Mohamed Boudiaf University - M’sila

FACULTY OF TECHNOLOGY

MECHANICAL ENGINEERING DEPARTMENT

Serial number:

Registration number: **D.GM/3C/09/21**

Thesis

Presented for obtaining the diploma of

L.M.D Doctorate

Specialty: Mechanical Engineering

Option: Material Engineering

THEME

Study the influence of growth conditions on the mechanical behavior of unidirectional and woven plant fibers polyester composites

Submitted by

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Defended on:16/12/2025

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Academic year: 2025 / 2026

Dedication

I dedicate this thesis to:

My extended family, whose continuous support and encouragement have accompanied me through every stage of my life.

To my father, the pillar and foundation of my strength.

To my dear mother, the source of boundless kindness and generosity, who taught me that determination makes the impossible attainable.

To my sister Souad, my support and companion on this journey, and a symbol of patience and wisdom.

To my brothers and sisters, who shared with me moments of challenge and joy.

To my small family: my supportive husband and life partner, and my children, whose love and enthusiasm inspired in me the perseverance and dedication to move forward.

And to all those who believed in my abilities and walked beside me with sincerity throughout this journey of learning and discovery,

I dedicate the fruits of years of work and effort.

Acknowledgements

I would like to begin by expressing my sincere gratitude to Allah for granting me the strength and perseverance to complete this dissertation.

I also extend my sincere thanks to my supervisor, **Professor Ben Hamida Mohamed**, for his support, encouragement, and valuable scientific guidance.

My deepest appreciation goes to my co-supervisor, **Professor Rokbi Mansour**, for his constant guidance, scientific expertise, and unwavering support throughout this research. His dedication, availability, and insightful advice were instrumental in the successful completion of this dissertation.

I wish to thank the members of the examination committee for accepting to evaluate this dissertation and for their constructive remarks, which will undoubtedly enrich the quality of this work:

Professor Rahmouni Zine El Abidine (University of M'sila), Chair of the committee, for his valuable expertise and support.

Professor Khaldi Abdelghani (University of BBA), for the honor of participating in the committee and for his constructive engagement.

Dr. Khelladi M. Reda (University of BBA), for his careful evaluation and valuable contribution.

Professor Farsi Chouki (University of M'sila), for his participation and insightful remarks.

My thanks are also extended to all those who supported this research at the University of M'sila, particularly the Heads of the Research Laboratories of the Faculty of Technology, as well as the Chemistry, Physics, and Biology Laboratory. I also express my gratitude to the Polymeric Materials Laboratory at the University of Béjaïa, especially Professor Dalila Hammiche, for providing essential facilities and support.

I am sincerely grateful to my colleagues and friends for their encouragement and moral support throughout this journey.

Finally, I extend my appreciation to all individuals and institutions who contributed, directly or indirectly, to the completion of this work.

Abstract

The aim of this thesis is to contribute to the advancement of scientific research on understanding the factors that influence the overall performance of plant fibers, with the objective of improving their properties and enhancing their functional behavior. In this context, a comprehensive study was carried out to evaluate the suitability of Alfa fibers (Stipa tenacissima L) as reinforcement in composite materials. The study investigated the effects of seasonal variations, and growth sites on the physical, chemical, thermal, morphological, and mechanical properties of fibers. Samples of Alfa fibers were collected from five different regions in Algeria throughout the four seasons of the year. Various analyses were performed, including chemical composition, thermal (TGA-DTG), structural (XRD), spectroscopic (ATR-FTIR), and morphological characterization using scanning electron microscopy (SEM). Additionally, the physical and mechanical properties of the fibers were evaluated. To improve fiber performance and enhance compatibility with the polymer matrix, Alfa fibers from the M'sila region were selected and chemically treated using sodium hydroxide and potassium permanganate. The structural, physicochemical, and morphological characteristics of the fibers were assessed, while the interfacial shear strength (IFSS) between treated and untreated fibers and the polyester matrix was determined through micro-bond test. Weibull statistical analysis was applied to evaluate the variability of IFSS values and to highlight the influence of chemical treatment on fiber structure and surface behavior. Furthermore, intra-ply woven fabrics combining treated and untreated Alfa fibers with jute yarns were developed to investigate the effect of fiber orientation within the matrix. Based on these hybrid fabrics, as well as unidirectional ply of Alfa fibers, polyester-based composite materials were fabricated, and their physical, mechanical, and morphological properties were analyzed. The results revealed a significant enhancement in the mechanical performance of the composite materials following chemical treatment, along with improved interfacial adhesion between the fibers and the polymer matrix. The findings also confirmed the strong influence of environmental and climatic factors on the chemical, structural, physical, and mechanical characteristics of Alfa fibers, providing valuable insights for determining the optimal harvesting period to achieve the desired properties and for guiding their targeted use in advanced industrial and engineering applications. Overall, this study highlights the great potential of Alfa fibers as a sustainable and efficient reinforcement material for high performance composites and contributes to a deeper understanding of local lignocellulosic fibers in green material development

Keywords: Stipa tenacissima L, Growth conditions, Chemical treatment, Fiber orientation Mechanical properties, Chemical composition.

ملخص

يهدف هذا البحث إلى الإسهام في تطوير المعرفة العلمية المتعلقة بفهم العوامل المؤثرة في الأداء العام للألياف النباتية، وذلك بهدف تحسين خصائصها وتعزيز سلوكها الوظيفي. وفي هذا الإطار، أُجريت دراسة شاملة لتقييم مدى ملاءمة ألياف الحلفاء (*Stipa tenacissima L.*) كعامل تدعيم في المواد المركبة.

تم خلال هذه الدراسة تحليل تأثير التغيرات الموسمية ومواقع النمو على الخصائص الفيزيائية والكيميائية والحرارية والمورفولوجية والميكانيكية لهذه الألياف. وقد جُمعت عينات من خمس مناطق مختلفة في الجزائر عبر الفصول الأربعة من السنة. وشملت التحاليل المنجزة كلًّا من التركيب الكيميائي، والتحليل الحراري (TGA-DTG)، والتحليل البنيوي(XRD) ، والتحليل الطيفي(ATR-FTIR) ، إضافة إلى الوصف المورفولوجي باستخدام المجهر الإلكتروني الماسح .(SEM) كما جرى تقييم الخصائص الفيزيائية والميكانيكية للألياف.

ولتحسين أداء الألياف وتعزيز توافقها مع المصفوفة البوليمرية، تم اختيار ألياف الحلفاء من منطقة المسيلة وإخضاعها لمعالجة كيميائية باستخدام هيدروكسيد الصوديوم وبرمنغنات البوتاسيوم. وقد تم تقييم خصائصها البنيوية والفيزيوكيميائية والمورفولوجية، إلى جانب تحديد قوة القص البينية (IFSS) بين الألياف المعالجة وغير المعالجة والمصفوفة البوليسترية باستخدام اختبار الميكروبوند .(Microbond Test) كما استُخدم تحليل وايبول الإحصائي لتقييم تباين قيم IFSS وإبراز تأثير المعالجة الكيميائية على بنية الألياف وسلوك سطحها.

بالإضافة إلى ذلك، تم تطوير أقمشة هجينة ضمن طبقات نسيجية تجمع بين ألياف الحلفاء المعالجة وغير المعالجة مع خيوط الجوت، بهدف دراسة تأثير اتجاه الألياف داخل المصفوفة. وبالاعتماد على هذه الأقمشة الهجينة، وكذلك على الطبقات أحادية الاتجاه من ألياف الحلفاء، تم تصنيع مواد مركبة أساسها بوليستر، ثم تحليل خصائصها الفيزيائية والميكانيكية والمورفولوجية.

أظهرت النتائج تحسنًا ملحوظًا في الأداء الميكانيكي للمواد المركبة بعد المعالجة الكيميائية، إضافة إلى تحسن واضح في الالتصاق البيني بين الألياف والمصفوفة البوليمرية. كما أكدت النتائج التأثير الكبير للعوامل البيئية والمناخية على الخصائص الكيميائية والبنيوية والفيزيائية والميكانيكية لألياف الحلفاء، مما يوفر رؤى علمية قيّمة لتحديد فترة الحصاد المثلى للحصول على الخصائص المطلوبة، وتوجيه استخدامها في التطبيقات الصناعية والهندسية المتقدمة. بصفة عامة، تُبرز هذه الدراسة الإمكانات الكبيرة لألياف الحلفاء كمادة تدعيم مستدامة وفعّالة لإنتاج مواد مركبة عالية الأداء، وتُسهم في تعميق الفهم المتعلق بالألياف اللجنوسليلوزية المحلية في تطوير المواد الصديقة للبيئة.

الكلمات المفتاحية:*Stipa tenacissima L* ، ظروف النمو، المعالجة الكيميائية، اتجاه الألياف، الخصائص الميكانيكية، التركيب الكيميائي.

Résumé

L'objectif de cette thèse est de contribuer à l'avancement de la recherche scientifique portant sur la compréhension des facteurs influençant les performances globales des fibres végétales, dans le but d'améliorer leurs propriétés et d'optimiser leur comportement fonctionnel. Dans ce contexte, une étude approfondie a été réalisée afin d'évaluer l'aptitude des fibres d'Alfa (Stipa tenacissima L) à être utilisées comme renfort dans les matériaux composites. L'étude a porté sur l'effet des variations saisonnières et des sites de croissance sur les propriétés physiques, chimiques, thermiques, morphologiques et mécaniques des fibres. Des échantillons d'Alfa ont été prélevés dans cinq régions différentes d'Algérie au cours des quatre saisons de l'année. Diverses analyses ont été menées, notamment la composition chimique, les analyses thermiques (TGA-DTG), structurales (XRD), spectroscopiques (ATR-FTIR), ainsi que la caractérisation morphologique par microscopie électronique à balayage (SEM). Les propriétés physiques et mécaniques des fibres ont également été évaluées.

Afin d'améliorer les performances des fibres et leur compatibilité avec la matrice polymère, les fibres d'alfa provenant de la région de M'sila ont été sélectionnées et soumises à des traitements chimiques à l'hydroxyde de sodium et au permanganate de potassium. Les caractéristiques structurales, physicochimiques et morphologiques des fibres ont été étudiées, tandis que la résistance au cisaillement interfacial (IFSS) entre les fibres traitées et non traitées et la matrice polyester a été déterminée par essai microbande. L'analyse statistique de Weibull a été appliquée pour évaluer la variabilité des valeurs d'IFSS et pour mettre en évidence l'influence du traitement chimique sur la structure et le comportement de surface des fibres. En outre, des tissus hybrides intra-plis combinant des fibres d'Alfa traitées et non traitées avec des fils de jute ont été développés afin d'étudier l'effet de l'orientation des fibres dans la matrice. À partir de ces tissus hybrides, ainsi que de plis unidirectionnels de fibres d'Alfa, des composites à base de polyester ont été élaborés, et leurs propriétés physiques, mécaniques et morphologiques ont été analysées.

Les résultats ont révélé une amélioration significative des performances mécaniques des matériaux composites après traitement chimique, ainsi qu'une meilleure adhésion interfaciale entre les fibres et la matrice polymère. Les résultats ont également confirmé l'influence marquée des facteurs environnementaux et climatiques sur les caractéristiques chimiques, structurales, physiques et mécaniques des fibres d'Alfa, fournissant ainsi des indications précieuses pour déterminer la période de récolte optimale et orienter leur utilisation dans des applications industrielles et technologiques avancées. Dans l'ensemble, cette étude met en évidence le fort

Résumé

Potentiel des fibres d'Alfa en tant que renfort durable et performant pour le développement de composites à hautes performances, et contribue à une meilleure compréhension des fibres lignocellulosiques locales dans le domaine des matériaux verts.

Mots-clés : Stipa tenacissima L, conditions de croissance, traitement chimique, orientation des fibres, propriétés mécaniques, composition chimique.

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Abbreviations

ATW: Alfa fibers harvested in Tiaret during Winter

ATSP: Alfa fibers harvested in Tiaret during Spring

ATS: Alfa fibers harvested in Tiaret during Summer

ATF: Alfa fibers harvested in Tiaret during Autumn (Fall)

ALW: Alfa fibers harvested in Laghouat during Winter

ALSP: Alfa fibers harvested in Laghouat during Spring

ALS: Alfa fibers harvested in Laghouat during Summer

ALF: Alfa fibers harvested in Laghouat during Autumn (Fall)

ADW: Alfa fibers harvested in Djelfa during Winter

ADSP: Alfa fibers harvested in Djelfa during Spring

ADS: Alfa fibers harvested in Djelfa during Summer

ADF: Alfa fibers harvested in Djelfa during Autumn (Fall)

ABW: Alfa fibers harvested in Boussaada during Winter

ABSP: Alfa fibers harvested in Boussaada during Spring

ABS: Alfa fibers harvested in Boussaada during Summer

ABF: Alfa fibers harvested in Boussaada during Autumn (Fall)

AMW: Alfa fibers harvested in Maadid during Winter

AMSP: Alfa fibers harvested in Maadid during Spring

AMS: Alfa fibers harvested in Maadid during Summer

AMF: Alfa fibers harvested in Maadid during Autumn (Fall)

AF: Alfa fiber

A_N: Alkali-treated Alfa fibers

A_p: Permanganate-treated Alfa fibers

ASTM: American Society for Testing and Materials

ATR-FTIR: Attenuated Total Reflectance -Fourier Transform Infrared Spectroscopy

A_{Un}: Untreated Alfa fibers

Comp-A: Intra-ply woven fabric (A_{Un} / J) / UP

Comp-B: Intra-ply woven fabric (A_N / J) / UP

Comp-C: Intra-ply woven fabric (A_P / J) / UP

Comp-D: Woven fabric (J / J) / UP

DTG: Derivative Thermogravimetric

IFSS: Interfacial Shear Strength

ISO: International Organization for Standardization

NASA/POWER: NASA Prediction of Worldwide Energy Resources (POWER) Project

SEM: Scanning Electron Microscopy

TGA: Thermogravimetric Analysis

UP: Unsaturated Polyester

Un-Comp: Unidirectional Alfa ply / UP

XRD: X-ray Diffraction

Notations

CN/tex: Specific tensile strength (centinewton per tex)

CrI: Crystallinity index (%)

D_e: Equivalent mean fiber diameter (µm)

