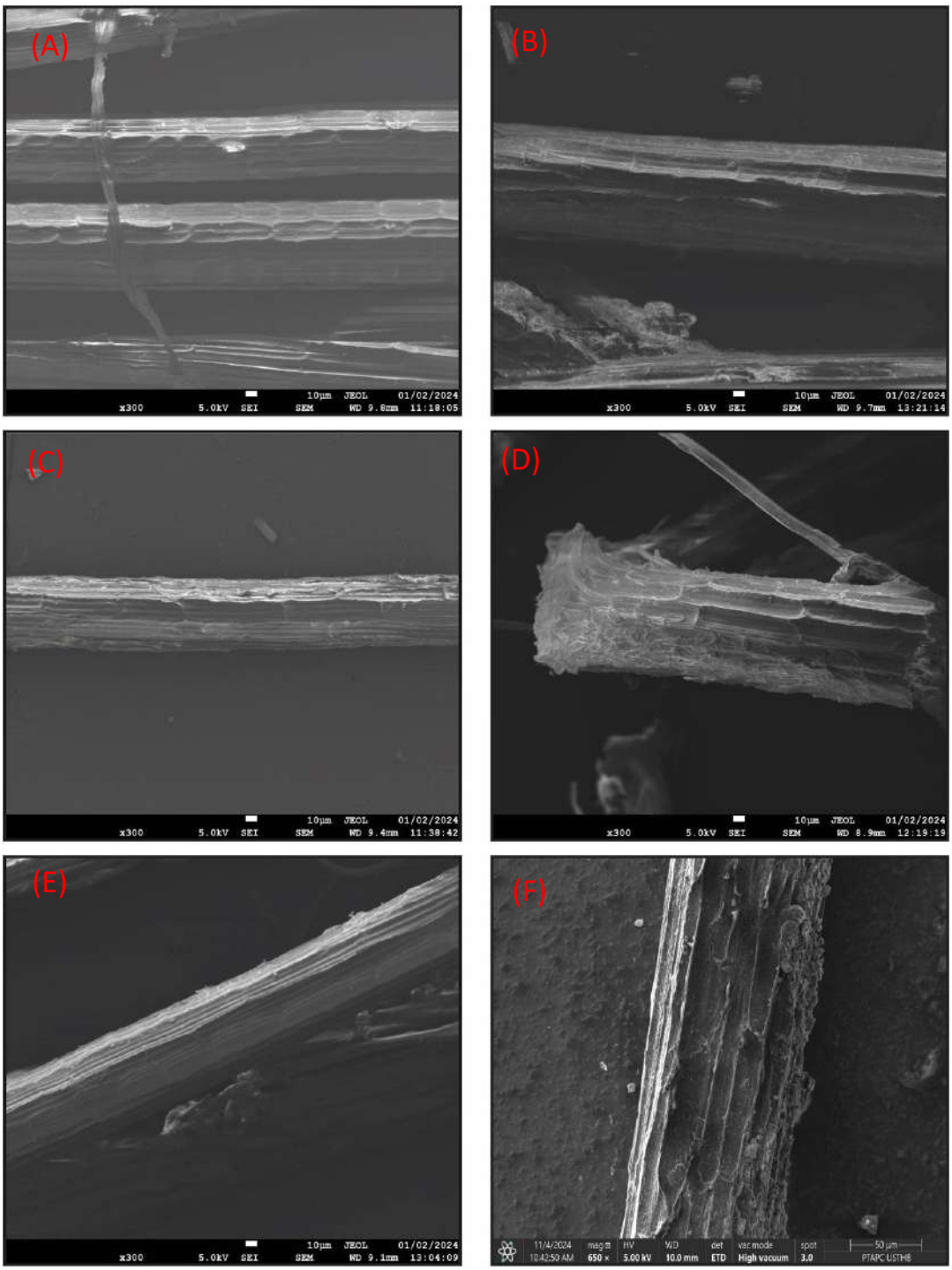

Figure 3.4 – SEM images of yucca fibers extracted via several extraction methods. (A) Surface condition of yucca extracted via water retting. (B) Surface condition of yucca extracted via traditional method. (C), (D) and (E) Represent the surface condition of yucca extracted via chemical method using NaOH at 3%, 5%, and 10% respectively. (F) Surface condition of yucca extracted via mechanical method.

The fiber extracted using the traditional method, as shown in **Figure 3.4(B)**, exhibited a rough surface morphology with numerous impurities. This is due to the fact that, while the extraction process removes organic matter, it does not entirely eliminate surface residues. Additionally, a significant number of voids can be observed within these fibers, likely resulting from the elevated temperature used during extraction. Higher temperatures facilitate the degradation of non-fibrous components, such as lignin and pectin, thereby easing fiber extraction. However, excessive heat exposure may also lead to thermal degradation, promoting void formation and potentially compromising the fiber's mechanical properties. In contrast, fibers extracted through chemical treatment, represented in **Figures 3.4(C)–(E**) with varying NaOH concentrations (3%, 5%, and 10%), exhibit improved surface quality, particularly at higher concentrations. SEM images confirm that increased NaOH concentrations enhance the removal of impurities, as the chemical solution aggressively dissolves non-cellulosic substances. Compared to the other extraction methods, the water retting process yields fibers with a notably smoother surface, as shown in **Figure 3.4(A).** This improvement is likely attributed to microbial activity inherent in the retting process, which effectively degrades and removes organic matter from the fiber's outer layers [163].

Furthermore, as illustrated in **Figure 3.4(E),** yucca fibers extracted mechanically display relatively smooth surfaces coated with non-cellulosic substances, including lignin and natural waxes. These components form a protective layer around the cellulose fibrils, potentially limiting interfacial adhesion with the polymer matrix. It is important to note that the presence of residual impurities can hinder fiber-matrix adhesion, negatively affecting the overall performance of bio-composites. Moreover, SEM analysis reveals that all categories of yucca fibers contain varying degrees of porosity, a critical parameter influencing adhesion between the fiber and the polymer matrix in bio-composites [164].

3.4.6 Physical proprieties

The physical properties of natural fibers, particularly fiber diameter, play a crucial role in determining their mechanical behavior and interactions with the matrix in composite materials. A reduction in fiber diameter is generally associated with improved mechanical performance, attributed to better alignment of cellulose chains and a decrease in structural defects. **Table 3.4** presents the average diameters of yucca fibers extracted using different methods, as measured from SEM images.

Table 3.4- Diameter proprieties of yucca fiber extracted via different methods.

Fiber extraction method	Average diameter (µm)
Water retting	74.89
Traditional	89.34
Chemical 3% NaOH	62.33
Chemical 5% NaOH	90.34
Chemical 10% NaOH	84.67
Mechanical	110.38

Yucca fibers obtained through mechanical extraction exhibited the highest diameter (110.38 µm), likely due to the absence of fibrillar bundle separation treatments. This method preserved the raw fiber structure without effectively removing non-cellulosic components such as lignin and hemicellulose. Water retting led to a significant reduction in fiber diameter (74.89 µm), indicating partial dissolution of amorphous constituents and gradual separation of fiber bundles. The traditional extraction method resulted in a slightly larger diameter (89.34 µm), suggesting less effective microfibril separation compared to water retting. In addition, chemical extraction with NaOH produced varying fiber diameters depending on the concentration used. At 3% NaOH, the diameter reached its lowest value (62.33 µm), indicating partial removal of non-cellulosic compounds and enhanced fibrillation. However, at 5% NaOH, the fiber diameter increased to 90.34 µm, potentially due to microfibril reorganization and swelling induced by water uptake. At 10% NaOH, the diameter decreased again to 84.67 µm, although it remained larger than that of fibers treated with 3% NaOH. This trend may result from excessive dissolution of amorphous cellulose, leading to microfibril embrittlement and limiting further diameter reduction [165, 166].

The results in this section highlighted the importance of the choice of extraction method in modifying the fibrillar structure. A reduction in diameter may be beneficial for improving fiber-matrix adhesion in composites, while excessive disorganization may alter the overall mechanical properties. Optimization of chemical extraction conditions and retting parameters could result in fibers with diameters suitable for specific bio-composite applications.

3.4.7 Single fiber tensile test

The mechanical properties of natural fibers are essential for assessing their suitability as reinforcements in composite materials. Tensile tests on individual yucca fibers provide insights into their

tensile strength, Young's modulus, and elongation at break. **Table 3.5** present the mechanical results for fibers extracted via different methods.

Table 3.5- Tensile proprieties of yucca fiber extracted via different methods.

Fiber extraction	Tensile strength [MPa]		Young's modulus [GPa]		Elongation [%]	
	Mean	SD	Mean	SD	Mean	SD
Mechanical	257.79	41.00	06.59	1.56	4.05	0.94
Water retting	467.00	98.50	14.00	2.86	4.3	0.81
Traditional	444.74	133.54	13.57	5.00	3.27	1.15
Chemical 3%NaOH	456.23	97.93	16.78	3.94	2.64	0.66
Chemical 5%NaOH	443.40	130.57	16.84	3.25	2.79	0.76
Chemical 10%NaOH	235.83	42.02	9.51	2.75	2.61	0.61

The extraction method significantly influenced the mechanical performance of yucca fibers. Water-retted fibers exhibited the highest tensile strength (467 MPa) and a Young's modulus of 14 GPa, suggesting that this method effectively preserved the fiber's structural integrity. Similarly, fibers extracted using the traditional method showed comparable mechanical properties, with a tensile strength of 444.74 MPa and a Young's modulus of 13.57 GPa, indicating minimal structural disruption. In addition, chemical extraction with NaOH produced varying effects on mechanical properties. At 3% NaOH, Young's modulus increased to 16.78 GPa, indicating fiber stiffening, while tensile strength decreased slightly to 456.23 MPa compared to water-retted fibers. A similar trend was observed at 5% NaOH, where Young's modulus reached 16.84 GPa, but tensile strength declined to 443.40 MPa. However, at 10% NaOH, a substantial deterioration in mechanical properties was observed, with tensile strength dropping to 235.83 MPa and Young's modulus to 9.51 GPa. This decline can be attributed to excessive cellulose dissolution, leading to fiber degradation [70, 163].

Elongation at break also follows a similar pattern. Water-retted and traditionally extracted fibers exhibited higher elongation values (4.3% and 3.27%, respectively), indicating retained flexibility. In

contrast, chemically extracted fibers, particularly those exposed to 3% and 5% NaOH, show reduced elongation (2.64% and 2.79%, respectively), suggesting increased brittleness due to the removal of amorphous components. In another context, mechanically extracted fibers exhibit the lowest tensile strength (257.79 MPa) and Young's modulus (6.59 GPa), likely due to structural damage and disruption of inter-fibrillar bonds during processing. The relatively high elongation at break (4.05%) suggests a higher retention of amorphous components, contributing to residual flexibility [167].

These results indicate that water retting is an effective method for preserving the mechanical integrity of yucca fibers, whereas chemical extraction, despite improving stiffness, may negatively impact strength depending on the concentration used.

Table 3.6 – Comparison of tensile propriety of yucca fiber extracted via water retting to other natural fibers reported in the literature [168].

Natural fibers	Tensile strength [MPa]	Youngs modulus [GPa]
Jute	393	13
Abaca	717	18.6
Sisal	350	12.8
Kenaf	282.60	7.13
Bamboo	500	27
Coconut	140	3

A comparison with other natural fibers reveals that yucca fibers extracted via water retting exhibit remarkable mechanical properties **(Table 3.6).** With a tensile strength of 467 MPa, they outperform and rival widely used fibers such as jute (~393 MPa) and sisal (~350 MPa), while demonstrating an intermediate Young's modulus (14 GPa). This combination of high strength and moderate stiffness highlights their potential as reinforcement materials in bio-composites for applications requiring both durability and flexibility.

3.5 Fiber surface treatment impact

The surface treatment of natural fibers plays a crucial role in enhancing their physicochemical and mechanical properties, particularly when used as reinforcements in bio-composites. Untreated fibers often contain non-cellulosic components such as lignin, hemicelluloses, and pectin, which can negatively

affect their compatibility with polymer matrices. Chemical treatments, including alkaline and acidic modifications, aim to selectively remove these components, improving fiber crystallinity, surface roughness, and interfacial adhesion with the matrix. However, the effectiveness of these treatments depends on various factors, such as concentration, treatment duration, and temperature, which influence the final structure and mechanical behavior of the fibers. This section explores the effects of chemical treatments on the structure and mechanical properties of yucca fibers, emphasizing their impact on composite performance [169, 170].

3.5.1 Chemical treatment of yucca fibers

Mechanically extracted yucca fibers were subjected to two types of chemical treatments, alkaline and acidic, aimed at enhancing their structural and chemical properties while optimizing their mechanical performance. Each treatment method selectively modifies the fiber composition by removing non-cellulosic constituents while preserving the mechanical integrity of the fibers. **Figure 3.5** illustrates the different stages of the chemical treatment process applied to yucca fibers.

Alkaline treatment primarily targeted the dissolution of hemicelluloses and partial modification of lignin, resulting in significant structural changes within the fibers. The hydrolysis of non-cellulosic components increases the relative cellulose crystallinity, leading to enhanced stiffness and mechanical strength. Additionally, the treatment roughens the fiber surface, which improves interfacial adhesion within bio-composite materials.

Conversely, acid treatment followed a distinct mechanism, inducing controlled hydrolysis that can lead to a reduction in fiber diameter while simultaneously altering its crystalline structure. However, if not carefully controlled, this process can cause excessive degradation, leading to embrittlement of the fibers. This underscores the necessity of optimizing treatment conditions to achieve a balance between eliminating non-fibrous components and maintaining the mechanical integrity of the fibers.

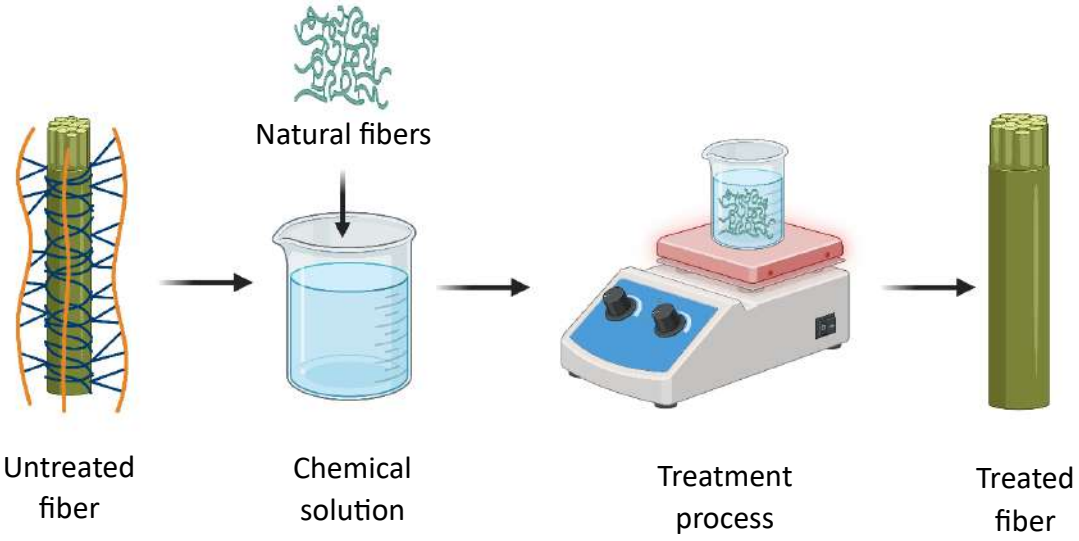

Figure 3.5- The chemical treatments process used in this part of this study.

3.5.2 Challenge

The chemical treatment of yucca fibers, whether alkaline or acidic, presented significant challenges that directly impact the final quality of the fibers. In this study, mechanically extracted yucca fibers were subjected to both types of treatment at varying concentrations to enhance their properties. However, several obstacles were encountered throughout the process.

Alkaline treatment, primarily aimed at removing lignin and hemicellulose, led to substantial fiber swelling, which increased fragility, particularly at higher concentrations or prolonged exposure times. Furthermore, post-treatment fibers exhibited a strong tendency to agglomerate, complicating their separation and potentially hindering uniform dispersion within a polymer matrix. The rinsing phase proved to be a critical step, necessitating a considerable amount of water to remove residual alkali and prevent further chemical degradation.

Acid treatment, although effective in selectively modifying the fibrillar structure, resulted in excessive fiber thinning, reducing their diameter and increasing brittleness. Partial dissolution of amorphous cellulose was also observed, which could compromise the mechanical performance of the fibers in certain applications. Additionally, the generation of acidic effluents required neutralization before disposal, adding an environmental challenge to the process. Similar to alkaline treatment, acid-treated

fibers exhibited strong aggregation tendencies, making their handling and incorporation into composite formulations more complex.

These observations underscore the necessity of optimizing chemical treatment parameters to enhance fiber properties while preserving their structural integrity. Achieving a balance between the removal of non-cellulosic components and the retention of fiber architecture is crucial to producing high-quality fibers suitable for advanced composite applications.

3.6 Treated natural fiber characterization

3.6.1 Visual inspection

The visual inspection of yucca fibers after chemical treatment reveals significant changes in color and texture, highlighting the effects of alkaline and acidic processes on fiber structure, as shown in **Figure 3.6.**

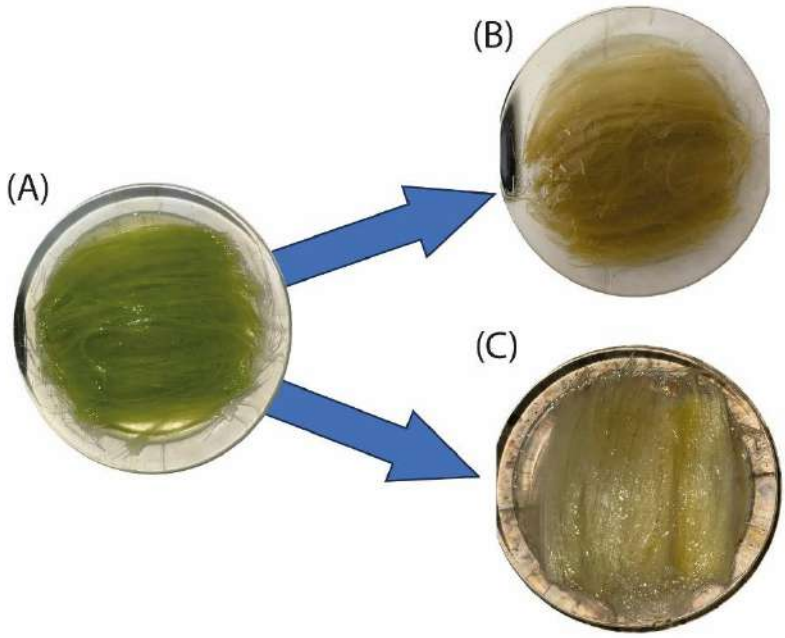

Figure 3.6 – Yucca fiber visual inspection after and before the chemical treatment. (A) Mechanically extracted yucca fibers before chemical treatment. (B) and (C) Mechanically extracted yucca fibers after NaOH and sulfuric acid chemical treatments respectively.