D_f: Mean diameter of the Alfa fiber

D_{AF}: Nominal Alfa fiber diameter (cm)

Ea: Activation energy

***E_f*:** Flexural modulus of elasticity (GPa)

***ε_f*:** Flexural strain (%)

***F*:** Breaking load (N)

***F_{max}*:** Highest pull-out load

***$F(\sigma)$*:** probability of fiber failure

***I_{002}*:** Crystalline peak intensity

***I_{am}*:** Amorphous phase intensity

K: Constant

***L_f*:** Fiber length(m)

***L_{ref}*:** Reference length

***L_s*:** Support span length(mm)

m: Weibull modulus

m_s: Slope of the linear elastic region of the load-deflection curve (N/mm)

L_0: Initial length

L_e: Length of embedded fiber in droplet

***M*:** Fiber mass (mg)

m_0: Initial weight of Alfa fibers (g

m_R: Weight of residue (g)

M_d: Weight of dry sample (g)

m_f: Final weight of Alfa fibers (g)

MC: Moisture content (%)

MW: Molecular weight (g/mol)

R: Universal gas constant (8.314 J/mol·K)

S_f: Mean cross-sectional fiber area (mm^2)

σ: Tensile strength (MPa)

$σ_f$: Flexural strength at fracture (MPa)

T: Absolute temperature (K)

Tex: Unit of linear density (g/km)

τ: Interfacial shear strength

***w*:** Maximum mid-span deflection (mm)

w0: Initial mass

wt: Residual mass at time t

W_f: Fiber weight fraction

W_m: Matrix weight fraction

$ρ_{AF}$: Apparent density of Alfa fibers (g/cm^3)

$ρ_f$: Fiber density

$ρ_m$: Matrix density

$ρ_M$: Methanol density (0.791 g/cm^3 at 25°C)

Δm1: Mass difference between the pycnometer containing chopped fibers and the empty one

Δm2: Mass difference between the pycnometer filled with methanol and the empty pycnometer

Δm3: Mass difference between the pycnometer containing both methanol and chopped fibers and the one filled with methanol only

General introduction

General introduction

In the context of growing environmental challenges, industries and academic institutions seek sustainable alternatives by utilizing various natural fibers in several industrial applications [1]. Natural fibers, with their specific properties and ability to regenerate, appear to be an attractive option for building a more sustainable future, contributing to reducing carbon emissions and conserving natural resources, Furthermore, it is an invaluable solution to the increasing demand for environmentally friendly products in various industries [2].

The use of natural fibers has recently attracted significant attention from researchers due to their good mechanical, thermal, and environmental properties. In addition, some features such as versatility, abundance, low cost, ease of processing, non-toxicity, reduced respiratory irritation, and non-corrosive nature compared to synthetic fibers[3]. Furthermore, manufacturing natural fibers consumes 17% less energy than synthetic fibers[2].

Until now, plant fibers such as hemp, flax, bamboo, jute, kenaf, sisal, Ramie, coir, alfa, and palm offer promising raw materials that can be used within the domain of lightweight polymer composites. For several years, natural fiber has been used in textiles and paper production (papermaking, packaging) to decrease plastic consumption. Moreover, lignocellulosic fiber has been used in the handicraft industry, agriculture, medicine, and renewable energy[4]. Beyond its traditional applications, natural fiber is now being extensively integrated as reinforcement for polymer composite materials, significantly enhancing their mechanical properties, where it enters into the manufacturing of lightweight parts for cars and aircraft, such as interior components, door panels, trunk liners, sandwich panels, bonnets, and boot lids[5]. Additionally, it is being incorporated into civil engineering such as building insulation, ceilings, walls, insulation panels, and roofing. [6]. The United States Department of Energy intends to produce 50% of the fundamental chemical building blocks derived from plants by 2050[7].

The properties of natural fibers are significantly influenced by various factors that, in turn, affect the performance of fiber-reinforced composite materials. These factors include climatic conditions (such as temperature, light, humidity, rainfall, and wind), also referred to as seasonal conditions, and soil quality (such as fertility, aeration, pH, and organic matter), which are associated with growth areas [8-10]. Furthermore, the time harvesting, the growth stage, the maturity of the plant, and the extraction method can have a significant impact on the physicomechanical and chemical properties of natural fibers[9, 11, 12]. Therefore, it is essential to consider these factors, especially the seasonal variation and growth areas of plants. When

selecting suitable plant fibers to ensure the desired performance in composite materials.

The investigation of the effect of seasonal variations and the selection of plant fiber collection sites on the lignocellulosic fiber performances have attracted the interest of many researchers. Tarchi, et al. [13] show that seasonal change plays a crucial role in influencing the chemical composition and the quality of Alfa fibers. Their results reveal that autumn exhibited the highest levels content of ash (2.46%) and cellulose (42.8%). Similarly, Belkhir, et al. [14] investigated the effect of soil composition, geographic location, and bioclimatic conditions on the chemical composition of Stipa tenacissima fibers from four Tunisian sites. Their results showed that fibers from the semi-arid Zulfan site had the highest cellulose content (46.6%), while other sites like Salloum showed lower cellulose (44.2%) but higher lignin and extractives. Seasonal variation also influenced cellulose levels, with the lowest values in winter and a gradual increase toward autumn, reflecting plant growth cycles and environmental factors such as temperature and rainfall Also, Mehdadi, et al. [15] studied how seasonal changes affect the biochemical composition of Stipa tenacissima and its adaptive responses. Their results confirm that during spring, favorable growth conditions promote the accumulation of pectin, hemicelluloses, and essential nutrients such as nitrogen, magnesium, and phosphorus, which are vital for growth and reproduction. In contrast, under harsh summer conditions, characterized by high temperatures and water scarcity, the plant exhibits increased levels of lignin, cellulose, potassium, and copper. Correspondingly, The research carried out by Liu, et al. [12] indicates that the maturity level of hemp plants and the harvest time have a significant impact on the mechanical properties and chemical composition of the extracted fibers. It was found that fibers harvested at seed maturity or late harvest (Sep 2013) are coarser and exhibit lower mechanical quality due to reduced cellulose deposition and an increase in secondary fibers. Conversely, fibers collected at the flowering onset or early harvest (July 2013) are more suitable to reinforce composites with high performances.

Among the existing plants in the semi-arid Mediterranean ecosystems of Algeria, Alfa grass (Stipa tenacissima) stands out as one of the dominant species [16]. This plant has attracted significant attention from researchers due to its remarkable properties. The extracted fibers from this plant are characterized by high tensile strength and elevated Young's modulus values, making them a promising material for various engineering applications [17]. Furthermore, this natural resource is available throughout the year in varying quantities, thereby promoting its sustainable utilization.

Several researchers have studied the properties of Alfa fibers, analyzing their morphological structural and physicomechanical, and thermal characteristics to better understand their potential as

reinforcement material composites. Hammiche, et al. [18] studied the physicochemical, thermal, and mechanical properties of Alfa fibers. Their findings revealed that the fibers exhibit high tensile strength and significant Young's modulus values. Additionally, Paiva, et al. [19] examined the tensile characteristics of Alfa fiber and found that individual filament tests yield approximately 20 GPa for tensile modulus and 250 MPa for yield strength. Brahim's study [20] not only highlights the favorable properties of Alfa fibers but also positions them as a viable alternative to synthetic fibers. Moreover, Khaldi, et al. [21] focused on the impact of relative humidity and temperature on the mechanical properties of Alfa fibers. Their findings reveal that thermal cycles at 200°C do not affect the mechanical properties. However, variations in relative humidity levels significantly influence the fibers' mechanical behavior, as the breaking strain increases with higher moisture content.

Various studies have investigated the chemical composition of Stipa tenacissima grass [1, 14, 15, 18, 22], revealing that cellulose is the predominant component, with typical levels ranging from 33% to 49%. This high cellulose content significantly enhances the tensile strength and stiffness of Alfa fibers, making them highly suitable for applications requiring durable materials [16], alongside cellulose, Alfa fibers also contain 20–39% hemicellulose, which enhances their flexibility and moisture absorption capabilities, and 15-25% lignin, which provides rigidity, thermal stability, and hydrophobicity essential for composite applications[23, 24]. Ayadi, et al. [25] analyzed the chemical composition of Posidonia, Alfa, and hemp fibers, finding that Alfa's cellulose content (47.4%) is higher than Posidonia's (45.3%) but lower than hemp's (62.8%). Posidonia showed a high lignin content (31.5%), affecting its structural and morphological properties. Variations in ash content among the fibers were attributed to differences in the mineral composition of their growth environments. These findings underscore the influence of chemical composition on the mechanical, thermal, and morphological behavior of natural fiber.

Several other factors greatly affect the properties of Alfa fibers, most notably the various chemical treatments and the fiber orientation, which can range from random, unidirectional, and bidirectional arrangements to more complex configurations such as hybrid structures, including intra-ply and inter-ply hybrid material composites.In this context, Amrane, et al. [26] indicates that Alfa fibers exhibit significant mechanical properties, which are influenced by factors such as fiber orientation, treatment methods, and porosity. Their studies reveal that optimal processing conditions enhance both tensile strength and rigidity. Accordingly, Messaoui, et al. [27]reported that treating Alfa fibers with 3% NaOH and compatibilizers enhanced the tensile strength of polypropylene composites due to improved fiber-matrix adhesion. Similarly, Rokbi and Osmani [28]demonstrated that treating Alfa fibers with 10% NaOH for 24 hours significantly improved the flexural strength

and modulus of the composites by approximately 60% and 62%, respectively. Additionally, Madhu, et al. [29]concluded that various chemical treatments applied to Americana agave fibers such as sodium hydroxide, stearic acid, benzoyl peroxide, and potassium permanganate led to a notable improvement in both tensile strength and elongation at break across all treated samples. In addition to chemical treatments, the structural configuration and orientation of the reinforcing fibers play a critical role in determining the mechanical performance of natural fiber composites. This is clearly demonstrated by the fact that continuous fibers aligned parallel with the load direction exhibit superior strength compared to randomly oriented ones. However, Jamshaid, et al. [30]reported that woven fabric composites offer more balanced mechanical properties than unidirectional laminates. This is due to the interlocking weave structure, which restricts fiber pull-out and enhances overall strength. Goutianos, et al. [31]also found that continuous textile reinforcements in natural fiber composites yield mechanical properties three to four times higher than those of non-woven flax composites.

Hybridization has emerged as an effective strategy to enhance the mechanical properties of natural fiber composites. For instance, Dr.C.Senthamaraikannan1 [32]reported that intra-ply hybrid composites of jute/sisal and flax/sisal exhibited higher tensile strength than their single-fiber counterparts. Another study by the findings of Alavudeen, et al. [33]demonstrated that interwoven banana/Kenaf hybrid composites outperform individual fibers in tensile, flexural, and impact strength due to enhanced fiber–matrix adhesion resulting from the hybrid structure. As well, the study of Bouchareb, et al. [34] found that intra-ply hybrid composites reinforced with intra-ply Alfa / Jute woven fabrics give acceptable tensile and flexural values compared to intra-ply Jute/ Ramie and Jute/Banana woven fabric composites.

Despite the diligent efforts of researchers to thoroughly investigate the factors influencing the performance of Alfa fibers as reinforcement materials composites, all the researchers on the works cited above[1, 15-17, 20, 25-34] as well as in other investigations [35-37]aiming at the physicochemical and mechanical and even thermal and morphological characterizations have not taken into consideration the effect of the seasonal changes and growth sites on the various properties of Algerian Alfa fibers. To address this knowledge gap, an attempt has been done to study the influence of growth conditions on selected Algerian Alfa fiber properties.

Our current work is composed of three chapters.

The first chapter (Literature Review) is devoted to the study of composite materials in terms of their importance and components, with an overview of their classification principles, manufacturing methods, influencing factors, and practical applications. It also addresses plant

fibers as a promising alternative to synthetic fibers due to their outstanding properties, highlighting their classification, morphological structure, chemical composition, extraction methods, as well as their thermal and mechanical characteristics. Furthermore, the chapter reviews studies focused on improving their performance through chemical surface treatments and fiber orientation within the matrix. The chapter concludes with an analysis of the impact of environmental and seasonal climatic factors, along with the pre-harvest stage, on fiber properties, emphasizing the scarcity of research in this field and the need for further in-depth investigation in the future.

The second chapter is one of the most scientifically rich sections, as it provides a comprehensive overview of the materials used and the methods adopted in carrying out this research work. Due to the diversity of topics covered in this chapter, it has been divided into two main parts to improve clarity and the organization of the data.

The first part is devoted to presenting the materials used and their physical and chemical characteristics. The chapter begins with a detailed description of the collection sites of Stipa tenacissima (Alfa grass) from five different regions of Algeria, sampled across the four seasons of the year, and explains the rationale behind selecting these sites and their significance within the scope of the study. This section also provides a detailed presentation of the extraction methods of Alfa fibers, based on systematic protocols and a variety of tools and techniques employed to carry out the subsequent analyses and characterizations, which include the anatomical analysis of the plant stem, chemical characterization, structural and thermal analyses, in addition to the physical and mechanical analyses of all fibers collected from the five regions throughout the four seasons.

The second part is focused on the chemical treatment of plant fibers using sodium hydroxide and potassium permanganate, followed by physical, mechanical, and morphological analyses to evaluate the effects of these treatments. Hybrid intra-ply woven fabrics are obtained by combining treated and untreated Alfa fibers with jute yarns. While untreated Alfa fibers are used to produce unidirectional plies, composite materials reinforced with these layers and hybrid intra-ply fabrics are manufactured, and comprehensive mechanical and physical characterizations are performed to evaluate their overall performance.

To clarify the work carried out and link the results to the experimental methodology used, the third chapter has been divided into two main sections. This will allow the data to be organized and analyzed systematically and sequentially.

The first section discusses the results of analyses of raw fibers collected from five different regions in Algeria during seasons of the year. This section includes a presentation and analysis of

the chemical composition, thermal analyses (TGA-DTG), structural (XRD), and spectroscopic (ATR-FTIR) analyses, as well as a study of the physical, mechanical, and morphological properties of Alfa fibers, with a discussion of the effect of seasonal factors and growth sites on these properties.