Before treatment, mechanically extracted yucca fibers exhibited a characteristic green hue due to the presence of non-cellulosic residues such as pectin and natural impurities (**Figure 3.6(A)**). Following chemical treatment, a gradual color change from green to yellow was observed. This transformation was

more pronounced in alkaline-treated fibers, where the removal of non-cellulosic components was more effective. Under the action of NaOH, fibers progressively loosed their green tint and acquire a pale yellow to light brown color, depending on treatment conditions (**Figure 3.6(B)**). Acid treatment also results in fiber lightening, but to a lesser extent than alkaline treatment, as illustrated in **Figure 3.6 (C).** This color change was primarily attributed to the partial degradation of lignin and hemicelluloses caused by acid hydrolysis, exposing a purer cellulose structure. However, acid-treated fibers retain a rougher texture and a certain degree of cohesion between individual fibers [153].

3.6.2 Functional group characterization

Fourier Transform Infrared (FTIR) spectroscopy was conducted to identify the functional groups present in yucca fibers before and after chemical treatment. The obtained spectra reveal characteristic peaks, indicating the chemical modifications induced by alkaline and acid treatments, as shown in **Figure 3.7**.

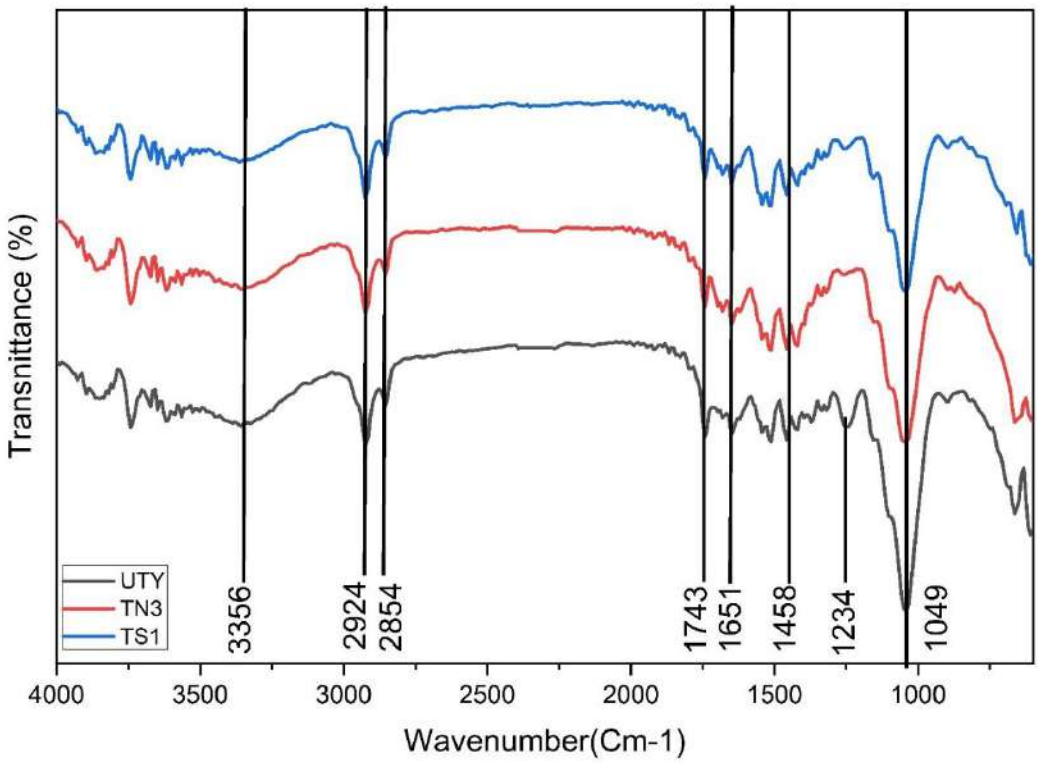

Figure 3.7- FTIR spectra on untreated and chemically treated yucca fibers.

Firstly, the peak at 3356 cm^{-1}, attributed to O–H bond stretching vibrations, signified the presence of hydroxyl groups in cellulose and lignin. After treatment, its intensity decreased, suggesting modifications in hydrogen bonding, likely due to the reduction of hemicelluloses and lignin. Similarly, the peaks at 2924 cm^{-1} and 2854 cm^{-1}, corresponded to the asymmetric and symmetric stretching vibrations of aliphatic C–H bonds, exhibit a slight reduction in intensity, indicating alterations in lipid compounds and the partial removal of natural surface waxes [171].

The peak at 1743 cm^{-1}, associated with C=O stretching vibrations in esters and aldehydes found in lignin and hemicelluloses, was significantly attenuated after chemical treatment. This reduction confirms the progressive degradation of these components, supporting the removal of non-cellulosic fractions. Additionally, in the 1700–1600 cm^{-1} region, the peak at 1651 cm^{-1}, corresponded to C=C vibrations of lignin's aromatic structures, exhibits considerable intensity variation, reflecting changes in the lignocellulosic network [172].

The peak at 1458 cm^{-1}, attributed to C–H deformation vibrations in methyl and methylene groups within cellulose and lignin, also undergoes intensity modifications, indicating structural rearrangements due to the partial dissolution of non-cellulosic components. Meanwhile, the peak at 1049 cm^{-1}, characteristic of C–O–C vibrations in glycosidic linkages, remained present, signifying the preservation of the cellulose backbone [173].

Interestingly, the peak at 1234 cm^{-1}, corresponded to C–O bonds in esters and C–O–C linkages in hemicelluloses, was only detected in untreated yucca fiber. Its disappearance post-treatment confirms the near-complete elimination of hemicelluloses and other non-cellulosic components [174].

These findings highlight the effects of chemical treatments on the fiber's chemical composition. The disappearance of specific peaks and intensity variations suggest progressive fiber purification, yielding a cellulose-rich structure. This transformation is crucial for improving fiber-matrix interactions in bio-composites, ultimately enhancing their performance.

3.6.3 Crystallinity characterization

X-ray diffraction (XRD) analysis was employed to assess the impact of chemical treatments on the crystalline structure of yucca fibers. Three samples were analyzed: UYF (untreated yucca fibers), TN3 (fibers treated with 3% NaOH), and TS1 (fibers treated with 1% sulfuric acid). The results are presented in **Figure 3.8** and summarized in **Table 3.7**.

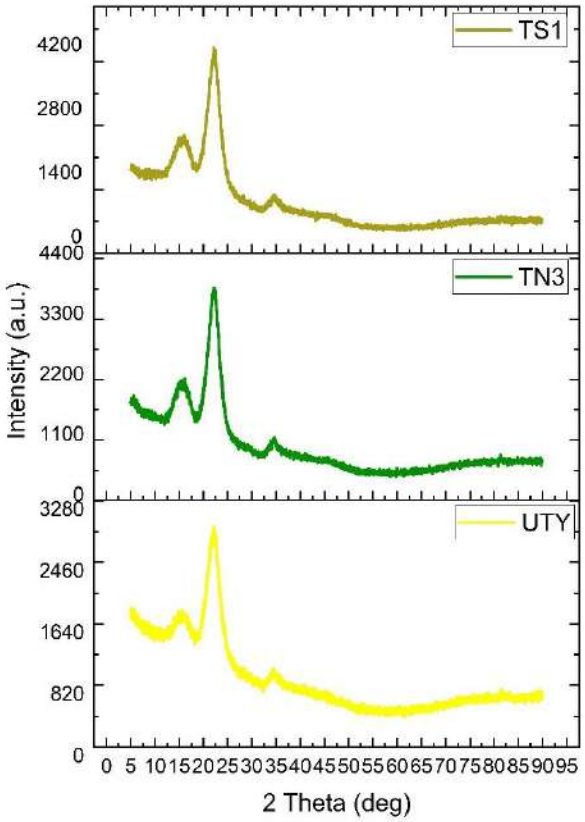

Figure 3.8- XRD results of untreated and chemically treated yucca fibers.

The XRD spectra revealed amorphous peaks at approximately 15.62° and 34.40° for untreated fibers (UYF), which shift slightly after chemical treatment to 16.14° and 34.71° for TN3 and 15.96° and 34.47° for TS1. This minor shift suggests partial modification of the fiber's disordered structure. Regarding the crystalline phase, a characteristic cellulose peak is observed at 22.10° for UYF, with slight shifts to 22.18° for TN3 and 22.16° for TS1, indicating a reorganization of cellulose chains that may influence fiber mechanical properties.

Table 3.7- Crystallinity results of untreated and chemically treated yucca fibers.

Sample nature	Amorphous peaks [°]	Crystalline Peaks [°]	Crystallinity index [%]	Crystallite size [nm]
UTY	15.62/34.40	22.10	46.40	1.30
TN3	16.14/34.71	22.18	52.43	1.47
TS1	15.96/34.47	22.16	54.97	1.34

Examination of the crystallinity index (CI) revealed a significant increase after chemical treatment accordingly to the **Table 3.7**. Untreated fibers (UYF) exhibit a CI of 46.40%, which increased to 52.43% for TN3 and 54.97% for TS1. This trend indicated the progressive removal of amorphous components, particularly hemicelluloses and a portion of lignin, facilitating better cellulose microfibril organization. However, the difference between alkaline and acid treatments suggests distinct effects on fiber structure. Acid treatment, though more aggressive, yielded a slightly higher CI, likely due to selective hydrolysis of amorphous fractions [175].

Additionally, an increase in crystallite size (CS) is observed following alkaline treatment, from 1.30 nm in untreated fibers to 1.47 nm in TN3, indicating an enlargement of crystalline domains. This phenomenon is likely due to the selective dissolution of amorphous chains and structural reorganization. In contrast, acid treatment results in a crystallite size of 1.34 nm, close to that of untreated fibers, suggesting that while crystallinity increased, crystallite growth was not significantly promoted [176].

These findings align with previous studies on chemically treated natural fibers. For instance, prior research on banyan aerial root fibers reported an increase in CI after alkaline treatment, reaching approximately 52% [177]. Similarly, studies on bamboo fibers have shown an increase in crystallinity following NaOH treatment, although excessive NaOH concentrations can lead to cellulose degradation [178]. Regarding acid treatment, its efficiency varies based on process conditions. Research on flax fibers indicated that low acid concentrations enhance crystallinity, whereas higher concentrations degrade crystalline structures [179].

Overall, these results confirmed that chemical treatments significantly influence the crystalline structure of yucca fibers. Alkaline treatment is more effective in promoting crystallite growth, while acid treatment primarily enhances the crystallinity index by selectively removing amorphous components.

3.6.4 Morphological characterization

Yucca fibers chemically treated with a 3% NaOH solution and 1% sulfuric acid (H_2SO_4) solution exhibit significant morphological alterations, as observed through scanning electron microscopy (SEM) as illustrated in **Figure 3.9**. This analysis aims to evaluate the impact of chemical treatment methods on the morphology and surface characteristics of yucca fibers. SEM observations provide insights into the removal of amorphous components such as lignin and hemicellulose, as well as the structural modifications induced by these treatments.

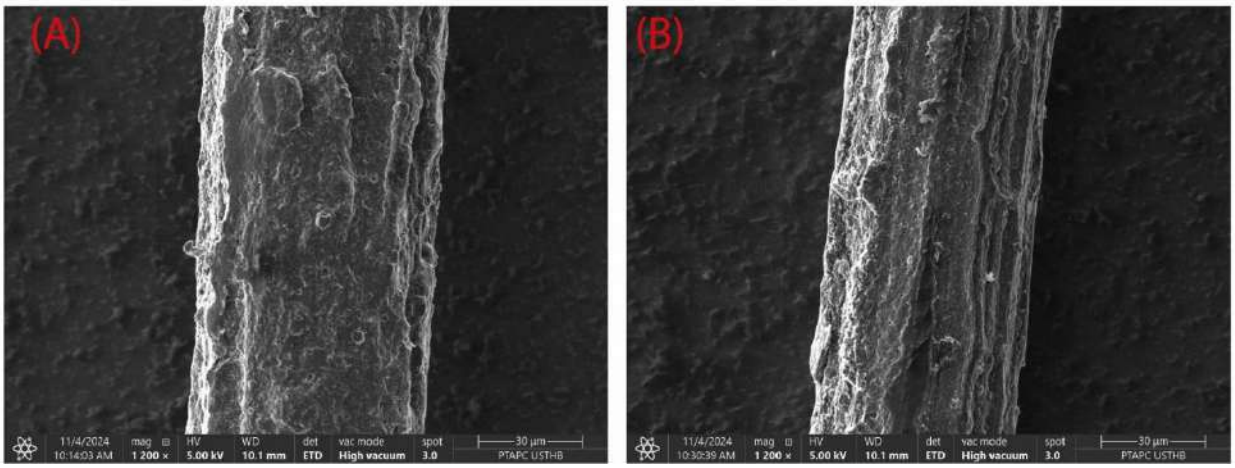

Figure 3.9- SEM images of chemically treated yucca fibers. (A) SEM image of surface condition of yucca fibers treated at 3% NaOH. (B) SEM image of surface condition of yucca fibers treated at 1% sulfuric acid.

As a result, the mechanically extracted yucca fibers treated with NaOH, as shown in **Figures 3.9(A**), exhibited a surface modification due to alkaline action, leading to the partial degradation of secondary components. Additionally, a fine residual layer is visible on both categories of fibers treated with NaOH, likely resulting from the incomplete removal of fiber constituents, particularly lignin and hemicelluloses. This residual layer may form a non-uniform film while simultaneously providing additional adhesion sites in composite applications [180].

In contrast, yucca fibers treated with sulfuric acid, presented in **Figures 3.9(B),** displayed a rougher and more eroded surface, characterized by the presence of micro-cracks. Acid treatment effectively dissolved amorphous components such as lignin and hemicelluloses, thereby exposing cellulose fibrils and increasing surface roughness. This enhanced roughness can be beneficial for fiber-matrix adhesion. However, excessive acid treatment may negatively impact the cellulose structure, increasing fiber brittleness. Notably, yucca fibers treated with 1% sulfuric acid retain a relatively intact cellulosic structure with a well-defined texture, showing no significant signs of excessive degradation. Consequently, this treatment offers a promising balance between enhancing adhesion and preserving the mechanical integrity of the fibers [181].

3.6.5 Physical proprieties

The evaluation of the physical properties of yucca fibers, particularly their average diameter, provides valuable insights into the impact of chemical treatments on their structure. The measurements

obtained from SEM images, presented in **Table 3.8**, indicate a significant reduction in fiber diameter following both alkaline and acidic treatments, reflecting notable morphological changes.

Table 3.8 – Diameter proprieties of untreated and chemically treated yucca fibers.

Samples type	Average diameter (µm)
UTY	110.38
TN3	76.55
TS1	59.63

Untreated yucca fibers (UYF) showed an average diameter of 110.38 µm, characteristic of raw fibers still surrounded by non-cellulosic compounds such as lignin, hemicellulose and natural waxes. After moderate alkaline treatment (TN3), the diameter of the treated yucca fibers decreased to 76.55 µm, a reduction of around 30.6%. This reduction was mainly attributed to the progressive elimination of amorphous components, leading to a more refined fiber that is better exposed to interactions with the matrix in a composite. In addition, acid treatment (TS1) resulted in an even greater reduction in diameter, reaching 59.63 µm, a reduction of around 45.9% compared with the raw yucca fibers. This sharp reduction can be explained by a more aggressive degradation of the non-cellulosic components, as well as by a possible partial dissolution of the amorphous cellulose, leading to greater compaction of the fibrillar structures [182].

These findings highlighted the influence of chemical treatments on fiber structuring, confirming that alkaline treatment enables effective fiber refinement while maintaining structural integrity. Conversely, acid treatment induces a more significant reduction in diameter, which may impact fiber reactivity and adhesion in composite applications. These trends align with previous studies on natural fibers, which have reported similar reductions in diameter following chemical treatments, thereby improving their compatibility with polymer matrices.

3.6.6 Mechanical proprieties

The mechanical properties of untreated and chemically treated yucca fibers were assessed through tensile testing, evaluating tensile strength, Young's modulus, and elongation at break. The results, presented in summarized in **Table 3.9**, demonstrate a substantial enhancement in mechanical performance following chemical treatments.

Table 3.9- Tensile test results of untreated and chemically treated yucca fibers.

Fiber type	Tensile strength [MPa]		Young's modulus [GPa]		Elongation [%]	
	Mean	SD	Mean	SD	Mean	SD
UYF	257.79	41.00	06.59	1.56	4.05	0.94
TN3	518.74	46.23	10.25	1.74	5.14	0.60
TS1	497.81	17.40	12.22	2.31	4.19	0.69

As a result, the untreated yucca fibers (UYF) achieved an average tensile strength of 257.79 MPa, with a Young's modulus of 6.59 GPa and an elongation at break of 4.05%. These values indicated a relatively weak structure, due to the presence of amorphous components such as lignin and hemicellulose, which limit the optimal alignment of the cellulose microfibrils. These results provided a benchmark for assessing the impact of chemical treatments on the structure and mechanical properties of yucca fibers. On the other hand, after alkaline treatment with 3% NaOH concentration (TN3), mechanical performance of yucca fibers was improved considerably. Tensile strength reached 518.74 MPa, representing an increase of 101.2% compared with the raw yucca fibers. At the same time, Young's modulus rose to 10.25 GPa, an increase of 55.6%, reflecting the increased rigidity of the fiber. Elongation at break also increased by 27% from 4.05% to 5.14%, indicating a stronger and slightly more flexible fiber. This mechanical strengthening is mainly attributed to the partial elimination of the amorphous components, allowing better organization of the fibrillar structures and greater cohesion of the cellulose chains. Moreover, yucca fibers treated with 1% Sulphuric acid (TS1) also showed a significant improvement in their mechanical properties. Tensile strength reached 497.81 MPa, an increase of 93.1% compared with untreated fibers. However, their Young's modulus is even higher, reaching 12.22 GPa, an increase of 85.4%. This increased stiffness is explained by more marked crystallization of the cellulose and a possible reorganization of intermolecular bonds. On the other hand, the elongation at break fell slightly to 4.19%, a drop of 21.2% compared with the NaOH-treated fibers, suggesting a stiffer but slightly less flexible fiber [183, 184].

Table 3.10- Comparison of yucca fiber treated at 3% NaOH to other different chemically treated natural fiber.

Natural fiber	Treatment method			Tensile strength [MPa]		Improvement rate (%)	Ref.
	Method	%	Time (h)	Untreated	Treated		
Yucca	NaOH	3	2	257.79	518.74	101.22	Present work
Sisal	NaHCO3	10	24	276.95	660.88	138.50	[185]
Kenaf	NaOH	6	4	105.06	282.75	169.20	[186]
Date palm	NaOH	6	3	233	366	57.1	[187]
Pineapple	NaOH	1	1	630	1560	147.62	[161]

The influence of chemical treatment on yucca fibers can be better understood by comparing it with other natural fibers, treated and untreated. Generally, raw fibers, such as untreated sisal, Kenaf, date palm or pineapple fibers, have a tensile strength of between 100 and 300 MPa. These values are comparable to those of untreated yucca fiber (UYF), which has a strength of 257.79 MPa. This similarity is due to the presence of lignin and hemicellulose, which confer a certain flexibility but reduce the cohesion between the cellulose microfibrils. However, after chemical treatment, most natural fibers show a significant improvement in their mechanical properties (**Table 3.10**). For example, sisal fibers treated with NaHCO3 show an increase in tensile strength of up to 660.88 MPa, with an increase of 138%. Similarly, pineapple fibers treated with NaOH show a significant increase in strength to 366 MPa, with an improvement of 57%, demonstrating improved rigidity and structural cohesion. This improvement is attributed to the partial dissolution of amorphous compounds, facilitating the alignment of cellulose chains and thus improving inter-fibrillar interactions.

Thus, chemical treatment, whether alkaline or acidic, significantly improved the mechanical properties of yucca fibers, making them comparable or even superior to other treated natural fibers. These results confirmed the interest of these fibers for structural applications in bio-composites, where optimized mechanical properties are required.

3.7 Conclusion

The findings of this chapiter highlighted the combined influence of the extraction method and chemical treatments on the physical and mechanical properties of yucca fibers. The choice of extraction method significantly impacted the fiber's initial structure, composition, and subsequent response to

chemical modifications. Additionally, chemical treatments played a crucial role in refining the fiber morphology, enhancing its mechanical performance, and tailoring its properties for bio-composite applications. Several key results are summarized in detail below:

- The yucca fiber may be extracted by several methods, including biological, mechanical and chemical techniques.
- Yucca fibers extracted through water retting maintain the highest cellulose content (80.25%), preserving a more natural and minimally degraded structure. Traditional extraction yields a slightly lower cellulose content (78.06%). In contrast, chemical extraction significantly modifies the fiber composition, with 10% NaOH reducing cellulose to 65.85% (a 17.9% decrease) due to partial degradation.
- Traditionally extracted fibers retain a rougher, more irregular surface, whereas biological extraction results in a smoother, more homogeneous surface with better exposure of the fibrils.
- Yucca fibers extracted via water retting method exhibited the highest tensile strength at 467 MPa, which is 81.2% higher than mechanically extracted fibers with 257.79 MPa. They also show a 6.2% increase in elongation at break at 4.3% compared to mechanical extraction with 4.05%, indicating better structural integrity and flexibility. This shows that this natural fiber can be effectively served as a bio-composite reinforcement.
- The biological extraction method demonstrated superior efficiency compared to the other extraction approaches, particularly in preserving the integrity of the cellulose structure within the fiber.
- Alkaline treatment (TN3) reduced the fibers diameter by 30.6%, while acid treatment (TS1) resulted in a greater reduction (45.9%), indicating a more aggressive effect.
- Alkaline treatment with 3% NaOH (TN3) significantly improves the tensile strength of yucca fibers (+101.2%) and Young's modulus (+55.6%), while treatment with 1% Sulphuric acid (TS1) confers increased stiffness (+85.4%) but slightly reduces elongation at break compared with fiber treated with NaOH.

The results emphasize the importance of selecting the appropriate extraction and chemical treatment methods to tailor fiber properties for bio-composite applications, balancing strength, flexibility, and stiffness to meet specific performance requirements.

Chapiter 4: Impact of yucca fiber extraction and chemical treatment on the structure and strength of sustainable 3D printed and molded bio-composites

4.1 Introduction:

The development of bio-composites is gaining increasing interest across various industrial sectors due to their low environmental impact, lightweight nature, and mechanical properties suitable for both structural and functional applications. Among natural fibers, those derived from yucca stand out due to their high cellulose content and potential as reinforcements in bio-based polymer matrices, particularly polylactic acid (PLA). However, the quality and performance of bio-composites are highly dependent on the fiber extraction method, which directly affects their chemical structure, morphology, and crystallinity.

Natural fibers can be extracted using various approaches, including mechanical, chemical, and biological processes. These methods influence the chemical composition, morphological structure, and crystallinity of the fibers, thereby affecting their interactions with the polymer matrix and ultimately determining the bio-composite's overall performance. Improper extraction can lead to the deterioration of mechanical properties, insufficient fiber-matrix adhesion, or premature thermal degradation of the material.

This chapter explores the influence of different yucca fiber extraction methods on the physicochemical and mechanical properties of PLA 3D-printed bio-composites, as well as on epoxy bio-composites. At the same time, a second part is presented in this chapter, focusing on the influence of the chemical treatments of yucca fiber on the mechanical properties of bio-composites. A comprehensive characterization was conducted using morphological (SEM), spectroscopic (FTIR), and thermal (TGA, DTG) analyses, along with mechanical tests (tensile, bending, compressive, Charpy impact), fatigue, and water absorption assessments. The objective is to determine the most suitable extraction method to optimize bio-composite performance by ensuring an optimal synergy between the fiber and the polymer matrix.

4.2 Development of bio-composite filament

4.2.1 Filament extrusion feasibility

The feasibility of filament extrusion is a crucial stage in the development of bio-composites, as it directly affects the printability, mechanical performance, and structural integrity of the final 3D-printed components. In this study, filaments were successfully extruded using a PLA matrix reinforced with yucca fiber powder at different concentrations (1% and 3% by weight). **Figure 4.1** presents the extruded filament spools, demonstrating good uniformity in filament diameter. However, an increase in fiber content to 3%

led to a slight rise in surface roughness, which may impact interlayer adhesion during 3D printing. These effects will be further analyzed in subsequent sections to evaluate their influence on the final properties of the printed bio-composites.

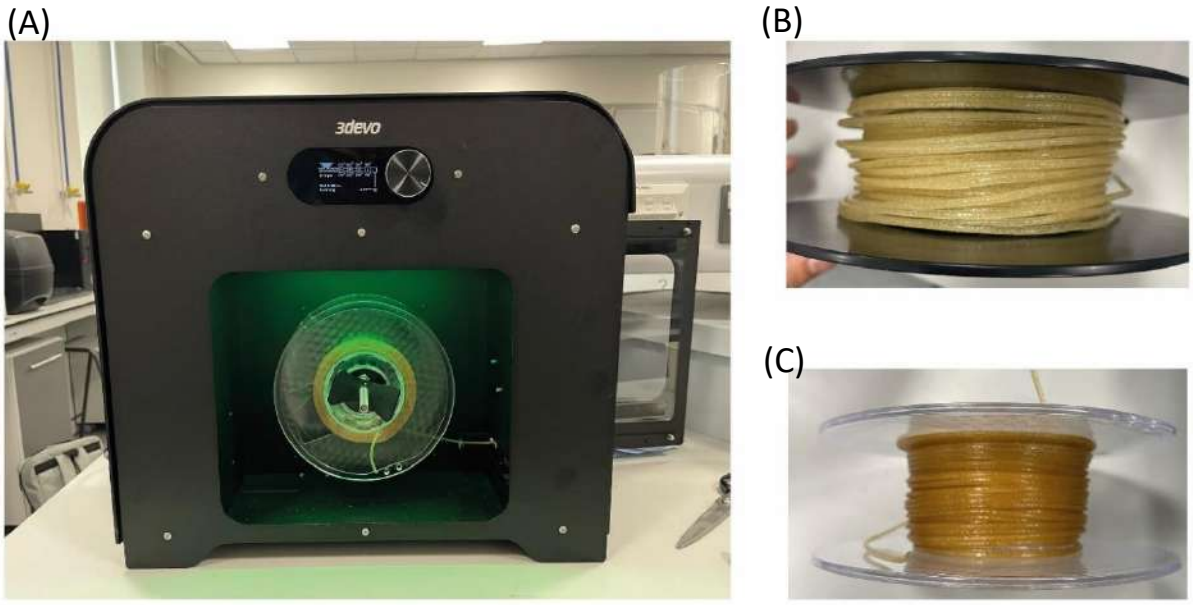

Figure 4.1- Biocomposite filament for 3D printed, (A) the 3devo machine after extrusion, (B) and (C) are the PLA/yucca fiber bio-composite filaments.

4.2.2 3D printing of bio-composites

The 3D printing of bio-composites based on PLA and yucca fibers was conducted to evaluate their suitability for additive manufacturing and to analyze the influence of fiber incorporation on printing behavior. A key objective of this study was also to investigate the impact of the fiber extraction method on print quality and the mechanical performance of the fabricated parts. The extruded filaments were used to manufacture test specimens for mechanical, thermal, and physico-chemical characterization.

Figure 4.2(A) illustrates the printing process of the bio-composite samples, demonstrating good inter-layer adhesion and satisfactory dimensional stability. Analysis of the printing behavior revealed that the bio-composites maintained adequate fluidity, enabling a homogeneous deposition of the material. However, a higher yucca fiber powder content (3%) affected the rheological properties of the filament, leading to an increase in surface roughness and slight dimensional variations in the printed parts. Additionally, the printed samples shown in **Figure 4.2(B)** exhibited uniform printing with no significant structural defects, such as cracks or delamination. The analysis suggests that the fiber extraction method

plays a crucial role in fiber dispersion and interaction with the polymer matrix, thereby influencing the final print quality.

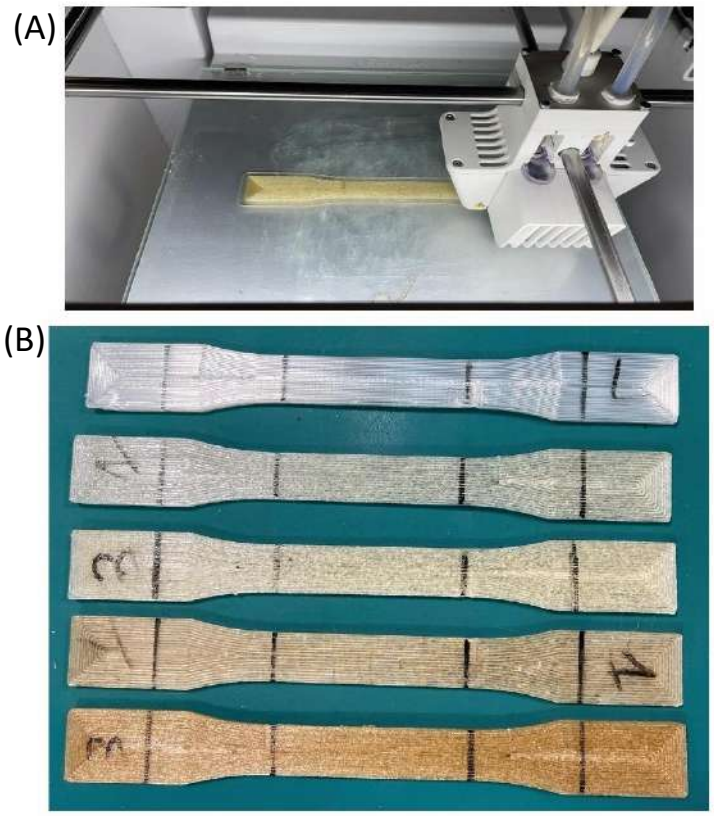

Figure 4.2- (A) The 3D printed biocomposite sample during the process. (B) The tensile test samples of all 3d printed biocomposite.

4.3 Elaboration and printing challenges

4.3.1 Filaments extrusion

Filament plays a key role in the bio-composite 3D printing process, as it directly influences extrusion quality, mechanical performance, and the dimensional stability of printed samples. The success of 3D printing largely depends on the ability to produce homogeneous filaments with suitable rheological properties and optimal compatibility between the polymer matrix and the natural fiber reinforcement. However, the fabrication of bio-composite filaments presents several technical challenges that must be addressed to ensure their effective use.

One of the primary challenges was the incorporation of natural yucca fibers into a thermoplastic matrix, which requires a prior transformation of the fibers from their original long form into a fine powder.

This grinding process is highly dependent on the inherent properties of the fiber and can be particularly complex. Additionally, fiber particle size plays a decisive role in filament quality. Oversized particles may disrupt filament continuity, leading to dimensional irregularities that can hinder the printing process. Moreover, non-uniform fiber distribution within the matrix can create local density variations, directly affecting the mechanical properties of the final bio-composite.

Furthermore, the proportion of yucca fiber powder incorporated into PLA was a critical parameter in determining the overall performance of the bio-composite. While a low fiber content may limit enhancements in mechanical properties, an excessively high fiber loading can introduce challenges such as poor interfacial adhesion between phases and uneven dispersion within the matrix. Additionally, a higher concentration of yucca fibers can significantly alter the rheological behavior of the filament, increase its viscosity and make extrusion more complex or even impractical. Achieving an optimal balance between fiber content and processability is therefore essential to ensure the successful fabrication of high-performance bio-composite filaments [188].

4.3.2 3D printing

3D printing of bio-composites represents a major advancement in the development of sustainable materials, offering the possibility to manufacture complex structures with a high degree of design flexibility. However, the integration of natural yucca fibers into a polymer matrix such as PLA introduces several technical challenges that must be addressed to ensure high-quality printing and optimal performance of the printed components.

One of the primary challenges lied in maintaining the fluidity and consistency of the extruded filament. The incorporation of yucca fiber powder altered the rheological properties of the material, affecting its flow behavior through the printing nozzle. Poor filament homogeneity can result in fluctuations in the extrusion rate, leading to surface defects and reduced interlayer adhesion. Moreover, the interfacial compatibility between the polymer matrix and the yucca powder plays a crucial role in determining the final quality of the printed samples. A high fiber content may cause nozzle clogging due to the accumulation of solid particles, hindering the flow of the molten material and potentially disrupting the printing process [189].

Another significant challenge was the dimensional stability of the printed samples. The presence of yucca fibers modified the thermal behavior of the polymer during cooling, which can induce deformations, shrinkage, or even microcracking. To mitigate these effects and ensure precise reproduction of the digital

model, optimization of key printing parameters, such as extrusion temperature, printing speed, and infill density is essential. Careful parameter tuning is necessary to balance printability, mechanical performance, and structural integrity in bio-composite 3D printing [190].

4.4 Physico-chemical characterization of bio-composites

4.4.1 Morphological inspection (SEM)

To analyze the morphological characteristics of the fracture surfaces of PLA-based bio-composites reinforced with yucca fiber powder obtained through two different extraction methods, water retting and the traditional method, Scanning Electron Microscopy (SEM) was performed. The corresponding micrographs, displayed in **Figures 4.3, 4.4** and **4.5**, provide a detailed examination of the interfacial adhesion, fiber dispersion, and fracture mechanisms, offering valuable insights into the influence of the extraction method on the structural integrity and performance of the bio-composites.

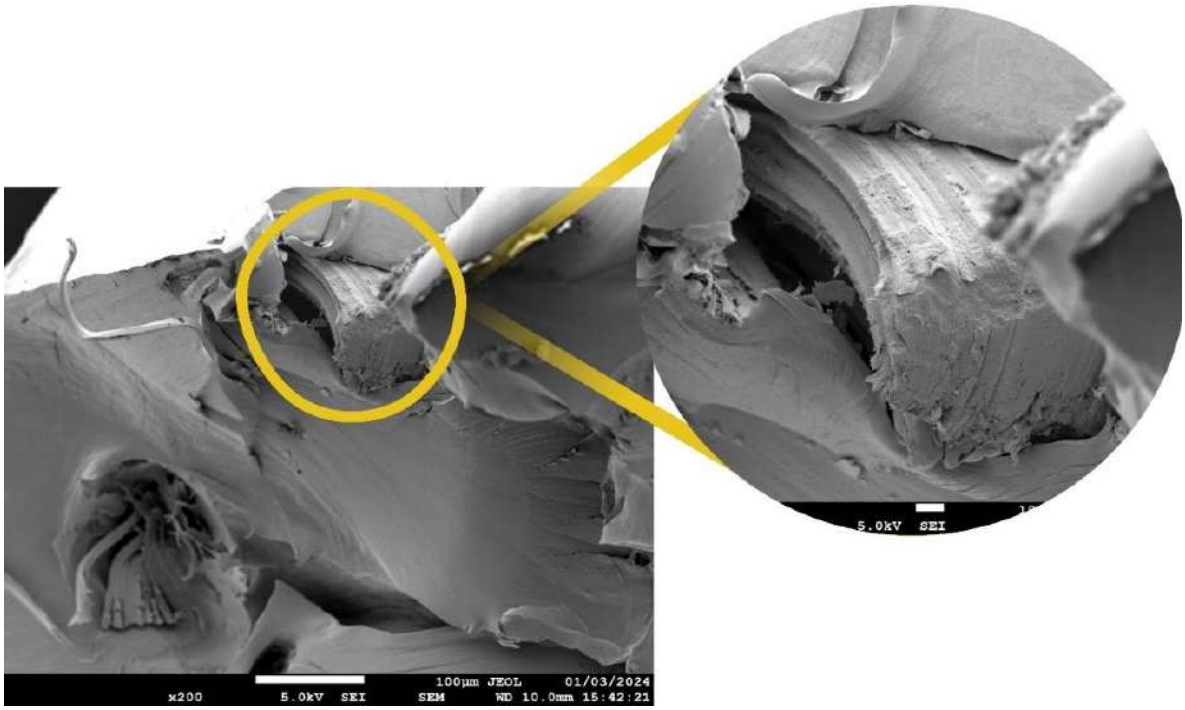

Figure 4.3- SEM image of PLA/ 1% yucca fiber powder extracted via water retting method.

As a result, **Figure 4.3** presents the microstructure of the bio-composite reinforced with 1% yucca powder extracted through the water retting method (PLA-WR1). The image highlighted a relatively homogeneous dispersion of the yucca powder within the PLA matrix. However, the presence of voids at the interface between the reinforcement (yucca powder) and the matrix (PLA) suggests weak interfacial

adhesion. This limited adhesion can be primarily attributed to the intrinsic surface characteristics of the fiber and its compatibility with the polymer matrix. As discussed in a previous section, yucca fibers obtained via water retting exhibited a smooth surface morphology with minimal porosity, which played a key factor in determining fiber-matrix interactions. The insufficient interfacial bonding observed in PLA-WR1 may negatively impact the overall performance of the bio-composite, particularly its mechanical properties, by facilitating crack propagation and reducing load transfer efficiency [164].