The second section deals with the results of the chemical treatment of Alfa fibers using sodium hydroxide and potassium permanganate, and the resulting changes in structural, physicochemical and morphological properties. This section includes an analysis of the performance of the treated Alfa fibers within the polymer matrix. This is achieved through the study of interfacial shear strength (IFSS) using the micro tensile test. It also presents the results of manufacturing composite materials reinforced with treated and untreated fibers. These are both in unidirectional configurations and intra-ply hybrid fabrics. The section also includes an evaluation of their general mechanical and physical properties.

This thesis concludes with findings that highlight the strong correlation between growth conditions, structural and chemical properties, and the mechanical behavior of Alfa fibers. emphasizing the pivotal role of chemical treatment and fiber orientation within the matrix in determining final performance.

Chapter I: Literature review

I.1 Introduction

The field of composite materials is a significant area of research in advanced materials, due to the variety of its structure and functions, and the broad range of industrial and engineering applications. These materials have attracted increasing attention due to their enhanced mechanical and thermal properties compared to conventional materials. In this chapter, the fundamentals of composite classification and manufacturing methods are reviewed, along with an analysis of the factors influencing their performance and the identification of their key practical applications. In this context, plant fibers have emerged as a promising alternative to synthetic fibers. Thanks to their natural abundance, low cost, renewability and environmental compatibility, making them a strategic option for promoting the development of more sustainable composite materials.

Accordingly, this review focused on the classification of plant fibers, their morphological structure, chemical composition, extraction methods, and thermal and physic mechanical properties, highlighting their contribution as natural reinforcing materials in composites. It also reviewed studies on methods to improve their performance, either through chemical treatments to modify the surface and enhance bonding with the matrix, or by studying the orientation of fibers within the matrix and its direct effect on the final properties of the composite. Finally, this chapter analyzed the factors affecting the properties of plant fibers, with a special focus on the pre-harvest stage, which is crucial in determining the quality and future performance of the fibers. Reference was made to the direct effects of environmental conditions, along with seasonal climatic changes, and the resulting significant variation in chemical composition and morphological structure, which in turn is reflected in the mechanical and thermal properties of the fibers.

Despite the significant impact of these influences, the available studies on them remain relatively limited, especially in Algeria, where the impact of the local climate and growth sites remains insufficiently explored. This lack of data reinforces the need to develop in-depth studies aimed at linking overall growing conditions, including environmental factors, climatic changes, and geographical variation, to the yield and functional properties of plant fibers. Such an approach would provide a broader knowledge base for understanding how to improve the overall performance of these fibers and open up new avenues for their development as a natural material that can compete with synthetic fibers in advanced, environmentally friendly composite applications.

I.2 Composite materials

Composite materials are defined as structures composed of two or more materials that differ in physical or chemical properties (Figure I.1) and are combined to produce a new material with distinctive properties [38].The principle of composite materials is based on the strategic combination of different materials to achieve properties that are not provided by the individual components, while each retains its distinctive properties. This is in contrast to metal alloys, whose components are mixed at the molecular level[39, 40].

Research on composite materials has become a fundamental field in science and engineering due to their ability to combine different materials to achieve superior mechanical, thermal, and functional properties that exceed those of individual components he utilization of composite materials can be traced back to ancient civilizations, wherein clay and straw composites were employed in construction. Over the course of history, this practice has undergone significant evolution, encompassing the incorporation of polymers, metals, ceramics, and nanomaterials [41] The importance of these materials is growing in a number of sectors, including aerospace, automotive, construction, and biomedicine, driven by the demand for lightweight, high-strength, and durable materials [42].

I.2.1 Components of composite materials

Composite materials typically consist of:

Matrix: This continuous medium surrounds the reinforcement, provides the final shape, protects the other components, and contributes to the transfer and distribution of mechanical stresses applied to the composite[43].

Reinforcement: This provides mechanical properties such as strength and stiffness [44].

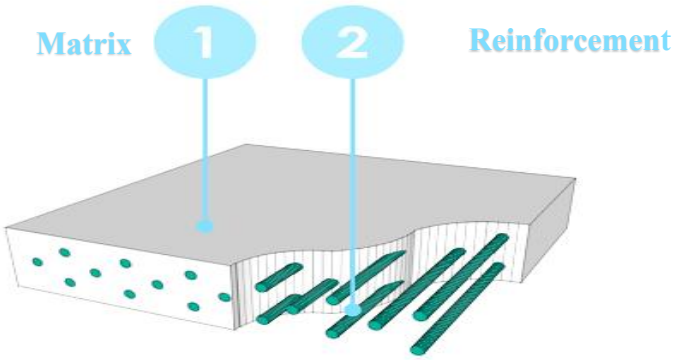


Fig I.1 Fundamental structure of composite material: matrix and reinforcement.

The integrated structural and functional synergy between the matrix and reinforcement materials provides the basis for developing materials that are precisely designed to meet specific requirements, by employing the wide variety of possible combinations between these two components [45].

I.2.2 Factors affecting the properties of composite materials

The final properties of a composite material are determined by a number of factors, the most significant of which are:

- Type and properties of the constituent materials.
- Arrangement and geometry of the reinforcing materials[46].
- Quality of the interfacial bonding between the matrix and the reinforcement.
- Composite materials manufacturing procedures.

Therefore, controlling the quantity, distribution, and orientation of the reinforcement material, in addition to optimally selecting the type of matrix and ensuring the interfacial strength between the fibers and the matrix, are critical factors in the design and development of composite materials [47]. Controlling these factors is also an essential requirement for achieving the best possible performance of the final product [48].

I.2.3 Classification of composite materials

Composite materials are commonly classified according to their constituents into two main categories: (i) classification based on the matrix, and (ii) classification based on the reinforcement.

I.2.3.1 Classification based on the matrix

Composite materials are classified according to their matrix, the continuous phase that surrounds and binds the reinforcement and ensures the structure's mechanical integrity, into the following main types:

I.2.3.1.1 Organic matrix composites / Polymer matrix composite

Polymer matrix composites are the most commonly used in industrial applications and are suitable for applications at temperatures below 250°C[49]. These composites consist of polymer resin that serve as the structural framework of the composite, reinforced with fibers or particles that provide the required mechanical and functional properties. They can be classified into two main categories based on the type of polymer matrix:

❖ Thermosetting matrix composites

This category uses cross-linked polymer networks that cannot be remelted or reshaped after processing. The most notable examples are epoxy, polyester, vinyl ester, and phenolic resins, and the most commonly used thermosetting resins' physical and mechanical properties are summarized in Table I.1. These materials are widely used in construction and the automotive industry, as well as in adhesives, boat building, toy manufacturing, and varnish coatings[50], and are combined strong mechanical properties, good thermal stability, and high resistance to chemical agents, making it suitable for use in structural applications with high performance requirements.[51].

Table I.1. Physicomechanical properties of common thermosetting resins[52] .

Resin	ρ (g/cm^3)	E (GPa)	σ (MPa)	Tmax (°C)
Epoxy	1.2	4.5	130	90–200
Urethane	1.1	0.7–7	30	100
Unsaturated Polyester	1.2	4	80	60–200
Urea-formaldehyde	1.2–1.5	6–10	40–80	140

E: Young's modulus, **σ**: Tensile strength, **Tmax;** Maximum operating temperature, **ρ**: density.

❖ Thermoplastic matrix composites

These consist of polymer matrices that can be melted and reshaped multiple times using heating and cooling cycles. Examples include polypropylene, polyethylene, nylon (polyamide), and polycarbonate. They are easy to process, recyclable, and quick to produce, making them suitable for high-volume industrial applications[53]. The physical and mechanical properties of the most commonly used thermosetting resins are summarized in Table I.2.

Table I.2. Physicomechanical properties of common Thermoplastic resins [52] .

Polymer	Tg (°C)	Tf (°C)	ρ (g/cm^3)	σ (MPa)	E (GPa)
PP	5	165	0.92	30	1.2
PLA	50-60	150	-	40-60	3-4
PVC	75-105	160-220	1.39	58	2.9
PS	90-100	-	1.05	55	3.2

Tg: Glass transition temperature, **Tf**: Melting temperature, **σ:** Tensile strength, **E**: Young's modulus, **ρ**: density.

In general, polymer matrix composites offer low weight, good corrosion resistance, high design flexibility, and relatively low manufacturing costs. However, their thermal stability is often limited to temperatures below 250°C, and their long-term properties can be affected by moisture absorption[51].

I.2.3.1.2 Metal matrix composites

These materials consist of a metal matrix reinforced with ceramic particles, fibers, or whiskers, with the aim of improving mechanical properties while maintaining the advantages of metals. The most common matrices are aluminum, magnesium, titanium, and copper and iron alloys[54]. Aluminum composites offer an optimal balance between strength, weight, and cost, making them ideal for use in automotive components. On the other hand, magnesium is perfect for aerospace applications due to its light weight[55], and titanium offers high strength and corrosion resistance in advanced applications. Metal matrix composites have high rigidity and greater mechanical strength than polymer matrix composites, with thermal resistance up to 600°C and excellent conductivity. However, it faces challenges such as high density, processing difficulty, and high manufacturing cost.

I.2.3.1.3 Ceramic matrix composites

Ceramic Matrix Composites (CMC) address the brittleness of traditional ceramics by incorporating ceramic fibers or particles into the matrix, thereby maintaining its thermal and chemical resistance while improving fracture toughness. These composites are classified as oxidized, such as alumina and zirconia, which provide chemical stability and oxidation resistance, and non-oxidized, such as silicon carbide, silicon nitride, and boron carbide, which provide higher mechanical properties and thermal shock resistance[56]. CMC operate most efficiently between 1000–1600°C and are used in turbine engines, rocket nozzles, and high-temperature applications. Despite their advantages, their brittleness, high cost, and difficulty in manufacturing remain major challenges[51].

I.2.3.2 Classification based on the reinforcement

Composite materials can be systematically classified based on their reinforcement characteristics, including type, form, and structural arrangement (Figure I.2) , as detailed in the following classification[57]:

I.2.3.2.1 Fiber-reinforced composites

I.2.3.2.1.1 Based on continuity

- Continuous fibers: Long lengths, high resistance when aligned with load.
- Discontinuous fibers: short lengths, less rigid, often cheaper.

I.2.3.2.1.2 Based on orientation

- Unidirectional: High anisotropy (strong in one direction)
- Multidirectional: Better property distribution.
- Random orientation: Nearly uniform properties, less efficient.

I.2.3.2.1.3 Based on fiber type

- Synthetic: Glass, carbon, aramid.
- Natural: Cellulosic, protein, mineral.
- Hybrid: Combination of two or more types for improved properties.

I.2.3.2.2 Particle-reinforced composites

- Large particles: Act as fillers, resist local deformation.
- Dispersion reinforcement: Fine particles impede crystal slip.
- Nanoparticles: <10 nm, significant property improvements with low weight.

I.2.3.2.3 Single crystal reinforced composites (Whiskers)

These materials are widely used to reinforce ceramics such as alumina (Al_2O_3) and silicon nitride (Si_3N_4), providing high hardness, excellent fracture resistance, and thermal stability exceeding 1500 °C.

I.2.3.2.4 Structural composites

- Laminate: Multiple layers with varying resistances (e.g., plywood).
- Sandwich: Lightweight rigid faces bonded to a lightweight core (high bending resistance).

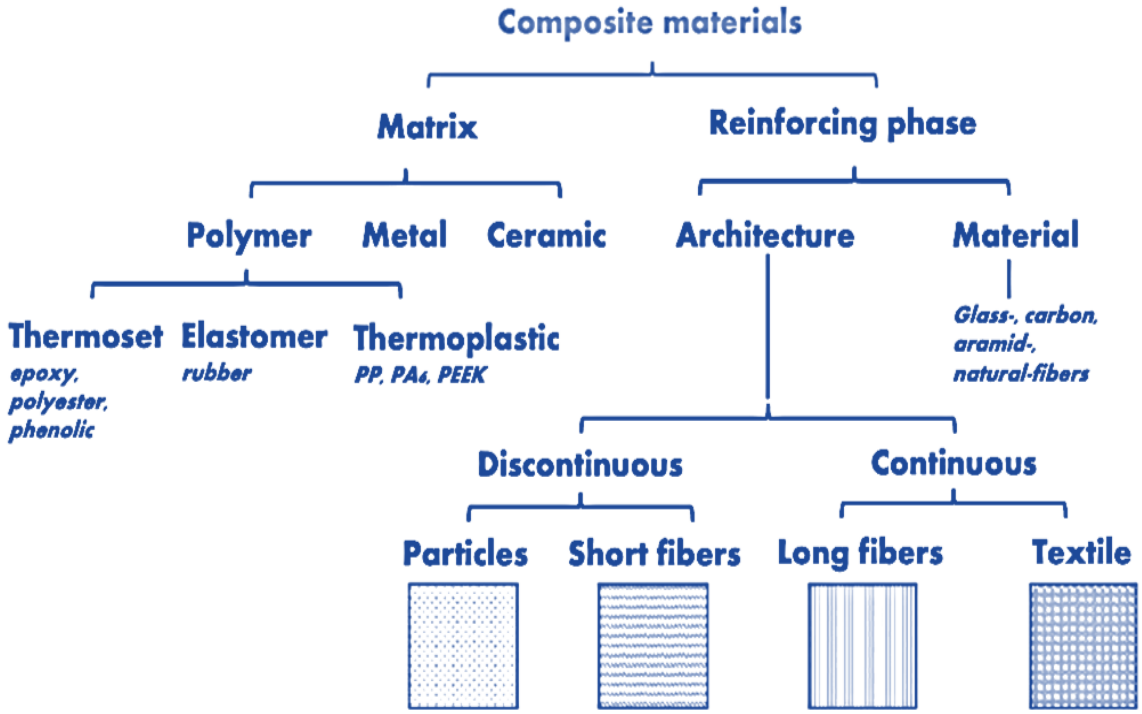


Fig I.2 Classification of different types of composites [58] .

I.2.4 Applications and significance of composite materials

Composite materials represent a vital part of many industrial sectors due to their high strength-to-weight ratio, customizability, and multifunctional performance[59]. Figure I.3 illustrates a wide range of engineering applications of advanced composite materials:

- Aerospace: Manufacturing lightweight, high-strength structures for aircraft and spacecraft[60].
- Automotive: Reducing vehicle weight while maintaining structural integrity, safety and performance[61].
- Construction: Reinforcing concrete and manufacturing durable components for bridges and buildings[59].
- Marine: Producing corrosion-resistant structures and components for ships and offshore platforms[61, 62].
- Medicine: Developing biocompatible implants, prosthetics and advanced imaging equipment[63].