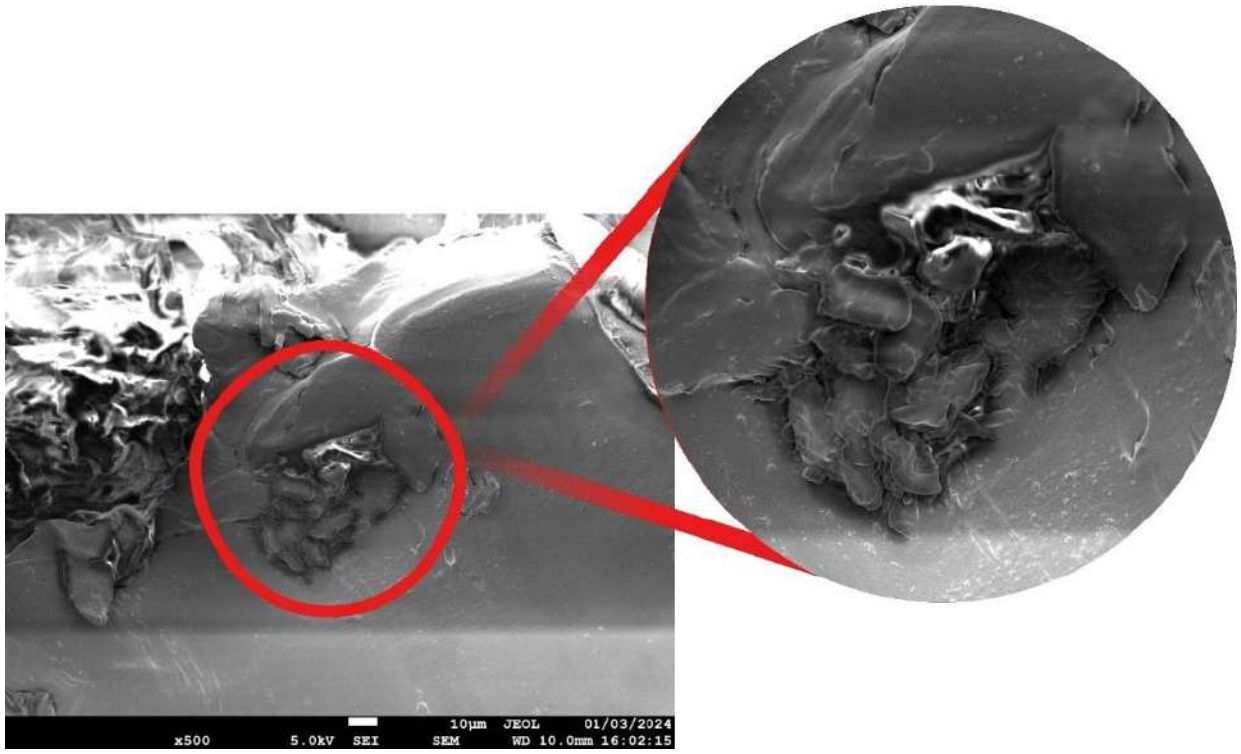

Figure 4.4- SEM image of PLA/ 1% yucca fiber powder extracted via traditional method.

On the other hand, **Figure 4.4** presents the microstructure of the bio-composite reinforced with 1% yucca fiber powder extracted using the traditional method (PLA-T1). In contrast to PLA-WR1, this bio-composite exhibited not only a homogeneous distribution of the reinforcement within the PLA matrix but also significantly improved interfacial adhesion. This enhanced adhesion can be attributed to the surface characteristics of the yucca fiber extracted via the traditional method, which featured a rougher texture and an optimal level of porosity, as observed in pervious section of this study. These factors promote better mechanical interlocking between the fiber and the matrix, facilitating stronger interfacial bonding and potentially improving the mechanical performance of the bio-composite [191].

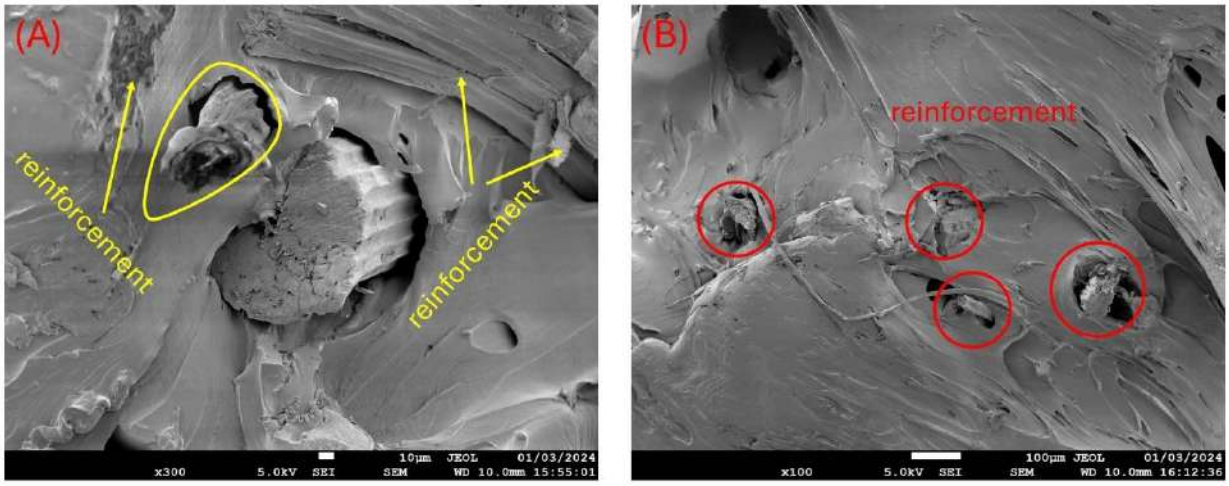

Figure 4.5- SEM analysis for bio-composite reinforced at 3% concentration, (A) bio-composite reinforced with yucca powder extracted via water retting, (b) bio-composite reinforced with yucca powder extracted via traditional method

Nevertheless, **Figures 4.5 (A and B)** illustrate the fracture surfaces of bio-composites reinforced with 3% yucca fiber powder, specifically PLA-WR3 and PLA-T3. The SEM analysis revealed a more complex surface morphology due to the higher concentration of reinforcement, which poses challenges in achieving a uniform dispersion of the yucca powder within the PLA matrix. This inhomogeneity was further exacerbated by the irregular orientation of the reinforcement particles, leading to inconsistencies in the material structure. Additionally, a significant presence of voids at the fiber-matrix interface was observed, indicating poor interfacial adhesion. This weak interfacial bonding negatively impacts the overall mechanical performance of the bio-composites, as confirmed by the diminished mechanical strength discussed in the following sections of this study.

4.4.2 Functional group identification (FTIR)

FTIR analysis was conducted to investigate the chemical interactions and structural modifications in both the pure PLA composite and the PLA-based bio-composites reinforced with yucca fiber powder. The FTIR spectra, presented in **Figure 4.6**, allow for a comparative assessment of the absorption bands, facilitating the identification of potential variations. Furthermore, this analysis provides insights into the influence of the fiber extraction method on the chemical composition and interfacial interactions within the bio-composites, thereby contributing to a deeper understanding of their structural and functional properties.

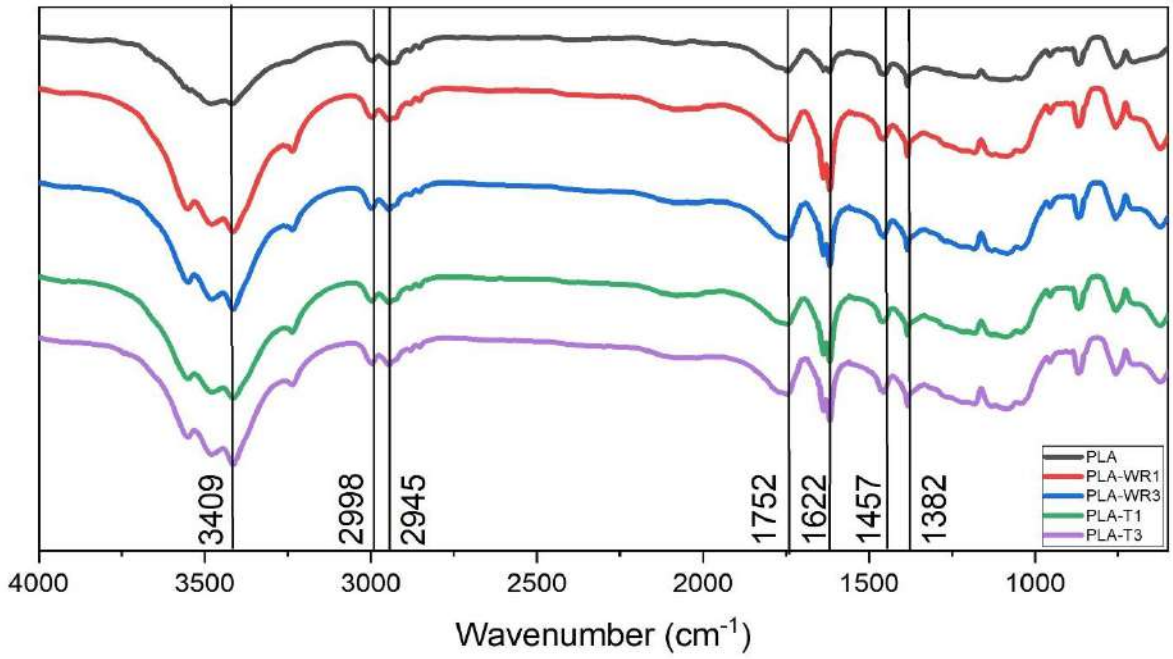

Figure 4.6- FTIR spectra of PLA/ yucca fiber powder bio-composites.

The FTIR spectra of both the pure composite and the bio-composites exhibited highly similar peak positions, observed at approximately 3409, 2998, 2945, 1752, 1622, 1457, and 1382 cm^{-1}. The primary distinction lied in the variation of peak intensity, which can be attributed to differences in cellulose content among the samples. Furthermore, the spectral region spanning 400 to 1200 cm^{-1} corresponds to the fingerprint region, which is known for its complexity and difficulty in detailed spectral analysis.

A broad peak centered around 3409 cm^{-1} was indicated of hydroxyl (-OH) groups present in both the matrix and reinforcement materials. The presence of double peaks at 2998 cm^{-1} and 2945 cm^{-1} has been widely attributed to the asymmetric and symmetric stretching vibrations of methyl ($-CH_3$) groups, as reported in previous studies [192]. Additionally, the distinct peak at 1752 cm^{-1} was characteristic of the stretching vibration of carbonyl (C=O) functional groups, commonly associated with the ester groups of PLA matrix. Moreover, the peak identified at 1622 cm^{-1} corresponded to angular deformation due to moisture absorption, which is a known phenomenon in biopolymeric materials. Within the spectral range of 1600 to 1200 cm^{-1}, two significant peaks were observed at 1457 cm^{-1} and 1382 cm^{-1}, which confirmed the presence of C-H bending vibrations. Specifically, these peaks were associated with the bending modes of -C-H- in CH_3 groups, as corroborated by previous investigations [193].

The overall similarity in the FTIR spectra across all samples can be directly attributed to the uniform use of the PLA matrix and the identical chemical composition of the reinforcing phase, namely yucca fibers. Despite morphological and mechanical variations induced by the different extraction methods, the fundamental chemical structure of the fibers remains unaltered, highlighting the molecular stability of the reinforcement. These FTIR findings further confirm the chemical compatibility between the PLA matrix and the yucca fiber powder.

4.5 Mechanical performance of bio-composites

4.5.1 Bio-composite tensile proprieties

The optimization of the mechanical performance of bio-composites requires a thorough understanding of the interactions between the polymer matrix and the reinforcing fibers. In this study, PLA-based bio-composites reinforced with yucca fibers extracted using different methods were developed and subjected to tensile testing to evaluate their mechanical properties. The results provide insights into the combined effects of the extraction method and fiber concentration on tensile strength, Young's modulus, and elongation at break. The key findings of this section are presented in **Figure 4.7** and summarized in **Table 4.1**.

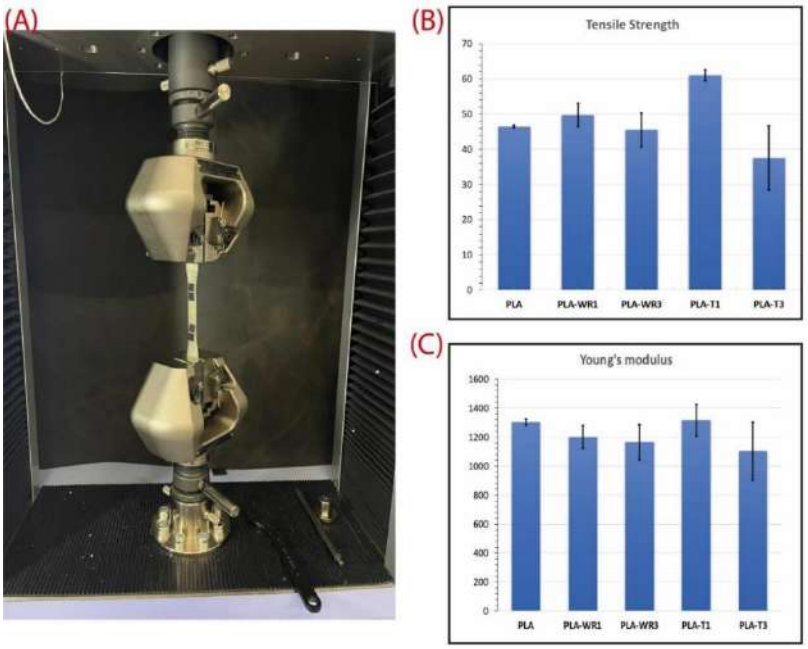

Figure 4.7 – PLA/ yucca powder tensile test results. (A) The bio-composite sample during the test. (B) and (C) are the tensile strength and Young's modulus results.

Table 4.1 – The bio-composite tensile test detailed results.

Sample Type	Tensile strength [MPa]		Young's modulus [MPa]		Elongation [%]	
	Mean	SD	Mean	SD	Mean	SD
PLA	46.42	0.40	1303	20	3.56	0.02
PLA-WR1	49.73	3.44	1201	80	4.14	0.17
PLA-WR3	45.48	4.83	1166	120	3.90	0.13
PLA-T1	61.06	1.60	1316	110	4.64	0.10
PLA-T3	37.46	9.17	1102	200	3.40	0.26

The results revealed that the incorporation of yucca fibers extracted via water retting (WR) and the traditional method (T) significantly influenced the tensile performance of the PLA composite. Pure PLA exhibited a tensile strength of 46.42 MPa, a Young's modulus of 1303 MPa, and an elongation at break of 3.56%, serving as the benchmark in this study. The addition of 1 wt.% yucca fiber powder extracted via water retting (PLA-WR1) led to a moderate increase in tensile strength to 49.73 MPa (+7.1% compared to pure PLA), while reducing Young's modulus to 1201 MPa, indicating a slight decrease in material stiffness. Conversely, the PLA-WR3 sample, containing 3 wt.% yucca fibers from water retting, exhibited a decrease in tensile strength to 45.48 MPa (-2% compared to pure PLA), along with a reduction in Young's modulus to 1166 MPa. These variations may be attributed to heterogeneous fiber dispersion or limited interfacial interactions between the matrix and the reinforcement [194].

In contrast, bio-composites reinforced with fibers extracted via the traditional method (PLA-T) demonstrated significant improvements in mechanical properties. The PLA-T1 bio-composite (1 wt.% fiber) achieved a tensile strength of 61.06 MPa, a 31.6% increase relative to pure PLA. This enhancement was accompanied by a slight increase in Young's modulus to 1316 MPa and an elongation at break of 4.64%. These improvements suggest superior fiber-matrix adhesion and a more homogeneous reinforcement distribution, likely influenced by the extraction method's impact on fiber surface characteristics. However, at a higher fiber concentration (3 wt.%, PLA-T3), a substantial decline in mechanical performance was observed. Tensile strength dropped to 37.46 MPa (a drop of 19.3% compared to pure PLA), while Young's modulus decreased to 1102 MPa, indicating a deterioration in mechanical properties, possibly due to fiber agglomeration, which disrupted composite homogeneity [194, 195].

These findings highlighted the critical role of both fiber concentration and extraction method in determining the mechanical performance of 3D-printed bio-composites. Yucca fibers extracted using the

traditional method appear to integrate more effectively within the PLA matrix, leading to notable improvements in tensile properties. In contrast, fibers extracted via water retting exhibited more variable performance. Therefore, optimizing the reinforcement ratio and enhancing fiber-matrix interactions remain key factors in improving the overall quality of bio-composites based on natural fibers.

Table 4.2 – Tensile test results of other PLA-based bio-composites reported in the literature.

Composite type	Tensile strength [MPa]	Young's modulus [MPa]	Elongation [%]	Ref.
PLA-yucca	61.06	1316	4.64	Present work
PLA-sisal	≈55.00	≈2100	≈2.60	[196]
PLA-jute	18.24	≈1220	11.48	[197]
PLA-kenaf	≈28.00	≈480	≈13.00	[198]
PLA-basalt	≈61.00	≈3000	≈2.70	[199]

A comparative analysis of the tensile properties of the PLA-yucca bio-composite with other similar composites underscores the potential of yucca fibers as an effective reinforcement in a polymer matrix as demonstrated in **Table 4.2**. The PLA-yucca bio-composite exhibited a tensile strength of 61.06 MPa, a Young's modulus of 1316 MPa, and an elongation at break of 4.64%. Compared to PLA-sisal (≈55 MPa, 2100 MPa, 2.60%), PLA-yucca demonstrates slightly higher tensile strength, although its Young's modulus is lower. This suggests that yucca fibers enhance the composite's ability to withstand mechanical loads while maintaining a degree of flexibility. Similarly, in comparison with PLA-jute (18.24 MPa, ≈1220 MPa, 11.48%), PLA-yucca exhibited significantly superior tensile strength, indicating the robustness of yucca fibers when integrated into the PLA matrix. On the other hand, PLA-kenaf (≈28 MPa, ≈480 MPa, 13.00%) showed much higher elongation at break, reflecting greater ductility but at the cost of lower tensile strength and stiffness compared to PLA-yucca. Additionally, when compared with PLA reinforced with basalt fibers (≈61 MPa, ≈3000 MPa, 2.70%), the PLA-yucca bio-composite displayed an equivalent tensile strength but a considerably lower Young's modulus. This suggested that while yucca fibers contribute to mechanical strength, basalt fibers provide significantly higher rigidity to the final material.

4.5.2 Bio-composite compression proprieties

The compressive strength of bio-composites is a crucial parameter, especially for structural applications subjected to axial loads. Unlike tensile failure, which is primarily governed by crack

propagation, compressive failure involves intricate mechanisms such as fiber buckling, matrix microcracking, and local instabilities. The addition of natural yucca fibers significantly influences these failure mechanisms by altering the fiber-matrix interaction and redistributing stresses within the composite structure. **Figure 4.8** presents the compression test results for PLA and PLA reinforced with yucca fiber powder, depicted through both a histogram and force-stroke curves. These graphical representations provide insight into the influence of fiber incorporation on the compressive behavior of the bio-composites.