- Sports and recreation: designing high-performance equipment that combines strength, flexibility and lightness.

Fig I.3 Engineering Applications of Advanced Composite Materials[64].

The adaptability of composite materials makes them suitable for many applications, ranging from everyday consumer goods to advanced engineering systems. Their ability to be designed to meet precise performance requirements, coupled with their potential environmental advantages through weight reduction and enhanced durability, establishes them as a vital driver of sustainable technological progress[65].

I.2.5 Composite manufacturing processes

Composite manufacturing processes can be classified into three basic categories: closed molding, open molding, and cast polymer molding. Each category includes a range of techniques designed to meet different requirements in terms of application, production volume, and final product shape. The selection of the most appropriate method is contingent upon on the required mechanical properties, cost, manufacturing precision, and design considerations[66, 67].

I.2.5.1 Open molding methods

I.2.5.1.1 Hand Lay-Up or contact molding

In this method, fibers are placed inside the mold and then manually saturated with resin using simple tools. This technique is often used in the production of prototypes or small quantities, and is suitable for large parts such as boat hulls and wind turbine blades[66].

I.2.5.1.2 Spray-Up

This technique applies a mixture of resin and chopped fibers onto the mold surface using a specialized spray gun. It offers faster production compared to hand lay-up, but the resulting mechanical properties are lower. It is typically used to manufacture simple shapes such as bathtubs and storage tanks[68].

I.2.5.1.3 Filament winding

Using this approach, continuous fibers are wound around a rotating cylindrical mold (mandrel), making it ideal for producing cylindrical parts such as pipes, pressure vessels, and drive shafts.

I.2.5.2 Closed molding methods

I.2.5.2.1 Resin transfer molding

The fabrication process starts with placing dry fiber fabric in the mold, then injecting resin under pressure to fill all voids. This method provides a high fiber content, consistent part quality, and low emissions, and is common in the manufacture of automotive and aircraft components[69].

I.2.5.2.2 Vacuum infusion

The procedure consists of positioning the fibers in the mold, and the resin is drawn through them using vacuum pressure, ensuring complete impregnation and good control of cohesion[70]. It is used especially for manufacturing large parts.

I.2.5.2.3 Prepreg molding

This method relies on the use of fibers pre-saturated with resin (prepreg) that are placed in the mold and then treated with heat and pressure. This technique offers the highest levels of strength and precision and is widely used in the aerospace industry[71].

I.2.5.2.4 Specialized methods

- **Pultrusion:**

This method is optimal for producing long elements with a continuous cross-section using a hot mold through which resin-saturated fibers pass to form solid sections with a uniform shape, such as bridge parts or stair railings[72, 73].

❖ **Braiding and weaving**

Fibers are woven or braided to produce seamless fabric structures, giving the product high flexibility and mechanical strength. This technique is used in the manufacture of prosthetic limbs and aerospace components[74].

I.2.5.3 Cast polymer molding

This process involves pouring a mixture of resin and fillers into a mold and can be performed with or without reinforcing fibers. This technique is used in the manufacture of industrial surfaces such as kitchen countertops and decorative panels[75].

I.2.6 Bio-composites

Composite biomaterials are materials composed of resins and fibers of natural origin, used as alternatives to synthetic fibers and resins such as glass or carbon fibers and epoxy resins. These materials have important environmental properties, including biodegradability, low energy consumption in production, and reliance on renewable resources. The natural fibers used in their production include plant fibers such as flax, jute, sisal, and bamboo, as well as animal fibers such as wool and silk. Natural composites include materials such as natural rubber and some plant-derived polymers. This type of material is an environmentally friendly option and offers acceptable performance in many engineering and industrial applications[76].

I.3 Natural fibers

Scientific research on natural fibers is an important field of study due to their renewable nature, biodegradability, and ability to reduce environmental impact compared to synthetic fibers[77]. In recent decades, this field has evolved from the early uses of natural fibers in textiles and construction to advanced applications in composite materials for the automotive, aerospace, and biomedical sectors [78]. Global production of natural fibers exceeds 38 million tons per year, reflecting their significant economic and environmental value [79].

Natural fibers are defined as fibrous materials resulting from geological processes or extracted from plant or animal sources. these fibers are characterized by a low density and light weight, coupled with excellent tensile strength and high biodegradability. Additionally, their low production costs, energy requirements, and ease of processing. In addition to being less harmful

to manufacturing systems than synthetic fibers based on non-renewable resources such as glass or carbon fibers, these properties make them a suitable choice for reinforcing matrices in composite materials intended for various industrial applications[80]. natural fibers are notable for their relatively high tensile strength and Young's modulus. They also have good thermal and acoustic insulation and electrical resistance properties. These properties are closely related to their high cellulose content and degree of crystallization[81, 82]. However, their performance is affected by factors that limit their effectiveness, including poor impact resistance, susceptibility to moisture absorption, inadequate interfacial adhesion with polymer matrices, and variability in quality, thereby necessitating the implementation of advanced enhancement techniques such as physical and chemical surface treatments[83]

I.3.1 Classification of natural fibers

Natural fibers can be classified according to their source into three main categories: plant fibers, animal fibers, and mineral fibers, a classification commonly (Figure I.4) used in academic studies and textile industry standards[84].

I.3.1.1 Cellulosic fibers

Lignocellulosic fibers are extracted from various parts of plants, including stems, leaves, bark, fruits, seeds, or roots, and are divided into subcategories[77, 82]:

- Bast Fibers: Taken from the outer layers of plant stems through a process called retting, where bacteria and moisture facilitate the separation of fibers. such as nettle, flax, ramie, kenaf, hemp, and jute ramie.
- Seed fibers: Taken from plant seeds or fruit husks, such as cotton, coconut, and kapok.
- Leaf fibers: Extracted manually or mechanically from leaves, they are characterized by their high stiffness compared to bast fibers, such as sisal, pineapple leaf fibers, and banana.
- Straw Fibers: Extracted from agricultural crop residues such as wheat straw, rice, and barley, they are characterized by low density and high durability.
- Grass Fibers: Taken from tall grasses such Stipa tenacissima L, elephant grass. corn and bamboo, they have unique properties.
- Wood Fibers: Extracted from soft and hard woods, they are often combined with virgin or recycled fibers.
- Fruit fiber: Such as coir, oil palm fiber

I.3.1.2 Protein fibers

Obtained from animal wool or hair, or from silkworm cocoons, including wool, silk, alpaca, llama, and angora rabbit fibers.

I.3.1.3 Mineral fibers

Extracted from minerals through mining, most notably asbestos, whose use has declined due to its harmful effects on health.

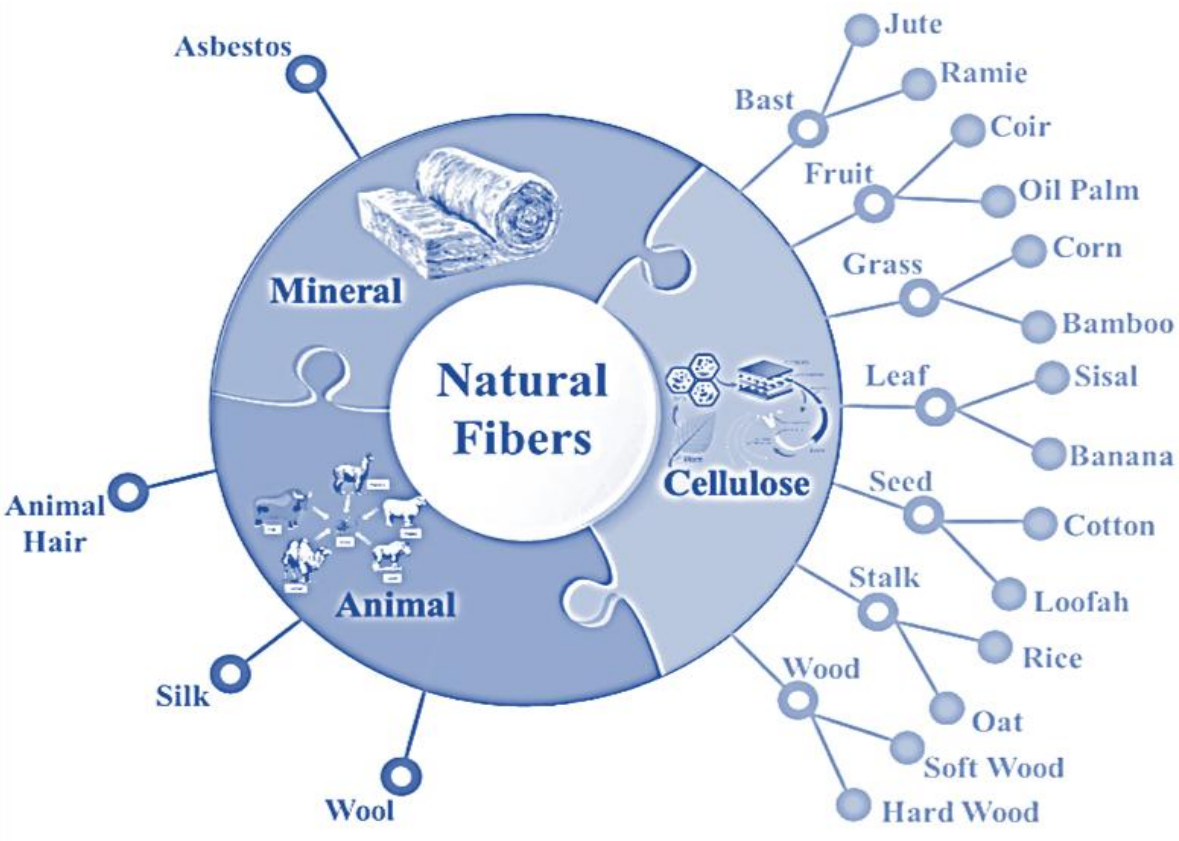


Fig I.4 Classification of natural fibers based on their origin and plant part [77].

I.3.2 Morphologic structure of cellulosic fiber

Plant fibers have a complex hierarchical structure that extends from the molecular to the macroscopic level. At the nanoscale, cellulose chains organize themselves into elementary fibers, which then aggregate to form microfibrils of cellulose. These microfibrils are oriented at a specific angle to the longitudinal axis of the fiber, known as the microfibril angle. This structure is incorporated into a matrix of hemicellulose and lignin to form the basic structure of the cell

wall[85]. The elementary unit of a plant fiber is a fiber cell; it consists of a central cavity (lumen) surrounded by a multilayered cell wall. The primary wall, a thin layer rich in hemicellulose and pectin, is the starting point. This layer endows the fiber with elasticity and allows the cell to expand during growth. The wall is surrounded by a secondary wall of greater thickness and complexity, consisting of three successive layers (S1, S2, S3) Figure I.5, which are characterized by a helical arrangement of cellulose microfibrils.

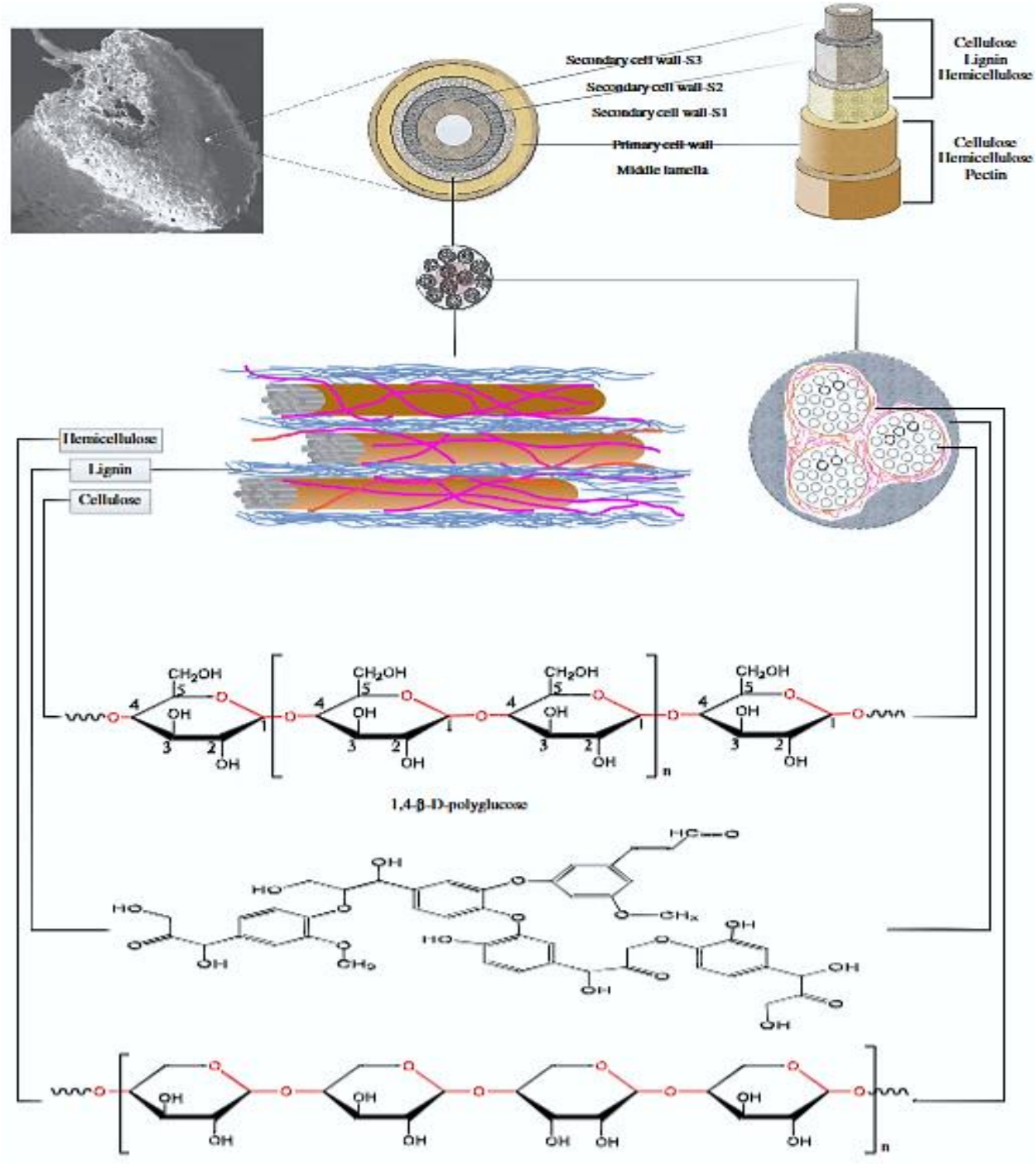


Fig I.5 Schematic representation of the natural fiber cell wall and its molecular structure[86].

The middle layer (S2) exerts the greatest influence on the physicomechanical properties of the fiber, as it contains the highest percentage of cellulose and the lowest microfibril angle, thereby conferring high stiffness and tensile strength. Additionally, the lignin matrix fills the spaces between the cellulose microfibrils and hemicellulose, enhancing the cohesion of the structure and determining its physical properties[87].

The content of the basic components within a single cell fiber varies from layer to layer, with the cellulose content increasing from the primary layer (S1) to the secondary layer (S2), while the lignin content decreases. Hemicellulose is distributed in similar amounts across the different layers[88, 89]. The structure of lignocellulosic fiber is complex and multilayered, combining flexibility and rigidity to achieve its structural functions within the plant.

I.3.3 Chemical composition of natural fibers

Plant fibers are regarded as natural bio-composite materials that are extracted directly from plants and consist mainly of cellulose, hemicellulose, and lignin[90]. These basic components largely govern the physical, mechanical, and thermal properties of fibers, as well as their cellular structure and cell size. In addition to these main elements, fibers contain smaller proportions of other components such as pectin, proteins, waxes, oils, ash, and minerals, as well as extractives. Cellulose represents the most critical structural element, occurring as tightly packed crystalline microfibrils with diameters ranging from 10 to 30 nm, which provide the fibers with high mechanical stiffness. Hemicellulose, an amorphous polymer, functions as a bonding matrix that connects the microfibrils through hydrogen interactions, whereas lignin, a hydrophobic aromatic polymer, fills the spaces within the structural network, enhancing the rigidity of the composite and contributing to compressive resistance[40]. The microstructure of the natural fiber cell wall and the molecular architecture of its fundamental components, as illustrated in Figure I.5.

The chemical composition of plant fibers varies significantly not only between different plant species, but also within the same species or even among different parts of the same plant[91]. This variability is influenced by several factors, including botanical origin, growth and maturity stage, climatic and geographical considerations, soil type, as well as extraction methods and preparation techniques[92, 93]. Such variations in chemical composition directly affect the final properties of fibers. A higher cellulose content combined with lower lignin levels enhances the mechanical properties, particularly tensile strength, whereas a higher lignin content contributes to increased stiffness at the expense of flexibility. Furthermore, both the microfibril angle and the degree of cellulose crystallinity are fundamental structural determinants that govern the quality of

mechanical performance[40]. Consequently, a thorough understanding of this pronounced variability in chemical composition, along with identifying its underlying causes and key influencing factors, represents a central challenge in the industrial utilization of lignocellulosic fibers, given its direct implications on their behavior and properties when employed in composite materials.

I.3.3.1 Cellulose

Cellulose is the main structural component of plant cell walls, is the most abundant organic molecule in nature, accounting for more than 50% of the biomass on Earth[94]. Cellulose is known for its long, regularly arranged chains, which primarily consist of repeating glucose units($C_6H1_2O_6$), with degree of polymerization ranges between 400 and 14,000, depending on the type and age of the plant. In some fibers, such as cotton and linen, it can reach around 10,000–15,000. As a renewable natural polymer, it has exhibited an increasing degree of importance in many industrial applications[95]. This compound is classified as a member of the family of β-D-glucans, where the cellobiose unit constitutes the fundamental structure, comprising two successive β-D-glucopyranose bonds[96], as illustrated in Figure I.6.

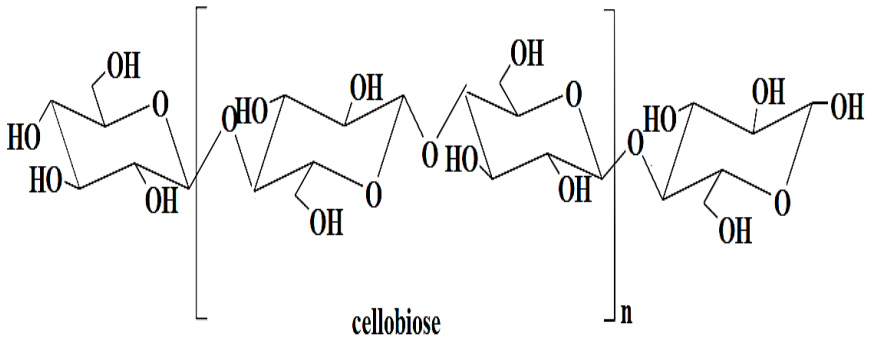


Fig I.6 Structural representation of the cellulose chain [96].

Cellulose has a semi-crystalline structure. Its molecular chains are distributed between highly ordered crystalline regions, which give the fibers rigidity and mechanical resistance, and less regular amorphous regions, which contribute to their flexibility[97, 98]. Hydrogen bonds (intra- and inter-chain) between hydroxyl groups (–OH) form the basis of the structural assembly (Figure I.7), leading to the formation of microfibrils, which in turn assemble into macro fibrils[99]. This microstructure is responsible for the high stiffness of crystalline cellulose, with an elastic modulus of approximately 136 GPa, which is significantly higher than that of glass fibers (75 GPa) .

Physiochemically, cellulose is a hydrophilic material due to the abundance of hydroxyl groups, which gives it a high moisture absorption capacity and poor dimensional stability when exposed to water. Conversely, its highly crystalline structure endows it with enhanced resistance to oxidation and superior thermal stability in comparison to hemicellulose[100].

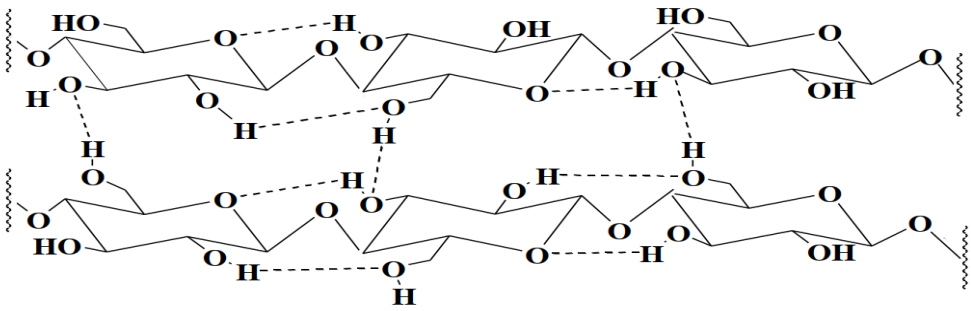


Fig I.7 Hydrogen bonding (intra- and inter-chain) in cellulose microfibrils[100].

Cellulose plays a pivotal role in determining the mechanical, thermal, and morphological properties of natural fibers. An increase in cellulose content is associated with improved tensile strength and stiffness [4].

I.3.3.2 Hemicellulose

Hemicellulose is a complex polysaccharide characterized by an irregular structure and shorter chains compared to cellulose, with an average degree of polymerization ranging between 50 and 300, making it less regular and more susceptible to hydrolysis. Unlike cellulose, which consists exclusively of β-D-glucopyranose units, hemicellulose is a heterogeneous polymer composed of a mixture of pentose and hexose sugars, including D-xylose, D-mannose, D-galactose, L-arabinose, as well as glucuronic and galacturonic acids (Figure I.8). This diverse nature gives it a branched and amorphous structure, which explains its solubility in alkaline media and its ease of extraction from cell walls[101, 102].

In terms of function, hemicellulose plays a vital role in strengthening the plant cell wall, acting as an adhesive matrix between cellulose microfibrils and interacting with lignin, thereby contributing to structural cohesion. Its helical structure provides it a degree of flexibility compared to highly crystalline cellulose[103].

However, its existence in natural fibers is associated with some undesirable properties; its hydrophilic nature increases its susceptibility to moisture absorption, biodegradation, and thermal degradation, which affects the stability and properties of the fibers when used in industrial applications[40, 104].

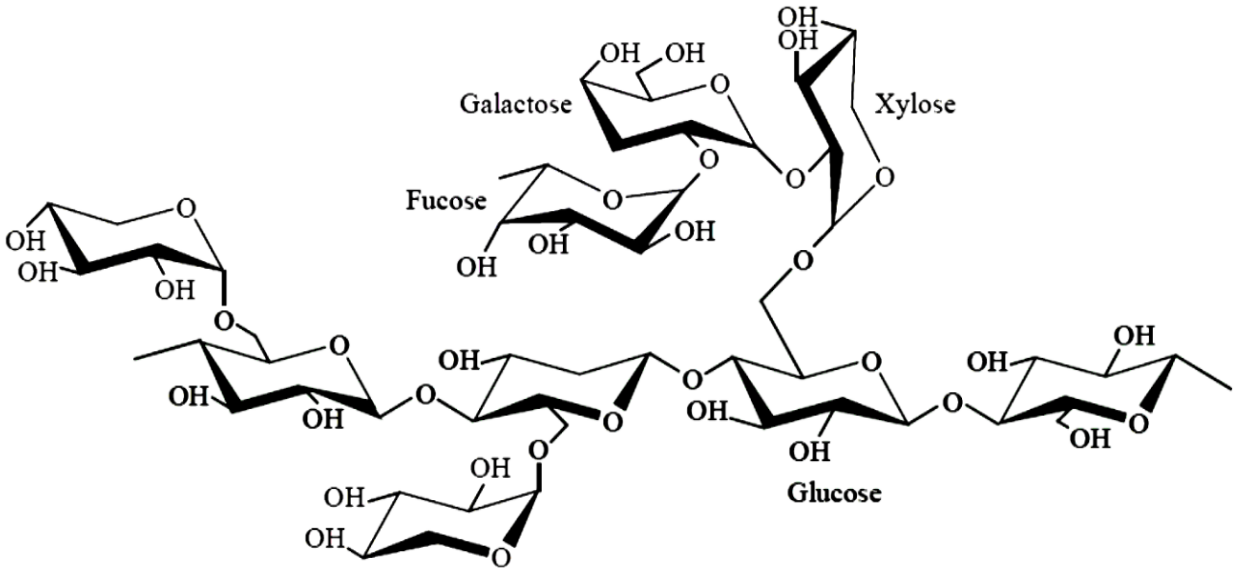


Fig I.8 Schematic representation of the hemicellulose molecule [103].

I.3.3.3 Lignin

Lignin is the second most abundant renewable organic material on Earth after cellulose. It is a highly complex, amorphous, three-dimensional aromatic polymer characterized by its hydrophobic nature and great structural diversity. It consists mainly of phenylpropane units (monolignols) Figure I.9, namely p-coumaryl alcohol, coniferyl alcohol, and sinapyl alcohol, which are polymerized by plant oxidation enzymes such as laccases during the lignification process. Unlike cellulose, lignin lacks a consistent repeating structure, featuring diverse intermolecular bonds that contribute to its unique physical and chemical properties[105, 106].

Lignin plays an essential role in providing plants with mechanical rigidity and structural support, and it also helps protect plant cells against biotic factors such as pathogens[107]. At the molecular level, lignin interacts with both cellulose and hemicellulose, forming a complex network through covalent and hydrogen bonds, which enhances cell wall resistance. Lignin has special properties, including: resistance to hydrolysis by acids, solubility in hot alkaline milieus, and ease of oxidation and condensation with phenolic compounds[106].

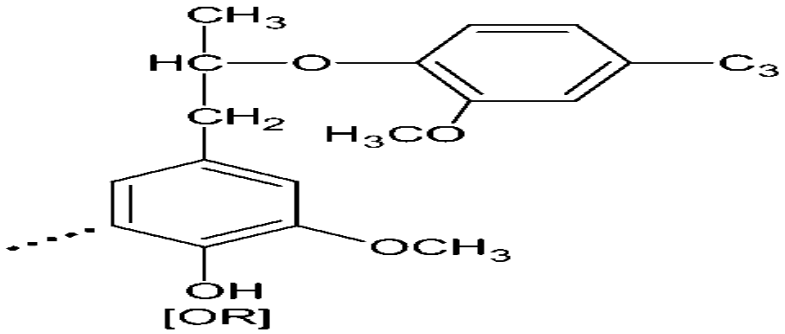


Fig I.9 Structural representation of the lignin [85].

I.3.3.4 Extractives

Extractives are non-structural organic compounds of low molecular weight, encompassing a wide range of substances such as fats, waxes, phenolics, as well as alkaloids, terpenes, and their derivatives. These compounds are characterized by their solubility in organic solvents such as ethanol. Although they are not considered structural components of the plant cell wall, they play an important protective role by shielding fibers from mold and insect attack and by reducing moisture loss [108].Moreover, certain extractives exhibit antimicrobial properties, which confer particular significance in pharmaceutical and food industries [109].

I.3.3.5 Minerals (Ash)

Ash content in plant fibers refers to the inorganic residue obtained after complete combustion at 500-600°C, mainly composed of oxides such as SiO_2, CaO, MgO, K_2O, Al_2O_3, and Fe_2O_3. The ash content ranged from 0.1% to 67%, depending on various factors, including plant characteristics (species, growth stage, part used), environmental factors (soil type, growing conditions), and processing techniques (harvest conditions, combustion temperature).the reported values include bamboo (3.0%), Arundo grass (3.8%), hemp (3.8%), lantana briquettes (67.3%), bamboo branches (1.2%), bamboo stems (2.0%), cotton stalks (8.6%), and rice straw (14.7%)[110].

The mineral components of ash have a significant effect on the physical and mechanical properties of fibers, balanced concentrations improve stiffness, thermal stability, ignition resistance, and compatibility with polymer matrices, while excessive ash content leads to deterioration of adhesion properties, equipment corrosion, and difficulties in homogenization and processing[111]**.** Therefore, controlling ash content and modifying its components is a key factor in ensuring the optimal performance of plant fibers and expanding their industrial and environmental applications, especially in composite materials and bioenergy. Table I.3.

shows the percentages of chemical components identified in some natural fibers, according to the literature.