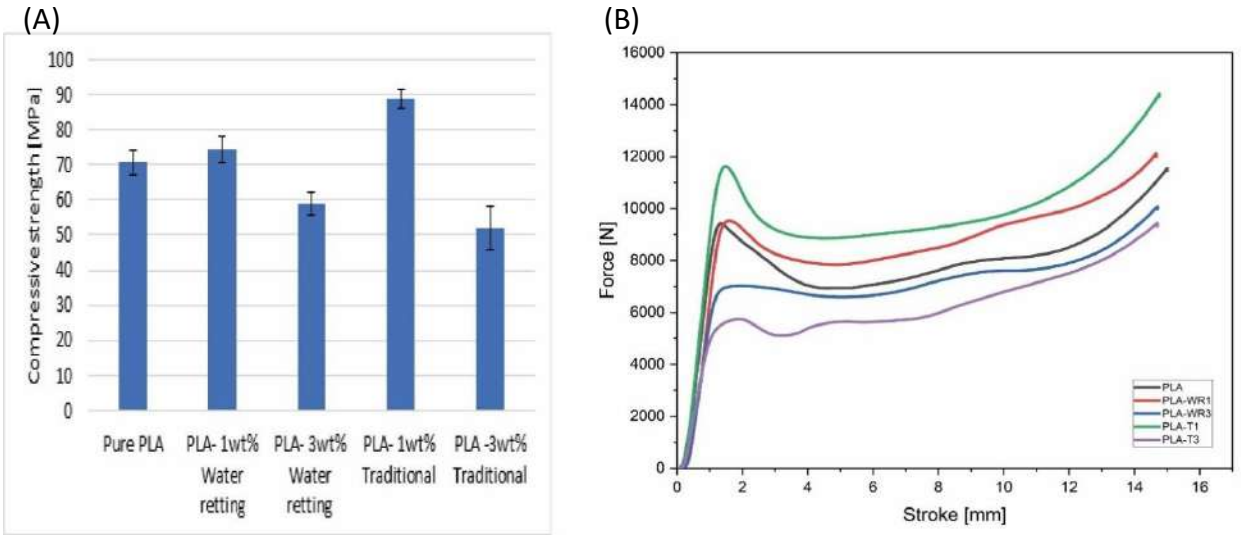

Figure 4.8- 3D printed bio-composite compression test results. (A) Histogram of the compressive strength. (B) Force graph results during the compression test.

The experimental results presented in **Figure 4.8(A)** highlight contrasting trends depending on fiber concentration and extraction method. Pure PLA exhibited a compressive strength of 70 MPa, serving as a reference for the comparative analysis of bio-composites. The incorporation of fibers extracted by water retting led to a notable improvement in the performance of PLA-WR1, which reaches a compressive strength of 74 MPa (+5.7%), suggesting good dispersion and effective interaction with the PLA matrix. In contrast, PLA-WR3 showed a decrease in strength to 59 MPa, which could be attributed to an inhomogeneous distribution of yucca fibers and the formation of micro-defects that compromise the material's overall integrity [200].

For yucca fibers extracted using the traditional method, the PLA-T1 bio-composite exhibited the best performance, with a compressive strength of 89 MPa, representing a significant increase of 27.1% compared to pure PLA. This result underscores the effectiveness of this extraction method in enhancing

fiber-matrix compatibility, likely due to better preservation of the fiber structure and improved interfacial adhesion. Conversely, the PLA-T3 bio-composite demonstrated a reduced compressive strength of 52 MPa (-25.7%), which may be associated with an excessive fiber concentration, leading to increased porosity and degradation of the matrix's mechanical properties [191].

Another critical aspect of the analysis of the compressive properties of bio-composites was the evaluation of the maximum forces sustained before failure or critical deformation, as illustrated in Figure 4.8(B). The results indicated that pure PLA withstands a maximum force of 9425 N, serving as a benchmark for reinforced bio-composites. The incorporation of yucca fibers extracted via water retting slightly improved the load-bearing capacity of PLA-WR1, reaching 9510 N (+0.9%), suggesting enhanced absorption of compressive stresses. In contrast, the PLA-WR3 bio-composite exhibits a marked decrease in maximum force to 6999 N (-25.7%), indicating a heterogeneous fiber dispersion and reduced interfacial cohesion, leading to a loss of mechanical integrity under high loads. Bio-composites derived from traditional fiber extraction showed similar trends to those observed for compressive strength. PLA-T1 supported the highest maximum load, reaching 11,588 N (+22.9%), confirming more efficient load transmission and improved interaction between the matrix and fibers. Conversely, PLA-T3 displayed a drastic reduction in maximum load to 5370 N (-43.0%), likely due to an excessive concentration of yucca fibers, leading to internal defects and increased brittleness under compressive stress.

These observations confirmed that the compressive performance of bio-composites is strongly influenced by the nature and quantity of incorporated fibers. Homogeneous fiber distribution and strong fiber-matrix adhesion are crucial for enhancing resistance to high loads, whereas excessive reinforcement content or uneven dispersion can compromise the material's structural integrity.

4.5.3 Bio-composite bending performance

Assessing the bending properties of bio-composites is crucial for understanding their mechanical behavior under load, particularly in structural applications where resistance to deformation is a key performance criterion. Bending loading is especially relevant for composite materials, as it emphasizes the interactions between the polymer matrix and the fibrous reinforcements. In addition to evaluating the stiffness and strength of bio-composites, this analysis investigates the influence of different fiber extraction methods on their mechanical performance. The results of this study are summarized in **Table 4.3**.

Table 4.3- 3D printed PLA-based bio-composite bending test results.

Samples type	Bending strength [MPa]		Bending modulus [MPa]	
	Mean	SD	Mean	SD
PLA	53.06	0.38	458.9	0.18
PLA-WR1	50.39	1.73	507.3	0.43
PLA-WR3	47.07	1.13	515.7	0.29
PLA-T1	56.57	0.87	488.4	0.27
PLA-T3	41.58	2.17	485.8	0.29

The results of the bending tests, presented in **Table 4.3**, revealed significant variations depending on the type and concentration of yucca fibers. Pure PLA, used as the reference material, exhibited a bending strength of 53.06 MPa and a modulus of 458.9 MPa. The incorporation of yucca fibers extracted via water retting into PLA led to a slight reduction in strength, reaching 50.39 MPa for PLA-WR1 (-5.0%) and 47.07 MPa for PLA-WR3 (-11.3%) compared to pure PLA. According to several studies, this decrease can be attributed to the influence of the extraction methodology on the fiber surface. Specifically, the hydrophilic nature of water-retted yucca fibers contrasts with the hydrophobicity of PLA, potentially limiting interfacial adhesion and promoting crack initiation under bending stress. However, these samples exhibited a significant improvement in bending modulus, with increases of 10.5% (507.3 MPa) and 12.4% (515.7 MPa) for PLA-WR1 and PLA-WR3, respectively. This increase is likely due to improved fiber dispersion and the stiffening effect induced by the reinforcement [201].

Conversely, bio-composites incorporating yucca fibers extracted using the traditional method exhibited distinct mechanical behavior. The PLA-T1 sample demonstrated the highest bending strength at 56.57 MPa, representing a 6.6% increase compared to pure PLA, suggesting a more effective fiber-matrix interaction for fibers obtained through this extraction process. However, when the fiber concentration increased to 3% (PLA-T3), the bending strength significantly decreased to 41.58 MPa (-21.7% compared to pure PLA). This reduction may be attributed to a less homogeneous fiber dispersion and diminished interfacial adhesion, which hinder effective load transfer between the matrix and the fibers. Additionally, the bending modulus remained relatively stable for both samples, with values of 488.4 MPa and 485.8 MPa, corresponding to increases of 6.4% and 5.9%, respectively, compared to pure PLA [202].

4.6 Thermogravimetric properties of bio-composites

4.6.1 Thermogravimetric analysis (TGA)

Thermogravimetric analysis was conducted to assess the thermal stability of pure PLA and PLA-based bio-composites reinforced with yucca fiber powder, with a particular focus on evaluating the influence of the fiber extraction method on the thermal properties of the final materials. The TGA results for both the pure PLA composite and the PLA-Yucca fiber powder bio-composites are presented in **Figure 4.9**, while **Table 4.4** summarizes key thermal parameters, including the maximum decomposition temperature (Tmax) and the total weight loss at 500 °C.

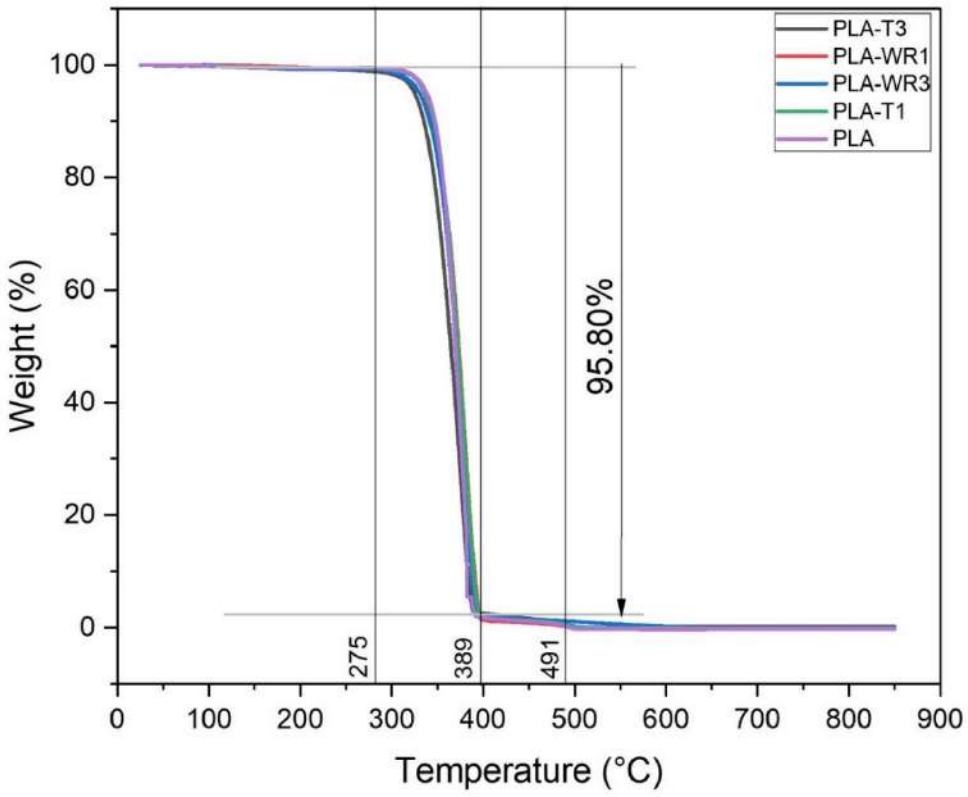

Figure 4.9- TGA analysis graphs for PLA-based bio-composites.

The TGA analysis revealed a strong similarity in the thermal degradation behavior of the pure PLA composite and PLA-based bio-composites, with three distinct thermal degradation zones: Zone 1 (25–275 °C), Zone 2 (276–389 °C), and Zone 3 (390–501 °C). These zones corresponded to the initial mass loss (IML), onset of decomposition (OD), and decomposition tray zone (DT), respectively. The close proximity of the TGA curves observed in **Figure 4.9**, aligns with previous studies, suggesting that the thermal degradation

behavior is primarily governed by the PLA matrix due to the relatively low reinforcement content (1% and 3%) [203].

In Zone 1, all samples exhibited a similar weight percentage at approximately 90 °C, with a minor weight loss recorded between 100 and 270 °C. This loss was attributed to the depolymerization of non-cellulose components, such as hemicellulose, and the evaporation of adsorbed moisture from the yucca fibers.

Zone 2 marks the primary thermal decomposition stage, where all composites and bio-composites begin degrading around 275 °C, with a significant mass loss of approximately 96% of the initial weight. Notably, the bio-composites reinforced with 1 wt.% yucca fiber powder exhibited a slightly higher maximum degradation temperature (Tmax) of 393 °C (PLA-WR1) and 394 °C (PLA-T1) compared to those reinforced with 3 wt.%, which have a Tmax of 387 °C (PLA-WR3) and 388 °C (PLA-T3). These findings suggested that incorporating yucca fiber powder into PLA marginally enhances the thermal stability of the composite, as the Tmax of pure PLA is 385 °C [204].

In Zone 3, the decomposition tray region, all samples demonstrated thermal stability, retaining approximately 4% of their initial mass. However, at 500 °C ±1, complete degradation occurs for both pure PLA and bio-composite samples, confirming that the PLA matrix remains the dominant factor in the thermal degradation process.

Table 4.4 – Exact TGA analysis results for PLA-based bio-composites.

Samples type (PLA- extraction method)	T_{max} (°C)	Total weight loss at 500°C (%)
Pure PLA	385	100
PLA-WR1	393	99
PLA-WR3	387	100
PLA-T1	394	98
PLA-T3	388	99

Based on **Table 4.4**, This analysis highlighted that incorporating yucca fiber powder as a reinforcement in PLA composites enhances the final thermal stability of the resulting bio-composites. However, this improvement remained relatively modest, likely due to the low variation in reinforcement concentration. The results indicated that composites reinforced with 1 wt.% yucca fiber powder exhibited better thermal stability compared to those containing 3 wt.%, as well as pure PLA, a trend consistent with findings from other studies [205]. Furthermore, the traditional extraction method yielded fibers with

superior thermal performance compared to those obtained through water retting. This difference may be attributed to the extraction process itself, as fibers exposed to water during retting tend to absorb more moisture. The higher water content in these fibers could reduce their thermal resistance, making them more susceptible to degradation.

Additionally, several studies have demonstrated that lignin content plays a crucial role in thermal stability by acting as an insulating layer around cellulose microfibrils, thereby improving the resistance of both the reinforcement (natural fiber powder) and the final PLA-based bio-composite. Notably, a previous section reported that fibers extracted via the traditional method contained a slightly higher lignin content (10.95%) compared to those obtained through water retting (10.45%), which may further explain the superior thermal performance of the traditionally extracted fibers.

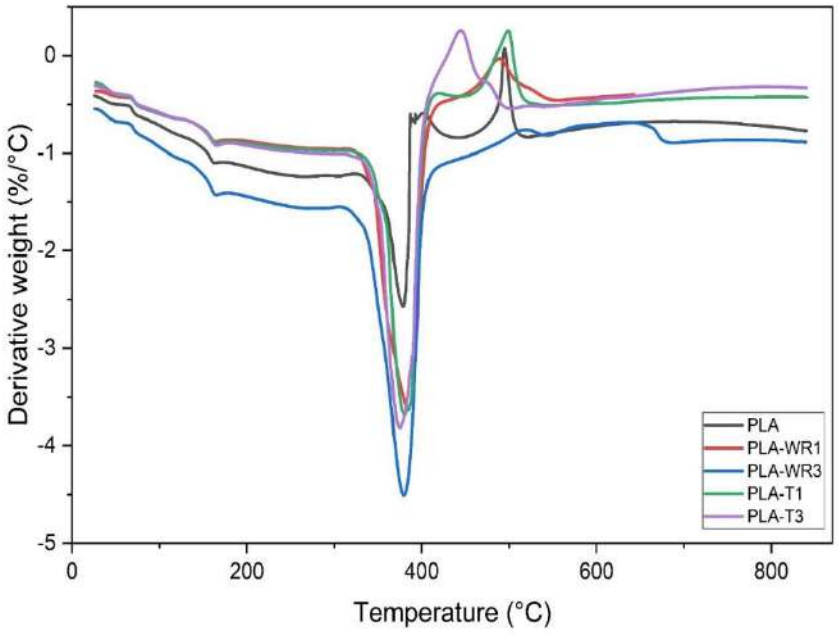

Figure 4.10 - DTG analysis graphs for PLA-based bio-composites.

Furthermore, the peak temperature of the DTG (**Figure 4.10**) was very similar to that of the TGA. However, it was therefore confirmed that the maximum temperature of the pure PLA composite and PLA-Yucca fiber powder bio-composite samples were around 390 °C.

4.7 Dynamic properties of bio-composites

4.7.1 Impact resistance

Figure 4.13 presents the Charpy impact strength results, divided into two graphs (A and B), illustrating the average absorbed energy and material toughness. The bio-composites demonstrated superior performance compared to pure PLA, which exhibited an impact strength of approximately 11 kJ/m² and an average energy absorption of 4.5 J. The relative improvements were estimated at 37%, 22.24%, 40.72%, and 7.45% for the bio-composites PLA-WR1, PLA-WR3, PLA-T1, and PLA-T3, respectively.

Among these, PLA-T3 exhibited the highest impact resistance, reaching 15.83 kJ/m², with an average absorbed energy of 6.3 J, followed by PLA-WR1 (15.41 kJ/m², 6.1 J), PLA-WR3 (13.73 kJ/m², 5.5 J), and PLA-T1 (12.08 kJ/m², 4.8 J). The enhanced impact strength observed in PLA-T1 and PLA-WR1 was attributed to optimized 3D printing parameters and an ideal fiber powder concentration, which improved the homogeneity and cohesion between the reinforcement and the matrix [206].

Conversely, PLA-T3 and PLA-WR3 exhibited lower impact strength values, a phenomenon frequently reported in the literature and attributed to the higher reinforcement content. Excessive fiber loading can lead to increased fiber pull-out during fracture, resulting in greater energy dissipation and a subsequent reduction in impact strength [207].

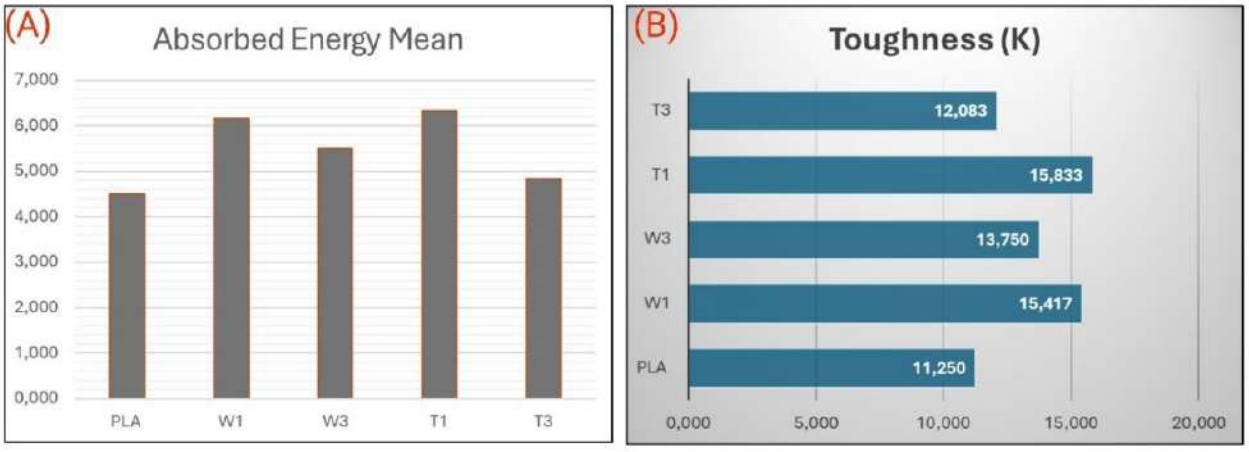

Figure 4.11- Charpy impact strength results. (A) represent the average of absorbed energy results for PLA based bio-composites. (B) represent the average of toughness results for PLA based bio-composites.