Table I.3. Chemical composition of the most common lignocellulosic fibers.

Component / Fiber	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Waxes (%)	Minerals (Ash) (%)	References
Cotton	90	6,3	-	0.7	1.10	[112, 113]
Chanvre	57-92	6-22	6	0.7	1,5	
Lin	60-81	10-21	1-5	1.3-3	1-1.5	
Jute	51-84	12-24	5-14	0.4-0.8	0.17-0.7	
Ramie	68-85	3-17	0.5-1	0.3	0.3	
Kenaf	36-72	20-21	9-19	-	-	
Noix de coco	32-53	0.2-0.3	40-45	-	0.3	
Sisal	43-88	10-15	4-14	0.2-2	-	
Abaca	56-68	25-109	5.13	0.2-3	-	
Nettle stem	80.27	9.53	1.16	0.36	-	[11]
Bamboo	26-73.83	12.49-31	10.15-36.88	-	1.7-5	
Hemp	57-77	14-22.4	3.7-13	0.8	0.8	[82]
Bagasse	55.2	16.8	25.3	-	-	
Curaua	73.6	9.9	7.5	-	-	
Sugar	32-43	1.5-5	19-24	-	1.5-5	

I.3.4 Extraction methods of plant fibers

Plants are among the most important natural sources of fiber, which can be extracted from various parts of the plant, including roots, leaves, stems, seeds, and fruits [114]. The extraction process involves separating the fibers from these components using techniques that vary in mechanism and effect, which is reflected in the microscopic structure and final properties of the fibers. At present, the most common methods are: manual extraction, mainly used in traditional applications; mechanical extraction, which relies on grinding or physical disassembly; and chemical extraction, which uses alkaline or acidic solutions to remove non-cellulosic components. Biological extraction is based on the activity of microorganisms or enzymes to break down materials binding the fibers. These methods directly impact fiber purity and chemical composition, which in turn determine their mechanical and thermal performance when used in composite materials [115]. Understanding these differences is essential for selecting the most appropriate extraction techniques for industrial applications.

I.3.4.1 Biological extraction methods

Biological extraction is defined as a technique that relies on microorganisms or specialized enzymes to break down non-cellulosic components, such as lignin and pectin. These components act as binding agents between fibers in the cell structure. This process is typically carried out through soaking or biological retting, where the degradation of these compounds leads to the release of fibers. This mechanism allows the recovery of highly pure fibers while preserving the molecular integrity of cellulose[116, 117], which directly contributes to the enhancement of the mechanical and functional properties of the extracted materials.

I.3.4.1.1 Dew retting

Dew retting is one of the oldest and most common methods of separating plant fibers. During this process, fiber crops are left spread out on the soil surface for several weeks typically between three and six weeks [118].During this period, microorganisms break down the bonds between the fibers and plant tissues, facilitating the separation process [119]. However, careful monitoring is required to avoid excessive decomposition, which leads to cellulose degradation and weak mechanical properties, or insufficient decomposition, which complicates subsequent processing. Although this technique is low-cost and environmentally friendly, it remains limited by its heavy dependence on climatic conditions that cannot be controlled, as well as the production of lower-quality fibers and a darker color resulting from prolonged contact with the soil[120].

I.3.4.1.2 Water retting

Water retting is widely used by immersing fibrous plants in water for durations ranging from a few days to more than a month[121], depending on the type and temperature of the water. Anaerobic bacteria play a key role in breaking down the pectin that binds cells and fibers together, allowing for high-quality fibers to be obtained[119, 122]. However, this method is associated with high water consumption and environmental challenges resulting from liquid waste and gas emissions [123].

I.3.4.1.3 Enzymatic retting

Enzymatic extraction is a modern technique for separating fibers. It relies on specialized enzymes such as hemicellulases (especially xylanases), pectinases, cellulases and laccases. These enzymes are responsible for the breakdown of pectin and binding components in the cell wall, a process which allows the fibers to be released while maintaining the integrity of the internal structure [124] .The process commences immediately after harvesting, with the fibers being lightly

crushed to facilitate enzyme penetration[125]. This is followed by the incubation of the fibers in an enzymatic solution for a period ranging from a few hours to a full day [126]. While this technique provides high-quality fibers with distinctive mechanical properties, it remains costly due to the price of enzymes and wastewater treatment. Research has demonstrated the efficacy of certain microbial consortia in boosting bacterial activity while preserving cellulose integrity, facilitating efficient fiber extraction[127]. Approaches based on selected fungi have also been developed to increase cellulose purity and improve the efficiency of gum removal without damaging the structural properties of the fibers[128].

I.3.4.2 Mechanical extraction methods

I.3.4.2.1 Manual extraction

Manual extraction is one of the oldest and simplest methods of separating fibers. It relies on primitive tools such as knives and scrapers to remove fibers from plant parts after retting. The quality of the resulting fibers is affected by the number of times they are scraped and the precision of the processing. This method may be combined with retting with water or dew on occasion [129].Its key benefits include cost-effectiveness and the fact that it does not require specialist equipment. It is also well-suited for local production in rural areas, with the option of selecting high-quality fibers [129]. However, this technique requires considerable manual effort and is time-consuming, in addition to yielding variable results and poor quality [130], making it unsuitable for large-scale industrial applications. Nevertheless, it remains a significant option in craft, research, and specialized applications.

I.3.4.2.2 Mechanical extraction

Mechanical extraction techniques are among the key methods for isolating plant fiber bundles, with the latest techniques relying on a crushing process using peeling machines that increase fiber productivity by approximately 20 to 25 times compared to traditional methods[116]. Among these methods, rolling stands out, which involves repeatedly passing the cut stems between rotating cylinders in opposite directions to ensure efficient fiber separation. However, the high cost and substantial size of this equipment limit its economic viability in small-scale production. Despite their contribution to fiber liberation, these methods are unable to completely remove the pectin binding the fibers together, and the intense mechanical stresses may adversely affect the final mechanical properties of the fibers. This represents a major challenge to their adoption in the industrial field [53].

I.3.4.3 Chemical extraction methods

Chemical extraction, also known as retting with surfactants, is among the most widely used methods for liberating plant fibers, as it relies on hot chemical solutions that break down non-cellulosic compounds bound to the fibers [131]. These solutions commonly include compounds such as potassium hydroxide KOH, sulfuric acid H_2SO_4, calcium hypochlorite $Ca(ClO)_2$, sodium carbonate Na_2CO_3, and sodium hydroxide NaOH [123]. Among these methods, alkaline extraction using NaOH is the most prevalent, as it enables the dissolution of hemicellulose, pectin, and lignin, in addition to waxes and surface substances, with enhanced efficiency at temperatures above 75 °C [132]. This process is often preceded by preparatory steps such as crushing or peeling to improve the penetration of chemical solutions, while treatment duration may vary from a few minutes to several hours depending on operational conditions [133]. Despite offering rapid fiber extraction, improved surface quality, and good interfacial adhesion with matrices [131], this technique is associated with high costs and environmental concerns related to water pollution.

Extraction methods are not merely isolation techniques but determinants of fiber functionality. Manual methods excel in quality retention, Mechanical processes focus on achieving high production rates and meeting large-scale manufacturing requirements, and biological approaches offer Eco-friendly refinement. Future advancements must balance efficiency, sustainability, and fiber integrity ,especially for high-performance composites.

I.3.5 Physicomechanical and thermal characteristics of plant fibers

Natural plant fibers have relatively good thermal insulation properties due to their cellular structure and low thermal conductivity, which typically ranges between 0.029 and 0.076 W/m·K. This property is a significant advantage that makes them well-suited for construction applications requiring thermal regulation efficiency. Additionally, the hollow structure of some fibers contributes to effective sound insulation properties by absorbing sound waves and reducing noise transmission[134, 135]. However, the thermal stability of natural fibers remains limited compared to synthetic alternatives, as they begin to thermally decompose at around 200 °C[136], limiting their use in high-temperature treatments or high-temperature environments[137]. Flammability is also a crucial challenge, as these fibers tend to burn more easily than their synthetic fibers, requiring surface treatments or the incorporation of flame retardants when used in fire-resistant applications. Current research efforts are focused on developing effective retardants that do not adversely affect the mechanical properties or environmental benefits of these fibers.

Plant fibers have a low density ranging between 1.2 and 1.6 g/cm³, making them an attractive option for applications requiring light weight and improved fuel efficiency with reduced emissions. Their incorporation into polymer matrices allows for a 40–50% reduction in composite weight while maintaining an adequate level of mechanical properties[134]. Morphological characteristics such as fiber length, diameter, and length-to-diameter ratio play a pivotal role in reinforcement efficiency[138]. From a mechanical standpoint, tensile strength and Young's modulus depend strongly on cellulose content and microfibril angle; fibers with high cellulose content and low angles exhibit superior tensile properties[87] Some physical and mechanical properties of various plant fibers are summarized in Table I.4.

Table I.4. Physical and mechanical properties of plant fibers reported in the literature.

Fibers	Tensile strength (MPa)	Young modulus (GPa)	Elongation at break (%)	Density (g/cm^3)	Diameter (µm)	References
Palm	377	2.75	13.7	1.03	400–490	[86]
Elephant Grass	185	7.4	2.5	0.81	70–400	
Sisal	227–400	9–20	2–14	1.45	30–300	
Ramie	400–1000	24.5–128	1.2–4.0	1.00–1.55	20–80	
Jute	393 – 800	10–30	1.5–1.8	1.46	-	
Coco	131 – 175	4 – 6	15 - 40	131 – 175	12 - 24	
Coton	287–597	5,5 – 12,6	3–10	1.60	-	
Bamboo	503	35–91	1.4	0.91	-	
Alfa	297.82± 55.76	16.78± 5.34	1.44 ± 0.28	1.26 ± 0.01	235.13 ± 27.34	[34]
Diss	149	9,3	1,7	0,89	-	[139]
Agave	507-855	9-28	2-2,9	1,3-1,5	100-300	
Bagasse	20-290	2,7-17	0,9	1,2	10-34	
Typha angustifolia	450	27	1,7	1,25	66,7	
Abaca	220–980	3–12	3–10	1.5	-	[77]
Banana fiber	350–980	2–33.8	3–53	1.35	-	
coir	106–593	1.27–6	14.21–59.9	1.1–1.6	-	
Pineapple	170–1672	60–82	1–14.5	0.8–1.6	-	

The reinforcement mechanism is based primarily on the efficient transfer of load through the crystalline microfibrils of cellulose, while the hemicellulose and lignin matrix distribute stresses, providing a good balance between stiffness and flexibility[40]. Despite these advantages, moisture

absorption remains one of the most significant challenges, as it can reach 8% or more in some fibers, leading to swelling, dimensional instability, and poor adhesion to hydrophobic matrices. and thus, a decline in the mechanical performance of composites. To address this challenge, numerous researchers have developed various strategies, primarily focusing on modifying the surface of the fibers and enhancing their compatibility with the matrix[84, 140].

I.3.6 Treatments of plant fibers

The hydrophilic nature of natural fibers is their most significant limitation, as it leads to high moisture absorption and weak bonding with hydrophobic polymer matrices, In addition to non-uniformity uniformity in physical and chemical properties[34], low thermal stability, and limited mechanical resistance compared to synthetic fibers [141, 142]. This poses challenges in terms of reducing these shortcomings. Various surface treatments have been developed to modify the properties of these fibers by removing undesirable components such as pectin, lignin, hemicellulose, oils, and various impurities. This improves their structural and chemical compatibility with the matrix and enhances their thermal and mechanical stability [143]. These developments have contributed to expanding the use of treated natural fibers in various engineering applications, including the automotive, construction materials, and aircraft industries [144]. the most prominent techniques for modifying plant fiber surfaces, which has attracted significant attention from researchers[80, 142, 145-155], are:

I.3.6.1 Chemical treatments

Chemical treatments are among the most common and effective methods for improving the performance of plant fibers when used to reinforce polymer matrices. The primary aim of these treatments is to modify the microscopic structure of the fibers and enhance their surface properties. These modifications contribute to reducing moisture absorption, removing non-cellulosic components such as waxes, fats, pectin, and hemicellulose, as well as exposing or modifying hydroxyl groups on the fiber surface, which increases their reactivity with the polymer matrix[144, 156]. Research has demonstrated that these treatments enhance the interfacial bonding between the fibers and the matrix, thereby increasing the tensile strength, Young's modulus, and thermal

and thermal stability of composite materials [4, 34]. The most common chemical methods employed include alkaline, silane, Permanganate, acid, or oxidant treatments, as well as treatments with compounds such as acetic anhydride or benzyl chloride, which increase the roughness of the fiber surface and improve adhesion[40] (Figure I.10). The direct result of these modifications is the formation of a cleaner and more uniform rough surface, capable of enhancing the bonding mechanisms with the matrix, thereby improving the mechanical and functional properties of natural fiber composites[157].

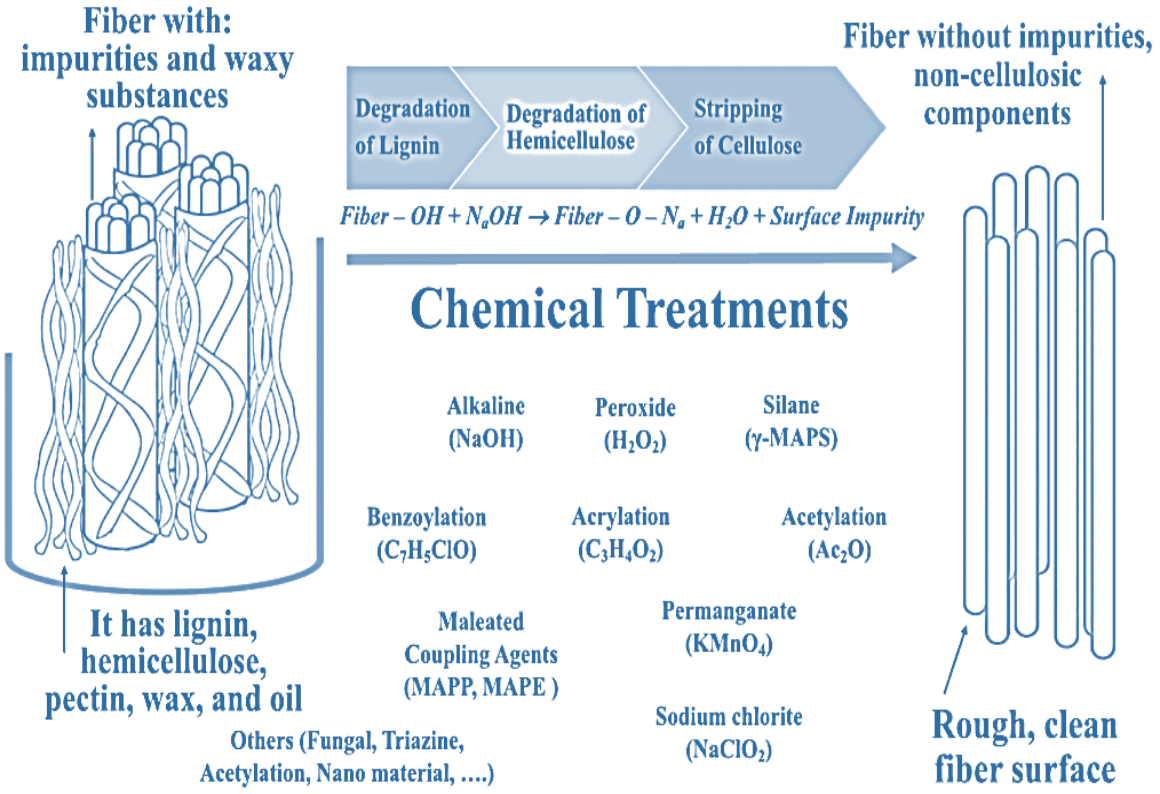


Fig I.10 Schematic illustration of chemical treatment processes and their impact on natural fibers[144].

I.3.6.1 .1 Alkaline treatment

Alkaline treatment using sodium hydroxide (NaOH) or potassium hydroxide (KOH) is one of the most common and effective methods for enhancing the performance of plant fibers. It involves immersing the fibers in an alkaline solution that modifies their chemical and physical structure[158]. This process removes non-cellulosic components of an amorphous nature, such as lignin, hemicellulose, pectin, waxes, and fats, resulting in a cleaner and rougher fiber surface that enhances interfacial adhesion with the polymer matrix[90, 106].At the structural level, alkaline treatment causes the fiber bundles to break down into microfibrils (fibrillation), increasing the effective surface area available for contact with the matrix, which improves fiber-matrix

adhesion[159]. These modifications also contribute to an increase in crystallinity due to the removal of amorphous components, which positively affects mechanical properties such as tensile strength and Young's modulus[160]. In addition to mechanical improvements, alkali-treated fibers exhibit better thermal stability and higher density compared to untreated fibers[161] . Furthermore, the following equation illustrates the chemical reaction that occurs between the fiber and sodium hydroxide [86].

$$\text{Fiber} - \text{OH} + \text{NaOH} \rightarrow \text{Fiber} - \text{O} - \text{Na} + \text{H}_2\text{O} \qquad \text{(I.1)}$$

Various studies have shown that combining alkali treatment with other techniques, such as permanganate and ultrasonic treatment, enhances stiffness and wear resistance, while improving surface structure and internal void distribution[34, 162]. However, the effectiveness of the results depends on the treatment conditions. It is important to note that high concentrations or long immersion times may lead to cell wall degradation and excessive extraction of components, which could potentially weaken the mechanical performance of plant fiber-reinforced composites.

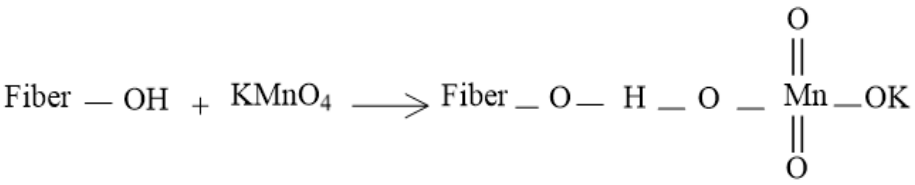
Several studies have reported on the role of treating fibers with sodium hydroxide in enhancing their effectiveness as reinforcement material composites. The research conducted by [84]indicates that treating palm fibers with hydroxyl solution at concentrations of (1, 4, 10) % for (3, 7, 24, 48) hours led to significant improvements in the mechanical properties of red clay composites reinforced with these fibers. The optimal concentration (4%) at a treatment time of 7 hours proved to be the most effective, with bending resistance increasing by 57% and compressive strength by 60%, respectively .Hestiawan, et al. [163]has reported that the alkaline treatment of Lantung (Artocarpus elasticus) using 4% and 6% of NaOH for 1 and 2 hours, has been shown to contribute to the removal of a significant portion of hemicellulose and lignin. This has had a positive effect on increasing the cellulose content and improving both the crystallinity index and tensile strength. The optimal results were achieved at 4% NaOH for two hours, yielding a crystallization index of 85.42% and tensile strength of 228.5 MPa. Sunny and Pickering [164]studied the effect of alkaline treatment of hemp fibers, using sodium hydroxide (NaOH). Their results indicated that this treatment enhances tensile strength, stiffness, and thermal stability, and improves interfacial adhesion with the polymer matrix, especially when the treatment is used at high temperatures .Meanwhile, Suárez, et al. [165]found that the use of sodium hydroxide and calcium carbonate in addition to sodium silicate to treat flax, pineapple, and ficus fibers contributed to the removal of

hemicellulose, lignin, pectin, and some surface impurities. This led to an increase in crystallinity and improved water repellency, as well as increased tensile strength compared to untreated fibers .As for García-Méndez, et al. [166], the investigations revealed that treating Agave angustifolia Haw fibers with a 5% NaOH solution for 10–60 minutes resulted in a reduction in lignin and hemicellulose content. This was accompanied by significant enhancements in morphological and crystalline structure, in addition to positive impacts on thermal and mechanical properties .Similarly, Kindole and Bigambo [167]demonstrated, through a study on sisal fibers, that alkaline treatment significantly reduces the content of hemicellulose and lignin. This is reflected in improved dyeability with reactive and permanent dyes, thus expanding their use in sustainable textile applications .On the other hand, Suriaman, et al. [168]highlighted that ramie, sugar palm, and coconut fibers treated with NaOH exhibited a substantial enhancement in tensile strength, with increases of 81%, 52%, and 56%, respectively, attributable to the elimination of natural dyes and impurities, with the optimal concentration varying for each fiber type. In addition, Karaduman, et al. [169]explained that combining alkaline treatment with sodium dodecyl sulfate in the case of hemp fibers helped to better remove non-cellulosic components, which was reflected in the enhancement of the tensile and flexural strength of hemp/epoxy composites .Furthermore, Prasad, et al. [170]shown that treating coir fibers with 5% of sodium hydroxide for 72 hours has effectively contributed to improving their mechanical performance, with a noticeable increase in both impact resistance and flexural strength of approximately 40%. This improvement is attributed to increased surface roughness, which enhanced the interfacial bonding strength with the polymer matrix. These results are consistent with those reported by Sreekala, et al. [171] and colleagues, where treating oil palm fibers with NaOH for 24 hours showed a significant increase in bending strength, which clearly exceeded that achieved by other chemical treatments. This superiority is attributed to the high efficiency of alkaline treatment in removing non-cellulosic materials and enhancing surface roughness, which contributes to improving the interfacial strength between the fibers and the polymer matrix.

The most recent and previous studies agree that alkaline treatment is one of the most effective methods for modifying the surface of plant fibers. However, achieving optimal results remains dependent on precise control of experimental conditions, particularly solution concentration, immersion time, and temperature, as the balance between these factors is crucial in enhancing the mechanical, thermal, and structural properties of the fibers.

I.3.6.1.2 Permanganate treatment

Permanganate treatment is an important chemical technique for modifying the surfaces of natural fibers, The mechanism of this treatment relies on generating radical sites in cellulose, thereby enhancing its reactivity with the polymer matrix [172, 173]**.** This treatment is usually carried out using a potassium permanganate ($KMnO_4$) solution in acetone, at varying concentrations and with an immersion time ranging from 1 to 3 minutes following the initial alkaline treatment [172-176]. This reduces the hydrophilic nature of the fibers, thereby minimizing water absorption in the composite materials reinforced with them. However, concentrations exceeding 1% have been shown to cause deterioration in the cellulose structure of the fibers [144, 173, 177] . The permanganate mechanism interacts with hydroxyl groups in cellulose and lignin components, producing new functional groups (hydroxyl, carbonyl, and carboxyl) that enhance surface properties. This oxidative effect contributes to the creation of micro-pits on the fibrous surface by removing parts of the amorphous regions, creating a rougher surface topography. This rough structure improves mechanical stabilization with the polymer matrix, while the newly introduced functional groups provide the possibility of forming covalent bonds or secondary interactions (such as hydrogen bonds) with the polymer, which enhances interfacial adhesion [144]. Furthermore, enhanced surface wetting enables the matrix to be more broadly dispersed on the fiber surface, thus enhancing interfacial adhesion. The following equation represents the reaction between the hydroxyl groups of the fiber and potassium permanganate.



(I.2)

The effectiveness of this technique has been confirmed by several experimental studies. Kudva, et al. [178] demonstrated that treating bamboo fibers with a 0.5% of $KMnO_4$, in addition to other treatments such as 5% sodium hydroxide and 1% ammonium hydroxide, enhanced tensile strength compared to untreated fibers. With the removal of impurities and the appearance of a cleaner and rougher surface structure. Similarly, a study by Abisha, et al. [179]on Butea parviflora fibers demonstrated a substantial enhancement in tensile strength, from 92 MPa to 198 MPa, and in Young's modulus, from 2.16 GPa to 4.40 GPa. These findings were associated with a notable increase in thermal stability, up to 240°C. SEM images demonstrated the effectiveness of the surface roughness for adhesion to the composite matrix. However, exceeding a concentration of 1% leads to fiber degradation and the formation of polar groups, which may have a negative impact

on the stability of the composite material and its sensitivity to ultraviolet degradation. A study by Bouchareb, et al. [34]also showed that treating Alfa fibers with NaOH (5%for 1hour) and permanganate (0.033% for 3 minutes) led to an increase in density, reflecting an improvement in strength and stiffness. ATR-FTIR results confirmed the removal of waxes and non-cellulosic components such as lignin and hemicellulose. These structural modifications contributed to enhancing mechanical performance and increasing crystallinity, as well as improving interfacial compatibility with the polymer matrix by enhancing wettability and resin penetration, which positively affected the mechanical properties of the reinforced composites. The results obtained by Sreekumar, et al. [180]showed that surface treatment of sisal fibers with a 0.02% potassium permanganate (KMnO4) solution for 3 minutes led to a significant improvement in the mechanical properties of the fibers and increased interfacial bonding efficiency with the polymer matrix. This enhancement can be attributed to the oxidizing effect of potassium permanganate, which contributes to the restructuring of the surface structure of the fibers by introducing new polar groups and increasing surface roughness. This, in turn, enhances stress transfer efficiency and leads to better performance of nano-composite materials in industrial applications. Meanwhile, a study by Acharya, et al. [181] on Helicteres isora fibers indicated that treatment with 0.5% $KMnO_4$,5% hydrogen peroxide and 5% sodium hydroxide reduced fiber hydrophobicity and enhanced mechanical and thermal properties compared to other treatments.

The results of previous studies agree that the effectiveness of the treatment depends mainly on fiber surface characteristics, adjusting the concentration of $KMnO_4$ and the treatment conditions. This contributes to improving tensile strength, Young's modulus, and thermal stability, as well as reducing moisture absorption and improving durability resistance in composite materials [86, 176, 182, 183]. However, further research is needed to reduce negative effects such as UV degradation and to develop processing methods that are more compatible with environmental standards.

I.3.6.1.3 Silane treatment

Silanes (generally represented by SiH_4 or by the functional formula $R_nSiX(4-_n)$) are classified as inorganic compounds such as silicone alkoxides, and are characterized by their hydrophilic properties and their ability to bind to various silicon-related groups. These last one's act as bonding agents, with one end interacting with hydroxyl-rich fiber surfaces and the other end binding to the polymer matrix, this enhances interfacial adhesion and improving mechanical and thermal properties of the material[144]. Research has demonstrated that silane treatment reduces the number of hydroxyl groups in cellulose, thereby decreasing moisture absorption and enhancing the interfacial stability of the natural fibers [173, 184]. The treatment process typically begins with

the dissolution of silane derivatives in a solvent such as alcohol or acetone. The silanization mechanism proceeds through four main stages: (i) Hydrolysis, during which silane molecules decompose in the presence of water and a catalyst, generating reactive silanol groups; (ii) Self-condensation, which must be carefully controlled to maintain the availability of silanols for bonding with natural fibers; (iii) Physical adsorption, occurring on the fiber surface or within its cell walls through hydrogen bonding, leading to the formation of a stable polysiloxane structure (–Si–O–Si–); and (iv) Thermal grafting, in which hydrogen bonds are converted into strong covalent bonds (–Si–O–C–) with the release of water [77]. Recent studies have demonstrated that the surface modification of coconut fibers using silane coupling agents effectively enhances their interfacial adhesion with a polylactic acid matrix [185]. Moreover, the use of different silane agents has been shown to improve the flexural modulus, impact resistance, and impact strength of the resulting composites [186]. Furthermore, incorporating silanes into a vitreous matrix of hemp oil and limonene improved fiber performance, increasing the glass transition temperature from 79.9°C to 90.8°C and reducing water absorption from 49% to 38%. These findings confirm that silane treatment is a promising technique for enhancing the overall performance and sustainability of natural fiber–reinforced composites [187].

I.3.6.1.4 Acetylation treatment

Acetylation represents a pivotal method for modifying the surface of plant fibers, whereby hydroxyl groups (-OH) are substituted with acetyl groups (CH_3CO-) through reaction with acetic anhydride, often in the presence of catalysts such as sulfuric acid or pyridine, which reduces hydrophilicity and improves thermal stability [171]. Research has demonstrated that this treatment contributes to enhanced interfacial bonding with the polymer matrix, increased strength, and dimensional stability by improving the compatibility between fibers and polymers [188] .This modification also contributes to stabilizing the internal structure of the fibers and reducing internal stresses, making them more resistant to mechanical and thermal changes. Despite the positive results in terms of reducing moisture absorption and improving mechanical and thermal properties, the process faces challenges related to the cost and toxicity of the reagents used, prompting the search for more environmentally friendly alternatives[188] .