4.7.2 DMA analyses results

Figure 4.12 presents the graphs obtained from the DMA analysis, providing insight into the thermo-mechanical behavior of the pure PLA composite and the PLA-based bio-composites reinforced with yucca fiber powder. This analysis also aims to evaluate the influence of the fiber extraction method on the dynamic mechanical properties of the final materials. A more detailed summary of the results is given in **Table 4.5**, which includes key parameters such as the glass transition temperature (Tg), storage modulus, and mechanical damping factor (tan δ) for each sample.

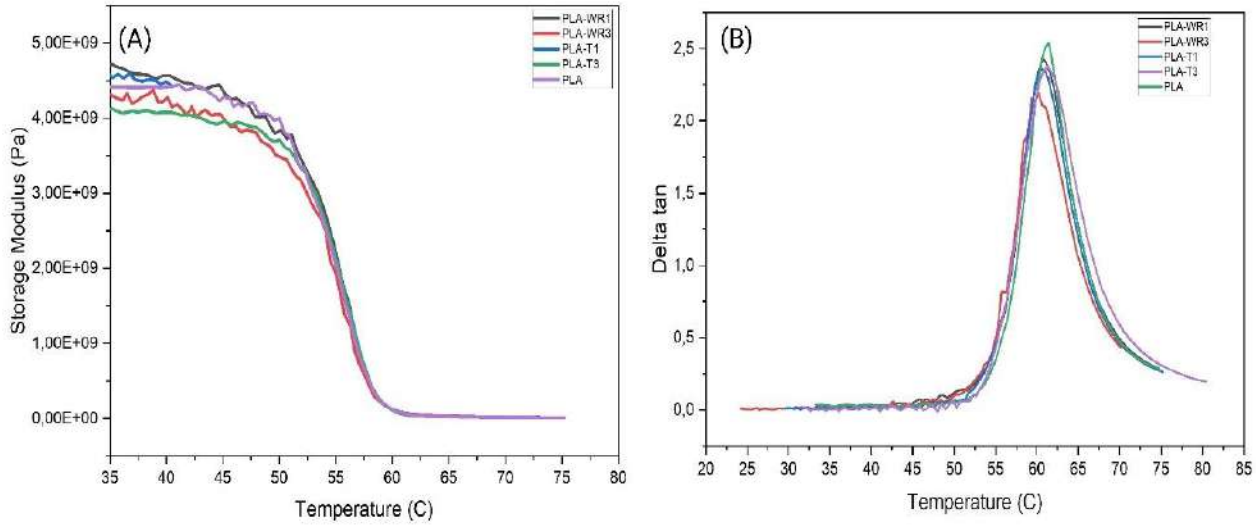

Figure 4.12 Dynamic mechanical analysis results. (A) and (B) represents the storage modulus and delta ten results of PLA based bio-composites respectively.

Table 4.5 - Detailed Dynamic mechanical analysis results of PLA-based Bio-composites.

Samples type	T_g (°C)	Storage modulus (GPa)		
		T = 35 °C	T = 50 °C	T = 65 °C
PLA	61.3	4.41	3.75	0.022
PLA-WR1	60.7	4.72	3.84	0.022
PLA-WR3	60.22	4.30	3.47	0.022
PLA-T1	60.98	4.53	3.80	0.023
PLA-T3	61.01	4.12	3.61	0.019

As a result, the addition of 1% yucca fiber powder to the PLA composite led to an increase in the storage modulus by 7.04% for PLA-WR1 and 2.72% for PLA-T1 compared to pure PLA, which was measured at 4.4 GPa (at 35 °C). The storage modulus values for PLA-WR1 and PLA-T1 were approximately 4.72 GPa and 4.53 GPa, respectively. Similar findings in previous studies attributed this improvement to the rigid structure of the fiber powder, enhancing load transfer efficiency within the composite and promoting better homogeneity in the matrix. However, a slight decrease was observed in PLA-WR3 and PLA-T3, with storage modulus values of 4.30 GPa and 4.12 GPa, respectively. This decline is likely due to higher reinforcement content affecting dispersion and homogeneity, leading to suboptimal load distribution and, consequently, reduced mechanical performance [208].

Moreover, **Figure 4.12(B**) illustrates the tan δ (damping factor) curves of the pure PLA composite and PLA-yucca bio-composites as a function of temperature. The pure PLA composite exhibited the highest tan δ peak, while the bio-composites (PLA-WR1, PLA-T1, PLA-WR3, and PLA-T3) showed reduced peaks, with PLA-WR3 presenting the lowest value. This behavior suggested that cellulose reinforcement restricts polymer chain mobility, leading to a decrease in molecular damping and enhancing the structural rigidity of the composites. Additionally, the glass transition temperature (Tg) for all samples remained around 61°C, indicating that fiber type and content had a negligible effect on this parameter [209].

These results further emphasized that the extraction method plays a role in determining the structural and thermal behavior of the composites, particularly in terms of fiber dispersion and interfacial adhesion.

4.7.3 Fatigue performance

The 3D-printed specimens, including both the pure PLA composite and the PLA-based bio-composites reinforced with yucca fiber powder, were subjected to repeated tension-compression loading until failure to evaluate their fatigue behavior. The results of the fatigue tests are presented in **Figure 4.13** as S-N curves (Wöhler curves), which illustrate the relationship between applied stress (S) and the number of cycles to failure (N). Additional quantitative details are provided in **Table 4.6**.

Table 4.6 - Fatigue proprieties of PLA based bio-composites studied.

Level (%)	Sample type	Maximum Stress (Smax)	Cycle number (N)
70	PLA	32.50	164
	PLA-WR1	34.81	538
	PLA-WR3	31.83	80
	PLA-T1	42.74	1013
	PLA-T3	-	-
40	PLA	18.56	1885
	PLA-WR1	19,89	4263
	PLA-WR3	18.19	121
	PLA-T1	24.42	4673
	PLA-T3	-	-
20	PLA	9.28	10679
	PLA-WR1	9.95	24123
	PLA-WR3	9.09	2654
	PLA-T1	12.21	40185
	PLA-T3	-	-

In the literature, the Wöhler curve defines the relationship between cyclic load levels and the number of cycles to failure. This curve is typically divided into two main regions: the low-cycle fatigue zone (less than 10^4 cycles) and the high-cycle fatigue zone (more than 10^4 cycles). As illustrated in **Figure 4.13**, a clear trend emerges where fatigue life increases as applied stress decreases. The results presented in **Table 4.6** highlight significant variations in fatigue performance among the studied materials, attributed to both the amount of yucca fiber powder incorporated into the PLA matrix and the extraction methodology used for the natural fiber [210, 211].

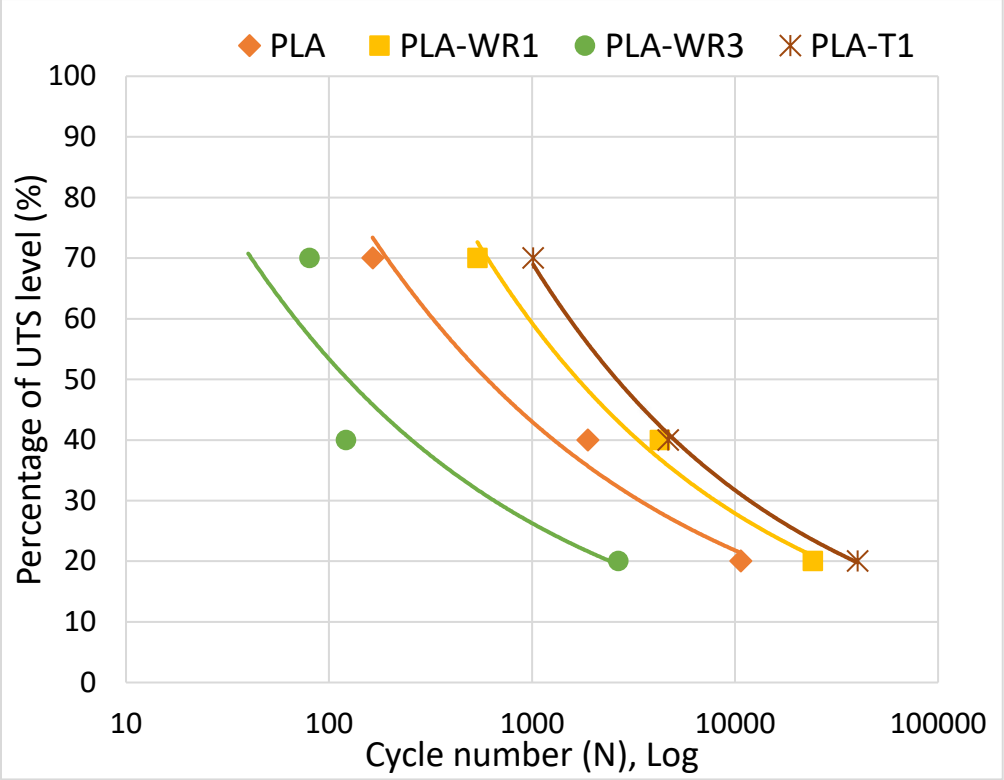

Figure 4.13- S-N curve obtained from the fatigue test on 3D printed PLA-based biocomposites.

In the low-cycle fatigue region, particularly at an amplitude of 70% of the ultimate tensile strength (UTS), pure PLA samples exhibited an average fatigue life of approximately 164 cycles. The absence of yucca bio-fiber powder reinforcement in these compositions makes them more susceptible to fatigue failure under high loads. However, the addition of 1 wt.% yucca fiber powder enhanced the lifespan of the bio-composite, extending it to 538 cycles for PLA-WR1 and 1013 cycles for PLA-T1. This improvement can be attributed to the presence of yucca particles, which acted as load-bearing elements, promoting a more uniform distribution of mechanical stresses and reducing local stress concentrations, key factors in delaying crack initiation and subsequent failure. Conversely, an excessive amount of yucca powder, as in PLA-WR3, resulted in a less homogeneous composite structure, adversely affecting fatigue resistance and leading to premature failure at around 80 cycles. Similarly, the PLA-T3 composite, characterized by a high fiber content obtained through the traditional extraction method, exhibited weak mechanical properties, particularly in compression. Consequently, it could not be tested in this section, as its low compressive strength caused premature specimen failure before the fatigue test could be conducted [211].

In the intermediate range of the low-cycle fatigue zone (40% UTS), a clear improvement in fatigue performance was observed. The PLA-T1 bio-composite demonstrated exceptional durability, reaching 4673 cycles, closely followed by PLA-WR1 at 4263 cycles. Both results significantly surpass the pure PLA composite, which endured 1885 cycles. In contrast, PLA-WR3 again exhibited the lowest fatigue life, failing after only 121 cycles. The superior performance of PLA-WR1 and PLA-T1 can be attributed to the presence of yucca fiber powder, which acted as a physical barrier, impeding crack propagation. When a crack encounters a fiber particle, it may be deflected or arrested, effectively slowing the propagation rate and enhancing fatigue resistance [212].

In the high-cycle fatigue zone, which corresponds to failure beyond 10^4 cycles under low-stress conditions, the fatigue behavior further differentiates among the tested materials. At 20% UTS, the pure PLA composite achieved a fatigue life of 10,675 cycles, whereas PLA-WR1 significantly outperforms it, reaching 24,123 cycles. Notably, PLA-T1 exhibited the highest fatigue resistance, with an impressive endurance of 42,185 cycles. This outstanding performance can likely be attributed to the superior quality of the yucca powder particles obtained through traditional extraction, which enhances interfacial adhesion with the PLA matrix, thereby promoting efficient stress distribution and reducing localized stress accumulation.

These findings demonstrated the importance of the fiber extraction process and treatment in optimizing the mechanical performance of bio-composites in fatigue. They also highlight the essential role of good fiber-matrix compatibility in ensuring durable mechanical resistance to dynamic stress.

4.8 Durability of bio-composites

4.8.1 Water absorption

In this study, 3D-printed pure PLA samples and bio-composites reinforced with yucca powder were evaluated under different environmental conditions, including seawater (collected from the Algerian coast of the Mediterranean Sea, pH = 8.1) and spring water. **Figure 4.14 (A and B)** presents the graphical representation of the results obtained from this analysis.

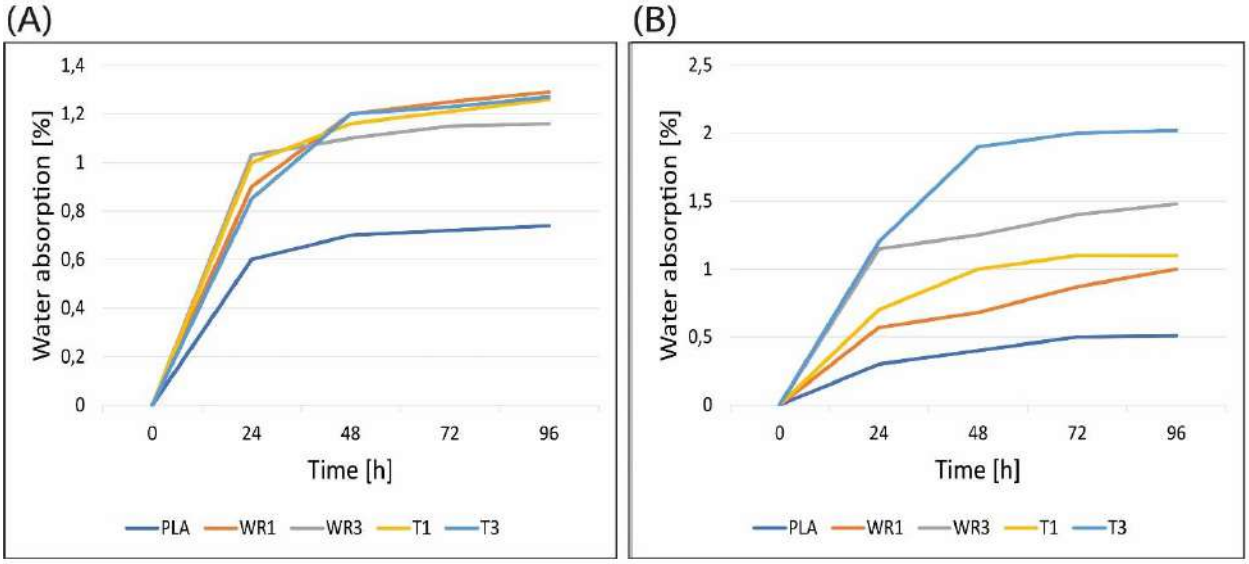

Figure 4.14- Water absorption graphs result. (A) the test results in the spring water condition. (B) the test results in the seawater condition.

The results indicated that pure PLA exhibits low water absorption, reaching approximately 0.3% of its initial weight after 24 hours in spring water, compared to 0.6% in seawater. A progressive increase was observed in both conditions, with absorption levels stabilizing at 0.75% and 0.55% after 72 hours for normal and seawater immersion, respectively. The incorporation of yucca powder enhanced the moisture resistance of PLA-based bio-composites but also increased water absorption due to the hydrophilic nature of the reinforcement, attributed to the presence of hydroxyl groups. The amphipathic behavior of the composite results from the interplay between the hydrophobic PLA matrix and hydrophilic fiber content, influencing water uptake.

- In spring water conditions, the PLA-WR3 bio-composite exhibited the highest absorption (1% after 24 hours), while PLA-T3 showed a peak absorption of 1.2% after 48 hours.
- Under seawater conditions, higher absorption rates were recorded across all bio-composites due to the presence of sodium chloride (NaCl), which may facilitate water retention within the material structure. The PLA-T3 bio-composite exhibited the highest absorption, reaching 1.2% after 24 hours and increasing up to 2% over time.

Overall, bio-composites reinforced with 3% yucca powder displayed higher water absorption than those with 1% reinforcement, confirming the influence of fiber content on the hydrophilic properties of the material.

4.9 Epoxy based bio-composite

4.9.1 Elaboration challenge

The fabrication of bio-composites based on epoxy resin and yucca fiber powder presents several technical and practical challenges that significantly influence the quality and performance of the final materials. These challenges are primarily associated with mixing conditions, the rheological behavior of the liquid composite, the curing process, and the necessary precautions to ensure operator safety.

One of the most critical factors was maintaining the appropriate mixing ratio between the epoxy resin and the hardener. Any deviation from the recommended ratio can hinder complete polymerization, leading to inadequate solidification of the composite and a substantial reduction in its mechanical properties. To prevent such issues, it is imperative to strictly adhere to the manufacturer's guidelines and employ precise measuring instruments, such as electronic scales, to ensure accurate dosing.

Another major challenge concerns the viscosity of the mixture, which varies with the concentration of yucca fiber powder incorporated into the matrix. An excessive filler content increased viscosity, making the mixture difficult to manipulate and potentially leading to inadequate particle impregnation within the resin. To optimize this stage, preliminary trialed should be conducted to determine the optimal powder concentration that balances fluidity and mechanical reinforcement. Additionally, the curing time was a key parameter in composite processing. A curing process that is too rapid may trap air bubbles, resulting in internal defects, whereas excessively slow curing can compromise the material's integrity. To mitigate these issues, it was advisable to maintain controlled temperature and humidity conditions throughout the curing process.

Another challenge encountered during curing was the adhesion of specimens to the walls of the silicone mold, which complicates demolding and may cause surface defects. To address this issue, the prior application of a release agent, such as demolding wax, was strongly recommended. Finally, the handling of epoxy resins poses health risks due to their toxicity and potential to cause skin and respiratory irritation. To minimize these hazards, the use of personal protective equipment, including gloves, masks, and safety goggles, was essential.

4.9.2 Fiber matrix adhesion inspection

Morphological analysis of the samples using scanning electron microscopy (SEM) was conducted to evaluate the interaction between mechanically extracted yucca fibers and the epoxy matrix. This examination is crucial for assessing fiber-matrix adhesion and detecting potential defects that may influence the mechanical properties of the bio-composites.

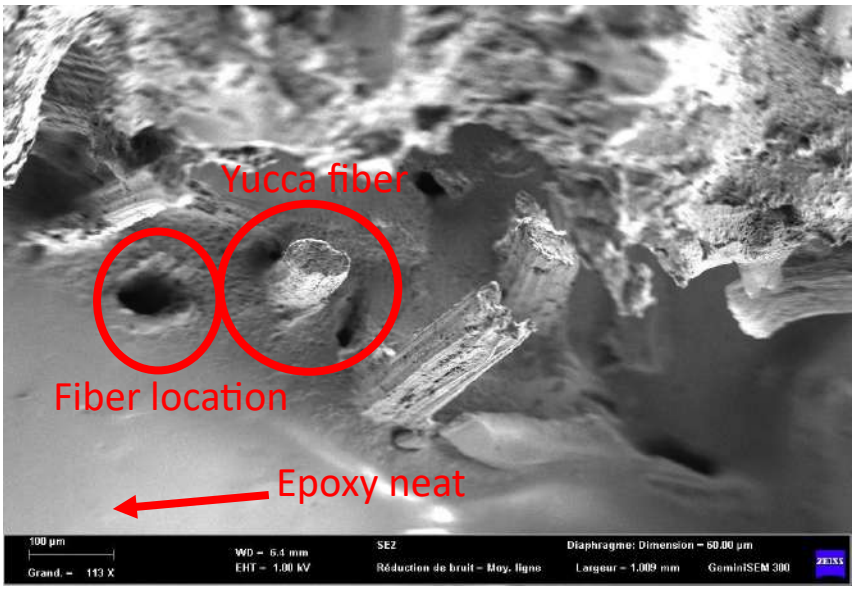

Figure 4.15- SEM image of epoxy/untreated yucca fiber powder**.**

The SEM images presented in **Figure 4.15** revealed a homogeneous structure in the absence of bio-based fibers, characterized by a predominantly smooth and glassy fracture surface. However, upon the incorporation of mechanically extracted yucca fibers, weak interfacial adhesion was observed. The presence of interfacial voids and zones of decohesion suggested inadequate fiber anchoring within the polymer matrix, likely due to chemical incompatibility between the fibers and the epoxy resin. This poor adhesion may be attributed to the absence of surface treatments that promote effective fiber-matrix interaction. Furthermore, the presence of pulled fibrils following fracture indicated a predominantly interfacial failure mode. Consequently, the mechanically extracted yucca fibers fail to establish a robust interfacial bond with the epoxy matrix, thereby hindering efficient stress transfer between the two composite phases [213].

4.9.3 Mechanical performance

This section evaluates the mechanical properties of bio-composites to gain deeper insights into the impact of natural fiber reinforcement on the structural performance of the material. Tensile, compression, and bending tests were conducted to assess the influence of fiber incorporation on both strength and stiffness. The results, summarized in **Table 4.7**, underscore the benefits and challenges associated with natural fiber reinforcement, particularly regarding structural enhancement, overall mechanical behavior, and the potential for optimization in specific applications.

Table 4.7 - Mechanical proprieties of neat epoxy composite and epoxy/untreated yucca fiber bio-composite.

Sample type	Tensile test			Compressive test	Bending test	
	Tensile strength [MPa]	Young's modulus [MPa]	Elongation [%]	Compressive strength [MPa]	Bending strength [MPa]	Bending modulus [MPa]
Pure epoxy	23.70 ±1.04	2132.36 ±178.03	1.33 ±0.11	59.57 ±0.52	40.45 ±20.50	884.21 ±318.57
Biocomposite	23.59 ±2.30	1598.12 ±242.82	3.01 ±0.75	72.41 ±0.15	39.34 ±05.07	1278.28 ±277.62

Firstly, the tensile test results revealed that the bio-composite exhibited a tensile strength of 23.59 MPa, which was nearly identical to that of pure epoxy (23.70 MPa), indicating that the incorporation of yucca fibers did not significantly affect tensile strength. However, a decrease in Young's modulus was observed, dropping from 2132.36 MPa for pure epoxy to 1598.12 MPa for the bio-composite. This reduction can be attributed to the heterogeneous distribution of fibers within the matrix and potentially poor fiber-matrix adhesion, which limits the efficiency of load transfer. Conversely, the elongation at break increased significantly from 1.33% for pure epoxy to 3.01% for the bio-composite, reflecting an improved ability to accommodate deformation before failure [214].

Furthermore, the compression test results indicated a notable improvement in the mechanical performance of the bio-composite compared to pure epoxy. The compressive strength increased from 59.57 MPa for epoxy to 72.41 MPa for the bio-composite, representing an enhancement of approximately 21.5%. This improvement can be attributed to the reinforcing role of yucca fibers, which help restrict crack propagation under compressive loading. Regarding bending properties, the bio-composite exhibited a

bending strength of 39.34 MPa, slightly lower than that of pure epoxy (40.45 MPa). However, the bending modulus showed a substantial increase, rising from 884.21 MPa for pure epoxy to 1278.28 MPa for the bio-composite, an improvement of approximately 44.6%. This increase indicates enhanced bending stiffness, which could be beneficial for applications requiring improved structural integrity under static loading [215].

These findings demonstrated that incorporating natural yucca fibers into an epoxy matrix significantly influences the mechanical properties of the composite. The improvements in compressive strength and bending modulus suggested that the material exhibits greater resistance to axial and bending loads. However, the reduction in tensile modulus underscored the need to optimize the fiber-matrix interface to enhance load transfer and achieve more effective reinforcement. Compared to other bio-composites reported in the literature, these values align with trends observed for plant fiber-reinforced composites. Previous studies have highlighted that improvements in mechanical properties are highly dependent on fiber-matrix adhesion and fiber orientation within the composite. Therefore, the implementation of appropriate surface treatments could further enhance the mechanical performance of the bio-composite under study [216].

4.10 Chemical treatments impact on bio-composite

4.10.1 Adhesion inspection

The scanning electron microscopy (SEM) analysis of bio-composites reinforced with yucca fibers treated with 3 wt.% NaOH and 1 wt.% sulfuric acid provides valuable insights into the effects of chemical treatments on the fiber-matrix interaction within the epoxy composite. This examination revealed significant differences between the two types of samples, particularly regarding interfacial adhesion and fracture morphology. The SEM images illustrating these observations are presented in **Figures 4.16** and **4.17**.

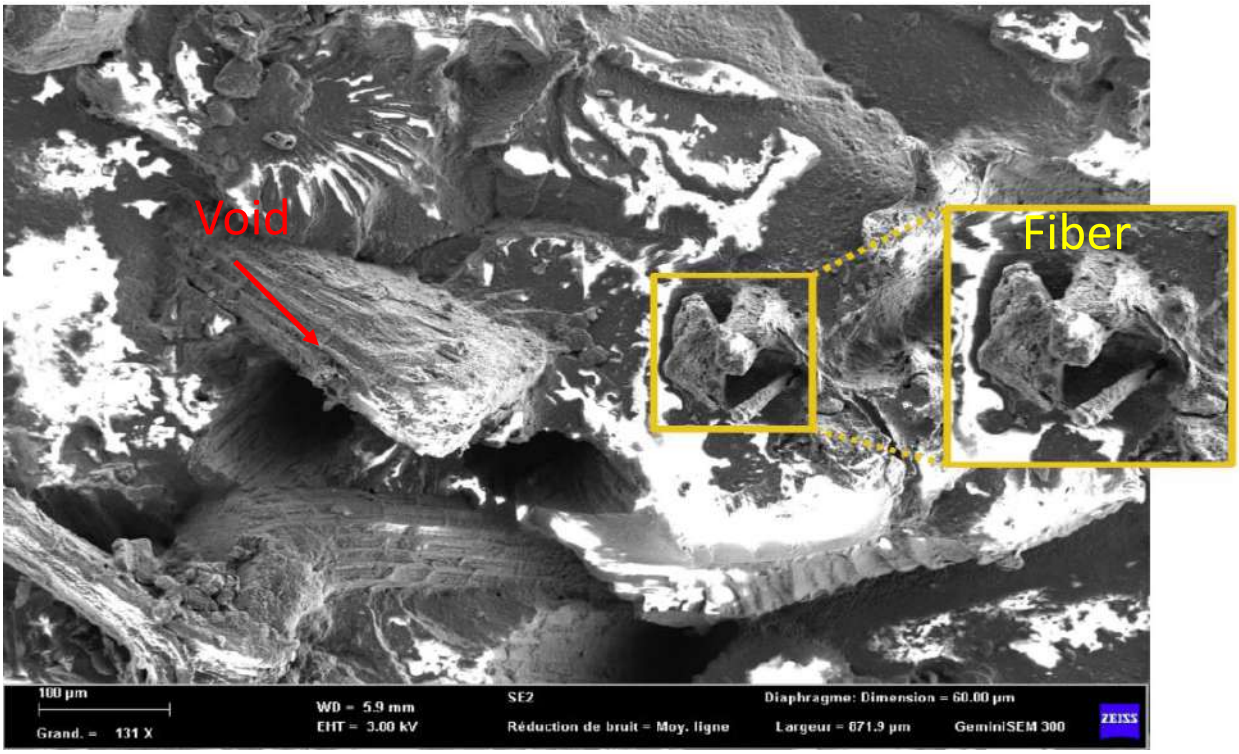

Figure 4.16 – SEM image of epoxy/ treated yucca powder at 3% NaOH.

The SEM analysis of EYN3 bio-composites, incorporating yucca fibers treated with 3 wt.% NaOH, revealed a marked improvement in fiber-matrix adhesion compared to untreated composites, as indicated in **Figure 4.16**. The epoxy resin exhibited more uniform impregnation around the fibers, with a significant reduction in interfacial voids. This enhanced compatibility can be attributed to the partial removal of amorphous components, such as lignin and hemicellulose, which resulted in a rougher fiber surface and improved wettability. Additionally, the fracture surfaces of EYN3 composites displayed a mixed failure mode, characterized by both ductile and brittle features. The presence of stripped fibrils indicated improved load transfer between the fibers and the matrix. However, the detection of a few microcracks suggested that further optimization of the alkaline treatment could further refine the interfacial interaction and mechanical performance [217].

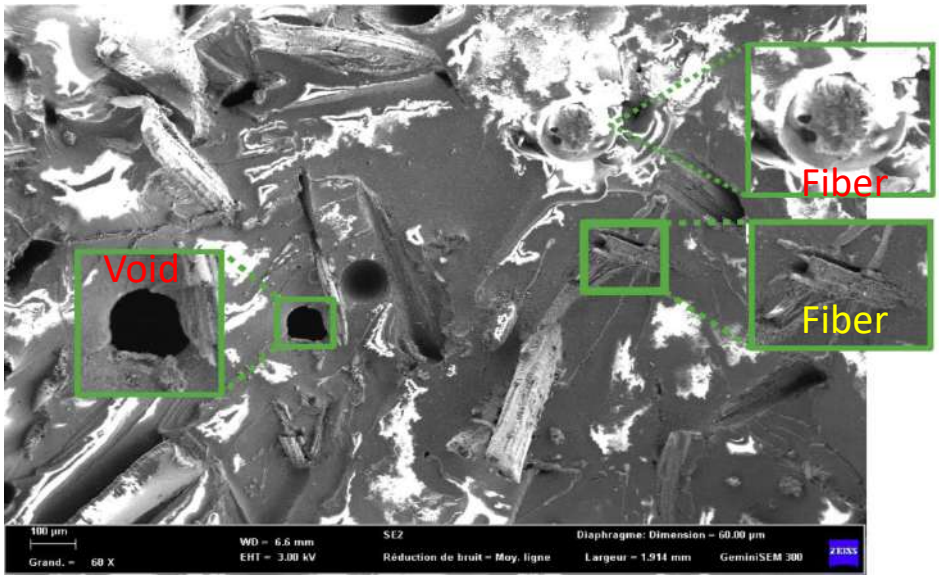

Figure 4.17- SEM image of epoxy/ treated yucca fiber at 1% sulfuric acid bio-composite.

In another context, SEM analysis of EYS1 bio-composites (**Figure 4.17**), incorporating yucca fibers treated with 1 wt.% H_2SO_4, revealed a distinct morphological evolution. The more extensive removal of amorphous components, coupled with the reorganization of cellulose microfibrils, resulted in a significantly rougher fiber surface, thereby enhancing fiber-matrix adhesion. Despite this improved interfacial bonding, the fracture surfaces of EYS1 composites exhibited a tendency toward embrittlement. More pronounced interfacial cracks were observed, and certain fibers appear to detach with minimal deformation, indicating increased composite stiffness. This phenomenon was likely attributable to excessive cellulose crystallization induced by the acid treatment, which reduces the flexibility of the fibers and leads to a more abrupt failure mechanism [218].

The SEM analysis of EYN3 and EYS1 bio-composites underscores the critical role of chemical treatments in modifying the microstructure and interfacial adhesion of yucca fibers. The alkaline treatment (3 wt.% NaOH) enhanced fiber wettability, leading to improved resin impregnation and a more progressive deformation during fracture. In contrast, the acid treatment (1 wt.% H_2SO_4) increased fiber rigidity, reinforcing its anchorage within the epoxy matrix but also inducing a more brittle fracture behavior. These findings highlighted the necessity of selecting the appropriate chemical treatment based on the desired balance between mechanical performance and durability in bio-composites.

4.10.2 Tensile proprieties

The analysis of the mechanical properties of epoxy-based bio-composites reinforced with mechanically extracted yucca fibers highlights the significant influence of chemical treatments on their tensile performance. The results, presented in **Figure 4.18** and **Table 4.8**, indicate a progressive enhancement in mechanical properties depending on the type of treatment applied to the fibers.

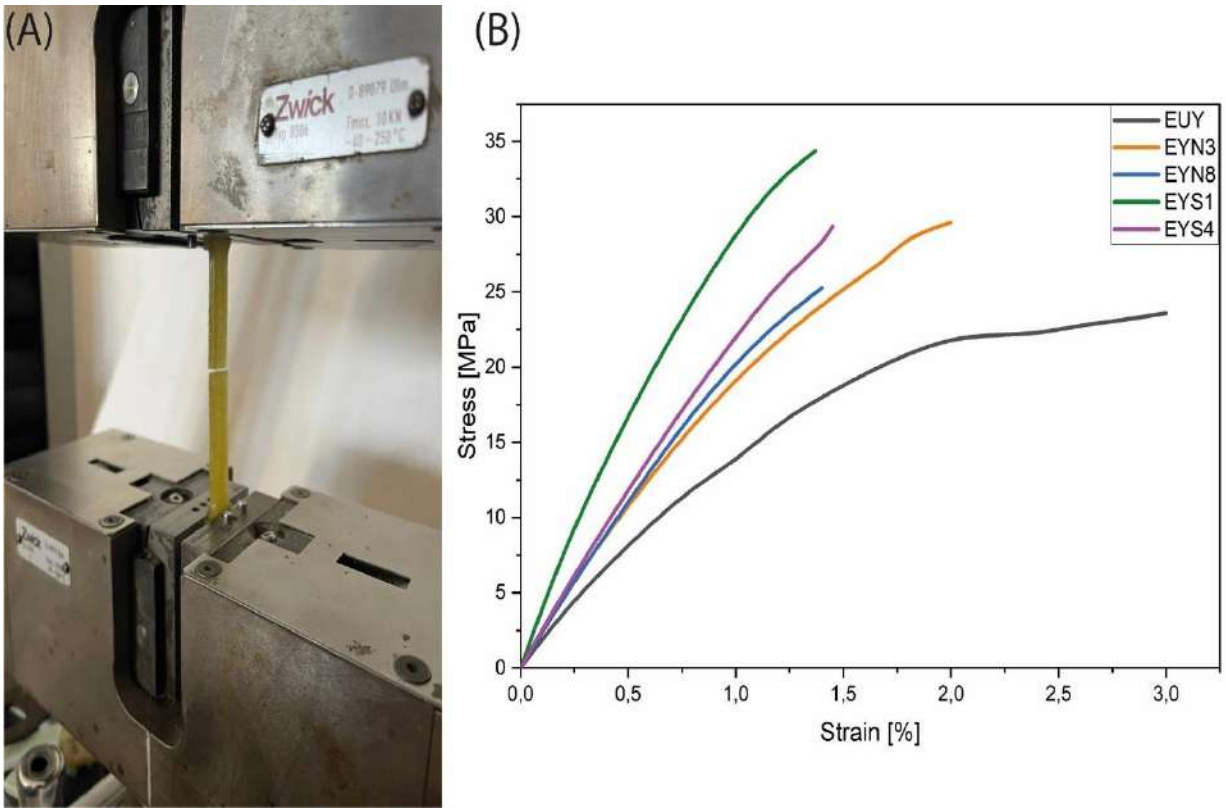

Figure 4.18 – Tensile test results of epoxy/yucca fibers powder bio-composites. (A) Bio-composite sample after the tensile test. (B) the stress strain curve of tensile analysis.

Table 4. 8 – Detailed tensile proprieties of epoxy/yucca fibers powder bio-composites.

Sample type	Tensile strength [MPa]		Young's modulus [MPa]		Elongation [%]	
	Mean	SD	Mean	SD	Mean	SD
EUY	23.59	2.30	1598.12	242.82	3.01	0.75
EYN3	29.63	3.22	2198.48	183.15	2.00	0.21
EYS1	34.37	6.61	2637.08	165.89	1.30	0.06

As a result, the bio-composite reinforced with untreated yucca fibers (EUY) exhibited an average tensile strength of 23.59 MPa, a Young's modulus of 1598.12 MPa, and an elongation at break of 3.01%. These relatively modest values can be attributed to the presence of residual lignin and hemicellulose, which hinder fiber-matrix interaction and reduce the internal cohesion of the composite. Following alkaline treatment, the mechanical performance improved significantly. The tensile strength of the bio-composite incorporating 3% NaOH-treated fibers increased to 29.63 MPa, representing a 25.6% improvement compared to composites with untreated fibers. Additionally, Young's modulus rose to 2198.48 MPa, marking a 37.6% increase, indicating enhanced material rigidity. However, the elongation at break decreased to 2.00%, suggesting reduced ductility due to improved cellulose crystallization and the partial removal of amorphous components [219, 220].

The bio-composite reinforced with sulfuric acid-treated fibers (EYS1) exhibited the highest mechanical performance, with a tensile strength of 34.37 MPa, a 45.8% increase relative to the untreated fiber-reinforced composite. Young's modulus reached 2637.08 MPa, reflecting a 65.1% improvement, indicative of increased stiffness due to fiber structural modifications and improved fiber-matrix interaction. However, the elongation at break further decreased to 1.30%, highlighting a loss of flexibility in favor of a stiffer, stronger structure [221].

These findings confirmed the beneficial impact of chemical treatments on fiber-matrix adhesion and mechanical performance. While alkaline treatment enhances cohesion and provides a balance between stiffness and strength, acid treatment maximizes stiffness and tensile strength at the expense of ductility.

4.10.3 Compressive and bending performance

The assessment of the compressive and bending mechanical properties of bio-composites reinforced with yucca fibers underscores the significant influence of chemical treatments on their mechanical behavior. The results, presented in **Figure 4.19** and **Table 4.9**, provide a comparative analysis of the performance of untreated (EUY) and chemically treated (EYN3 and EYS1) samples, highlighting the impact of fiber modification on the structural integrity and load-bearing capacity of the composites.

Table 4. 9 - Compressive and bending tests results of epoxy/yucca fibers powder bio-composites.

Sample type	Compressive strength [MPa]		Bending strength [MPa]		Bending modulus [MPa]	
	Mean	SD	Mean	SD	Mean	SD
EUY	72.41	0.15	39.34	5.07	1278.28	277.62
EYN3	79.54	2.47	41.03	4.76	1855.36	108.07
EYS1	89.29	1.25	45.27	4.33	1971.66	313.05

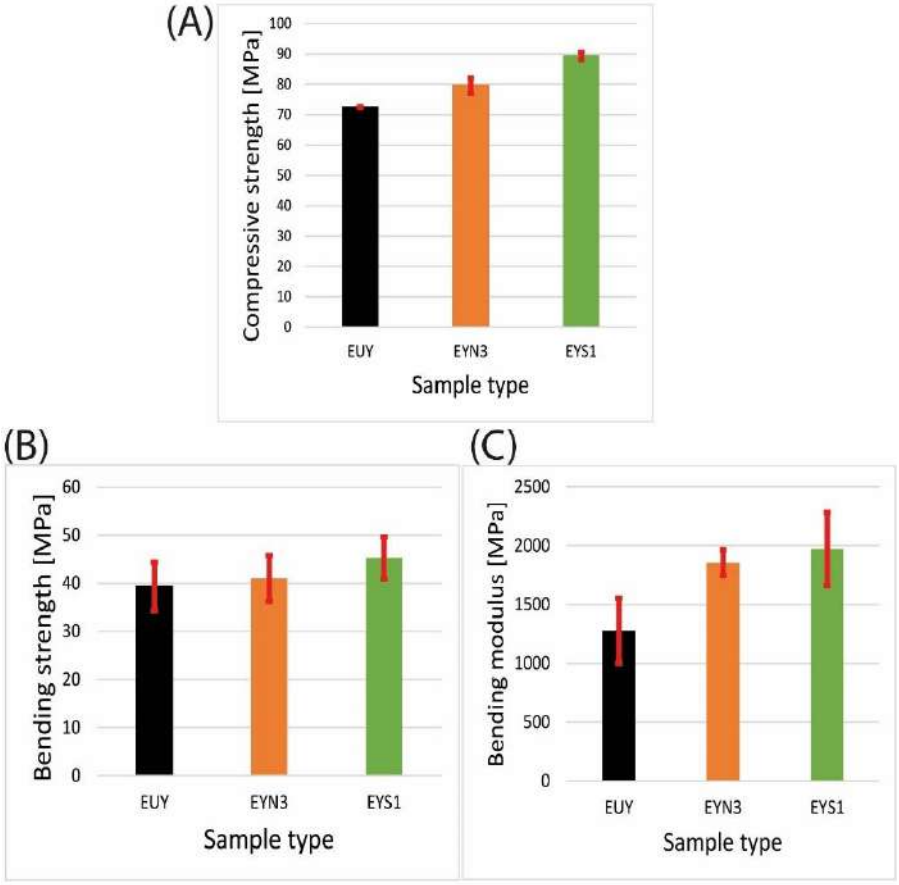

Figure 4.19- Mechanical performance of epoxy/yucca fibers powder bio-composites. (A) The bio-composites compression performance. (C) and (D) are the bending test results on bio-composites.

The compression tests conducted on bio-composites reinforced with yucca fibers revealed significant variations depending on the chemical treatments applied (**Figure 4.19A**). The bio-composites reinforced with untreated yucca fibers (EUY) exhibited an average compressive strength of 72.41 MPa, serving as a reference for evaluating the impact of chemical modifications. In comparison, the EYN3 bio-composites, reinforced with yucca fibers treated with 3% NaOH, displayed an enhanced compressive

strength of 79.54 MPa. This improvement was attributed to the partial removal of lignin and hemicellulose, which increases fiber surface roughness and enhances fiber-matrix adhesion. Furthermore, treatment with sulfuric acid led to a more pronounced enhancement in compressive strength. The EYS1 bio-composites, incorporating yucca fibers treated with 1% sulfuric acid, exhibited the highest compressive strength of 89.29 MPa. This increase was likely due to the selective hydrolysis of amorphous fiber regions, which promotes better alignment of cellulose chains and an increase in crystallinity index. Consequently, the fibers become more rigid, leading to improved fiber-matrix cohesion and superior mechanical performance [222].

Similarly, the bending test results further underscore the influence of chemical treatments on the mechanical behavior of the bio-composites (**Figure 4.19B**). The untreated yucca fiber reinforced bio-composites (EUY) exhibited relatively low mechanical performance, with a bending strength of 39.34 MPa and a bending modulus of 1278.28 MPa. However, chemically treated bio-composites demonstrated substantial improvements, with variations dependent on the type and concentration of the applied treatments.

Notably, the EYN3 bio-composites reinforced with 3% NaOH-treated fibers recorded a bending strength of 41.03 MPa and a bending modulus of 1855.36 MPa. This enhancement was likely due to improved interfacial adhesion between the fibers and the epoxy matrix, facilitated by the removal of surface impurities through alkaline treatment. Meanwhile, the EYS1 bio-composites, incorporating fibers treated with 1% sulfuric acid, achieved the highest bending strength of 45.27 MPa and a bending modulus of 1971.66 MPa. This improvement can be attributed to the enhanced chemical activation of fiber surfaces by sulfuric acid, leading to effective removal of surface impurities and stronger fiber-matrix interactions [223].

This improved adhesion facilitated more efficient stress transfer, thereby enhancing both stiffness and strength while preserving the intrinsic mechanical properties of the fibers. In contrast to certain alkaline treatment conditions, low-concentration acid treatment appears to better preserve the structural integrity of the fibers, optimizing their role as reinforcing agents in bio-composites [224].

4.11 Conclusion

The study of the chemical, thermal and mechanical properties of bio-composites reinforced with yucca fibers highlighted the decisive influence of the fiber extraction methods and the treatments applied, on their final performance. However, from the results of the analyses, the bio-composite manufactured via

3D printing and casting molding exhibited variations in performance depending on the method of fiber extraction, as well as an overall improvement in properties compared to the pure PLA composite. The main conclusions of this study can be summarized as follows:

- The integration of yucca fiber powder alters the overall properties of the material, with its impact varying according to the specific type of yucca fiber incorporated into the bio-composite.
- The concentration of yucca powder in the PLA matrix significantly impacts the adhesion between the reinforcement and the polymer directly affecting the final properties of the bio-composite.
- Thermal analysis confirmed that the addition of yucca fiber powder preserved the thermal stability of PLA, while microstructural analysis revealed a uniform fiber dispersion at low filler content (1%).
- The optimal reinforcement was achieved with 1% yucca fiber powder, significantly enhancing mechanical and thermal properties compared to pure PLA. However, increasing the fiber content to 3% led to a decline in compressive and bending strengths, likely due to fiber agglomeration and weaker interfacial adhesion.
- The bio-composite reinforced with 1% yucca fiber powder extracted using the traditional method exhibited superior mechanical and thermal properties, making it the most performant category in this study. With a tensile strength of approximately 61 MPa, a bending strength of 56 MPa, a compressive strength of 89 MPa, and a Tmax of 394 °C, this formulation enhanced all the properties of the pure PLA composite.
- The fatigue test demonstrated that the bio-composite reinforced with 1% yucca fiber extracted via the traditional method withstood at least 40,185 cycles before failure, highlighting its potential for applications requiring resistance to cyclic loading.
- The application of chemical treatments to yucca fibers significantly influenced the mechanical performance of the bio-composites.
- Chemical treatments significantly enhanced the tensile strength of bio-composites, with a 25.6% increase for EYN3 (3% NaOH) and a 45.8% increase for EYS1 (1% H_2SO_4) compared to the untreated sample (EUY)

General conclusion

General conclusion

This study underscores the potential of yucca fibers as a sustainable reinforcement in polymer matrix bio-composites, particularly in light of global efforts to reduce plastic pollution and replace synthetic fibers. By investigating different extraction and treatment methods, we have demonstrated that both the mechanical and physicochemical properties of yucca fibers can be significantly optimized to enhance their structural applications.

In the first part of this experimental study, we explored different extraction methods for yucca fibers in order to assess their potential as reinforcement in polymer matrix bio-composites. This research is part of an effort to identify yucca fiber as a new alternative to conventional natural fibers and to analyses the influence of extraction processes on its physico-chemical and mechanical properties. The results showed that the extraction method had a significant influence on the mechanical strength of the fibers. The water retting method produced the strongest fibers, with an average tensile strength of 467 MPa and a Young's modulus of 14 GPa. The traditional method produced slightly less resistant fibers, reaching 444.74 MPa in tensile strength. Chemically extracted fibers with a concentration of 3% NaOH showed a significant improvement in Young's modulus (16.78 GPa), while a higher concentration of 10% NaOH resulted in a significant deterioration in mechanical properties, with tensile strength reduced to 235.83 MPa.

Furthermore, targeted chemical treatments significantly modified the mechanical properties of yucca fibers. Alkaline treatment with 3% NaOH resulted in the highest tensile strength (518.74 MPa), marking a 101.2% improvement over untreated fibers. Similarly, acid treatment with 1% H_2SO_4 enhanced the tensile strength to 497.81 MPa, demonstrating the positive impact of chemical modifications on fiber structure. These enhancements were also reflected in elongation at break, which increased from 4.05% in untreated fibers to 5.14% after alkaline treatment.

Once the yucca fibers had been extracted and characterized, the next step was to produce polymer matrix bio-composites using two distinct processes: 3D printing and molding casting. The aim was to assess the influence of the fiber extraction methods on the mechanical properties and the quality of the interface between the fibers and the matrix in each production method. 3D printing has made it possible to structure bio-composites with a controlled architecture, offering great flexibility in the design of the final parts. However, the homogeneous incorporation of yucca fibers into the polymer filament has been a major challenge, due to their irregular dispersion and variations in the viscosity of the composite material.

In addition, the interfacial adhesion between the printed layers showed certain limitations, influencing the overall mechanical strength of the samples. In parallel, the casting molding technique has made it possible to produce bio-composites with better embedding of the fibers in the matrix, thereby reducing the risk of internal defects. However, this process presents constraints linked to the curing time, the shrinkage of the polymer after solidification and the challenges associated with demolding. Fiber-matrix compatibility has remained a key issue, requiring adjustments to formulations and the use of specific surface treatments to improve interfacial adhesion.

The experimental results highlighted the influence of the different yucca fiber extraction methods on the mechanical performance of PLA-Yucca bio-composites. In tensile tests, the highest tensile strength was obtained for sample PLA reinforced with yucca fiber powder extracted via traditional method at 1wt.% concentration (PLA-T1), reaching 61.06 MPa, while stiffness varied slightly according to fiber type, with Young's modulus ranging from 1102 MPa for PLA reinforced with yucca fiber powder extracted via traditional method at 3wt.% concentration (PLA-T3) to 1316 MPa for PLA-T1. However, in bending, the results showed that PLA-T1 also had the highest strength at 56.57 MPa, followed by pure composite PLA at 53.06 MPa, while PLA-T3 had the lowest value. Fatigue performance confirmed these trends, with PLA-T1 demonstrating superior endurance with a number of cycles reaching 4673 at 40% of Smax and 40185 cycles at 20% of Smax, clearly outperforming the other categories.

As part of the assessment of the thermal stability of PLA-based bio-composites, thermogravimetric analysis (TGA) revealed a significant improvement in the maximum degradation temperature (Tmax) after fiber incorporation, compared with pure PLA (385°C). In particular, the PLA-T1 sample has a Tmax of 394°C. Furthermore, the total mass loss at 500°C remains similar to that of pure PLA, indicating that the addition of fibers, regardless of the extraction method, does not significantly affect the overall thermal stability of the composite. However, subtle variations between samples highlight the potential influence of the extraction process on the thermal resistance of bio-composites.

Dynamic mechanical analysis (DMA) was used in this study, to assess the viscoelastic properties of PLA bio-composites reinforced with yucca powder fibers extracted using different methods. The results show that the glass transition temperature (Tg) remains relatively constant, varying between 60.2°C and 61.3°C, indicating that the addition of fibers does not significantly alter the molecular mobility of the polymer matrix. However, the storage modulus showed significant differences depending on the method used to extract the fibers. At 35°C, the PLA-WR1 and PLA-T1 samples show the highest values (4.72 GPa and 4.53 GPa respectively), reflecting increased stiffness compared with pure PLA.

In addition, the results of the mechanical tests highlighted the influence of the incorporation of yucca fibers on the mechanical performance of epoxy matrix bio-composites. Epoxy, known for its rigidity and good resistance to loads, has a tensile strength of 23.70 MPa, a Young's modulus of 2132.36 MPa and an elongation at break limited to 1.33%. In contrast, the addition of yucca fibers slightly reduced the tensile strength (23.59 MPa) and Young's modulus (1598.12 MPa), while significantly increasing ductility, with elongation reaching 3.01%. In compression tests, the bio-composite showed a clear improvement in compressive strength (72.41 MPa) compared with pure epoxy (59.57 MPa).

Otherwise, tensile tests on epoxy-based bio-composites revealed a significant increase in mechanical strength after chemical treatment of yucca fibers. The untreated fiber-reinforced bio-composites sample (EUY) had a tensile strength of 23.59 MPa, while the fiber-reinforced treated with 3% NaOH bio-composite sample (EYN3) reached 29.63 MPa, an improvement of around 25%. Similarly, acid treatment at 1% H_2SO_4 of yucca fiber (EYS1) further optimized this property, with a strength reaching 34.37 MPa, an increase of 46% compared to the untreated fibers. The Young's modulus follows the same trend, rising from 1598.12 MPa for EUY to 2198.48 MPa for EYN3 and reaching 2637.08 MPa for EYS1. However, these improvements were achieved at the expense of elongation at break, which fell after treatment, from 3.01% for EUY to 2.00% for EYN3, and down to 1.30% for EYS1.

Future studies could explore the use of long fibers to improve the mechanical properties and long-term behavior of bio-composites. Exploring new, more environmentally-friendly and efficient methods for extracting and processing fibers would optimize their compatibility with polymer matrices. In addition, the integration of other matrices, such as polypropylene (PP), would open the way to a wider range of industrial applications. Finally, we hope that this study will contribute to the advancement of bio-composites by offering a promising alternative for the development of innovative and sustainable materials. Through exploring the impact of different fiber extraction and treatment methods, as well as their interaction with polymer matrices, our work aims to address the challenges of performance and sustainability. These findings could support the adoption of bio-based composites in diverse industrial sectors, promoting environmentally friendly solutions while maintaining high mechanical integrity and durability.

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Abstract

This study evaluates the impact of different extraction methods on the properties of Algerian yucca fibers and their incorporation into bio-composites, aiming to develop eco-friendly materials. Four extraction methods were compared: mechanical extraction, water retting, the traditional method, and chemical extractions with NaOH (3 %, 5 %, and 10 %). A complete characterization of the fibers and corresponding composites was conducted to assess structural, mechanical, and physicochemical features. Water-retted yucca fibers exhibited the highest tensile strength (467 MPa) and Young's modulus (14 GPa). Similarly, fibers extracted via 3 % NaOH showed excellent performance (456 MPa, 16.78 GPa). In contrast, 10 % NaOH severely degraded fiber structure, lowering tensile strength to 235 MPa and crystallinity to 61.75 %. Furthermore, two composite processing techniques were explored: 3D printing (FFF) using PLA-yucca filaments, and molding casting with epoxy-yucca powder. Mechanical tests showed that PLA-T1 (with traditionally extracted fibers) exhibited the best performance (61.06 MPa tensile strength, 488.4 MPa flexural modulus). SEM revealed strong fiber/matrix adhesion. FTIR, TGA, and XRD analyses confirmed the influence of extraction method on thermal and chemical properties. Lastly, fatigue and Charpy impact tests demonstrated the composites' durability and toughness.

Keywords: Natural fibers; Extraction method; Biocomposites; Sustainability; Ecofriendly; Performance.

Résumé

Cette étude examine l'impact de différentes méthodes d'extraction sur les propriétés des fibres de yucca algérien et leur intégration dans des biocomposites, en vue de développer des matériaux écologiques. Quatre méthodes ont été comparées : extraction mécanique, rouissage à l'eau, méthode traditionnelle et traitement chimique au NaOH (3 %, 5 %, 10 %). Une caractérisation complète a été effectuée sur les fibres et les composites associés, en évaluant leurs propriétés mécaniques, structurales et physico-chimiques. Les fibres obtenues par rouissage à l'eau ont montré les meilleures performances mécaniques (467 MPa, 14 GPa), suivies de celles extraites avec 3 % de NaOH (456 MPa, 16,78 GPa). En revanche, l'utilisation de 10 % de NaOH a fortement dégradé la structure des fibres, réduisant la résistance à la traction à 235 MPa et la cristallinité à 61,75 %. Deux procédés de fabrication de composites ont été testés : l'impression 3D (FFF) avec des filaments PLA-yucca, et le moulage-coulée avec une matrice époxy et de la poudre de yucca. Les meilleurs résultats mécaniques ont été obtenus avec le composite PLA-T1 (fibres traditionnelles). Les analyses MEB, FTIR, TGA et DRX ont confirmé l'influence des méthodes d'extraction sur les propriétés des composites. Des tests de fatigue et d'impact ont mis en évidence leur durabilité.

Mots-clés : Fibres naturelles ; Méthodes d'extraction ; Biocomposites ; Performances mécaniques ; Matériaux écologiques ; Durabilité.

الملخص

تتناول هذه الدراسة تأثير طرق الاستخلاص المختلفة على خصائص ألياف الطبيعية ودمجها في المواد الحيوية المركبة، بهدف تطوير مواد صديقة للبيئة. تم مقارنة أربع طرق للاستخلاص: الاستخلاص الميكانيكي، والبيولوجي، والطريقة التقليدية، والكيميائية بهيدروكسيد الصوديوم (NaOH) بنسب تركيز 3%، 5%، و10%. تم إجراء توصيف شامل للألياف والمواد المركبة المرتبطة بها، من خلال تقييم الخصائص الميكانيكية والبنيوية والفيزيائية-الكيميائية. أظهرت الألياف الناتجة عن التعطين بالماء أفضل أداء ميكانيكي (467 ميغاباسكال، و14 غيغاباسكال)، تليها الألياف المستخلصة باستخدام NaOH بتركيز 3% (456 ميغاباسكال، و16.78 غيغاباسكال). في المقابل، أدى استخدام 10% من NaOH إلى تدهور كبير في بنية الألياف، مما خفّض مقاومة الشد إلى 235 ميغاباسكال ودرجة التبلور إلى 61.75%. تم اختبار طريقتين لتصنيع المواد المركبة: الطباعة ثلاثية الأبعاد (FFF) باستخدام خيوط PLA واليوكا، والقولبة الصبّية باستخدام مصفوفة إيبوكسي ومسحوق اليوكا. سُجلت أفضل النتائج الميكانيكية لدى العينة PLA-T1

الكلمات المفتاحية :الألياف الطبيعية؛ طرق الاستخلاص؛ المواد الحيوية المركبة؛ الأداء الميكانيكي؛ المواد الصديقة للبيئة؛ الاستدامة.