I.3.6 .2 Physical treatment

Physical treatments are focused on modifying the surface and structure of fibers without direct chemical intervention. This process enhances the bond strength with the matrix and improves the mechanical and thermal properties of biomaterials [189].In this context, ultraviolet treatment has

been shown to be effective in increasing polarity and enhancing the strength of adhesion to the matrix by controlling the distance and duration of exposure. However, excessive treatment may weaken the fibers [190]. Similarly, gamma ray treatment has been demonstrated to contribute to cross-linking or decomposition within polymer chains, thereby enhancing durability and synergy between composite components while maintaining biodegradability [86]. Plasma treatment is considered to be one of the most widely used and environmentally sustainable methods, as it has been demonstrated to increase surface energy and enhance interactions between fibers and the matrix, which is reflected in improved mechanical properties of composites[191]. However, it is important to note that the treatment time must be adjusted to avoid fiber degradation. In a similar vein, the efficacy of corona treatment has been demonstrated, particularly in the context of polyolefins. This treatment has been shown to enhance the compatibility between fibers and polymer chains, resulting in enhanced thermal stability, tensile strength, stiffness and, and impact resistance [144, 191]. Fig I.11 shows a selection of the most common physical treatments applied to natural fibers.

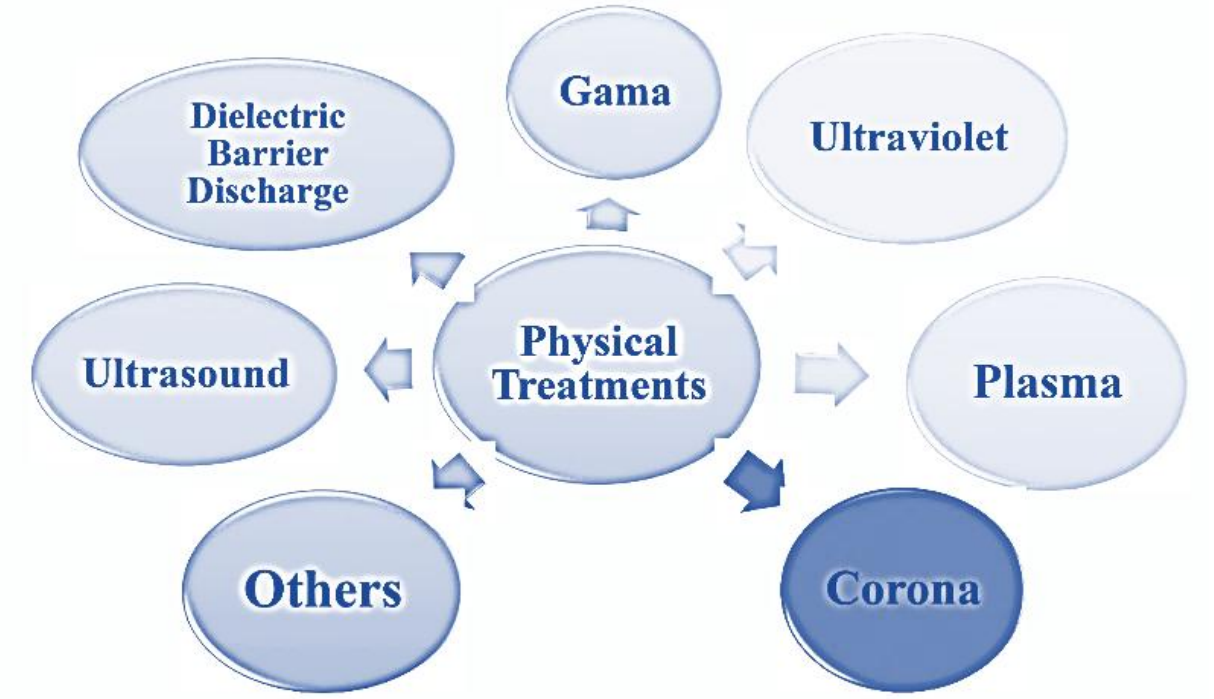


Fig I.11 Some common physical treatments of natural fibers[144].

I.3.6 .3 Environment friendly treatments

Environmentally friendly treatments represent promising alternative to traditional chemical methods for modifying plant fibers. These treatments focus on improving structural and surface

compatibility with polymer matrices, thus ensuring a reduced environmental impact. Plasma treatment has been shown to be effective in activating the surface and increasing roughness, to enhance the mechanical properties of the fibers. However, it should be noted that excessive treatment can lead to degradation[86]. The process of cellulosic degradation by bacteria is dependent on the action of enzymes that convert cellulose into glucose, thereby enhancing the material's porous structure and surface characteristics [192, 193]. The application of bacterial nanocellulose to fibers has been shown to improve the mechanical bonding between fibers and the matrix by enhancing distribution and adhesion [194]. Conversely, fungal treatment has demonstrated efficacy in removing lignin and reducing hemicellulose, leading to an enhancement in the contact area with the matrix and an increase in the rate of crystallization. White fungi have been shown to enhance the performance of hemp fibers [195]. Finally, enzymatic treatments (e.g. laccase and peroxidase) offer specialized and environmentally friendly solutions for modifying fibers and polymers, proving superior to traditional urea-formaldehyde bonds in bonding fiber boards [86].

I.3.7 Fiber reinforcement architecture in composite materials

Fiber orientation in the matrix is a one of critical factors governing the overall performance of the composite[196], as it directly affects how loads are transferred, stresses are distributed, and how the material responds under different loading conditions. Fiber alignment can be categorized as unidirectional, bidirectional, or random, with each pattern contributing to the composite's distinct behavior[197, 198]. Fiber orientation is also a determining factor in whether the composite will exhibit isotropic or anisotropic properties, depending on the extent to which it aligns with the loading direction[199]. Precise control of fiber orientation enables designers to tailor properties to suit specific application requirements, achieving an optimal balance between mechanical efficiency, weight reduction, and cost. Significant advances in manufacturing technologies, such as automated fiber placement and transfer molding, have made it possible to control alignment with high precision. However, manufacturing defects, such as irregularities and waviness, can negatively affect expected performance. Therefore, it is vital to study the effect of fiber orientation as a basic step in ensuring the reliability and performance of high-performance composite materials in advanced engineering applications[77, 197].

I.3.7.1 Classification of the principal fiber orientation patterns

In composite manufacturing, a range of strategies are used to orient fibers in order to achieve specific load conditions and performance requirements. The following categories represent the

most common arrangement of fibers reinforcements in composite materials, The arrangement of fibers within a composite matrix can be categorized into several primary types, each imparting distinct properties to the final material. The choice among these orientations depends on the specific application and the desired performance characteristics

I.3.7.1 .1 Random fiber orientation

Random fiber orientation (chopped strand mat) is a common pattern in composite materials, where fibers are distributed within the polymer matrix without a specific direction (Figure I.12a) [200]. This arrangement is typically the result of processes such as injection molding of short-fiber composites or the use of short-fiber chopped strand mats[201]. This gives the materials isotropic properties, making them suitable for applications that require balanced strength in all directions. However, this random distribution frequently leads to weaker mechanical properties compared to regularly arranged fibers, such as woven or unidirectional fibers[201], due to the absence of optimal orientation for load bearing[202, 203] (Pickering et al., 2016). These structures are low-cost and easy to process, making them more popular in industries that do not require high structural performance (Faruk et al., 2012). This structure also allows for the production of complex shapes, while ensuring relatively balanced properties in all directions. It is particularly useful in the manufacture of secondary components, insulators, and automotive interior parts, as well as simple marine structures. Thus, the practical value of random fibers lies in balancing acceptable performance with cost efficiency and ease of operation.

I.3.7.1.2 Unidirectional fiber orientation

Unidirectional composites are defined by the arrangement of all fibers parallel to a single axis (the principal load axis), giving them a highly anisotropic nature[204]. This structure, Figure I.12b a, is characterized by its high ability to achieve maximum tensile strength and stiffness when the load is applied in the direction of the fibers at an angle of 0°, while the properties decline significantly when deviating from this direction[197]. This is due to the fact that fibers are dominant over longitudinal properties, as opposed to transverse properties that depend on the polymer matrix, which often exhibits poor performance[205] .

Therefore, these composites are ideal for applications where the load path is defined and unidirectional, such as in aircraft longitudinal structures, wind turbine blades, rotating shafts in automobiles, high-performance sports equipment, and compressed vessel reinforcements [206].

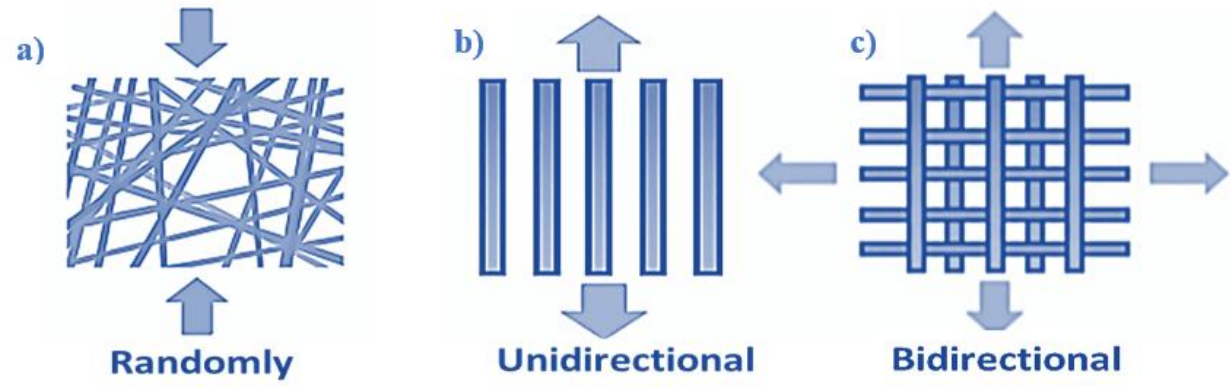


Fig I.12 Typical fiber reinforcement architectures used in composite materials [207].

I.3.7.1.3 Bidirectional and woven fiber orientation

Bidirectional composites contain fibers oriented in two primary directions, often perpendicular to each other (e.g., 0° and 90°) Figure I.12c. This distribution is achieved either by alternating layers of unidirectional layers or by using woven fabrics, where two sets of threads (warp and weft) interlock to form an integrated and stable structure [199, 208]. Two-dimensional woven fibers, as well as three-dimensional woven structures, provide a more uniform distribution of mechanical properties, reducing areas of weakness compared to unidirectional or random composites[209]. Consequently, these composites demonstrate balanced strength and stiffness in two directions, and the interlocking nature of the fibers contributes to enhanced durability and impact resistance by creating a more complex path for crack propagation [210].

The mechanical performance of these composites can also be controlled by choosing the weave pattern (plain, twill, satin) or by changing the orientation of the layers within the laminate [211].However, the presence of crimp in woven fibers can reduce the maximum mechanical performance in one direction when compared to straight fibers in unidirectional composites. These materials also exhibit improved fatigue behavior and higher damage resistance, especially in the case of 3D woven structures, which offer superior mechanical strength and stability. The homogeneous fiber distribution also facilitates the manufacturing process and improves resin flow[212].Bidirectional composites are widely used in applications requiring multidirectional strength, such as pressure vessels, structural plates and liners, flat panels, as well as aerospace components such as wing covers and airframe structures[213].

I.3.7.2 Effect of fiber orientation on composite performance

Fiber orientation in the matrix is a main factor in determining the overall performance of composite materials. It directly affects mechanical, thermal, and dynamic properties by controlling load distribution and material response under different loading conditions. This factor is a key element in controlling the "anisotropy" that distinguishes composite materials from homogeneous materials. In this context, many studies have focused on evaluating the effect of fiber orientation in different patterns (unidirectional, bidirectional, woven, and hybrid) in order to understand the close relationship between orientation structure and the properties of strength, stiffness, and performance sustainability.

For instance, composites with continuous fibers aligned at 0° to the load direction exhibit higher tensile strength and modulus of elasticity compared to those with fibers at 90° or random orientations [197]. In a theoretical and experimental study on carbon fiber/epoxy composites, Mohamed and Abdelbary [214]demonstrated that the highest tensile strength was recorded when the fibers were oriented at an angle of 0° compared to the other orientations. In addition, unidirectional kenaf composites at 0° demonstrated exceptional mechanical properties, with a tensile strength of 50 MPa and a flexural strength of 90 MPa. These are three times higher than those of commercially available random-oriented products [215]. Conversely, deviating the fiber angle from the load direction results in a substantial reduction in mechanical performance. For instance, Zalacca Midrib/ High-Density Polyethylene fiber composites demonstrated a marked decline in mechanical resistance as the fiber angle shifted from 0° to 90° [216]. While 0° orientation maximizes tensile and flexural strength, angled orientations often provide superior impact energy absorption. For example, woven kenaf composites with a 45°/-45° orientation absorb more impact energy than those with a 0°/90° weave [217]. In hybrid basalt/E-glass composites, changing the fiber orientation to ±45° enhanced impact strength by up to 46% compared to 0° samples, as this arrangement allows for higher plastic deformation and is more effective at dissipating impact energy [218]. Similarly, in carbon fiber reinforced PA6/PPO composites, impact strength was higher when fibers were oriented perpendicular to the direction of crack propagation [219]. Fiber orientation also affects tribological properties. Studies by Harsha, et al. [220]have shown that a parallel orientation of carbon fibers within a composite is more beneficial for wear resistance than a normal (perpendicular) orientation, as the latter can lead to increased fiber fracture and debonding at the wear surface. A study on carbon fiber-reinforced polymer composite produced using additive manufacturing technology has demonstrated that the lowest wear rate and coefficient of friction are achieved when the fibers are oriented at 0°, as

opposed to 45° and 90°[221]. A further study on carbon fiber reinforced liquid crystal polymer composites revealed that fiber orientation in parallel to the sliding direction exhibited superior wear resistance performance in comparison to a perpendicular orientation[222].

Woven composites offer a compromise, providing good but not maximal strength in two directions. Studies on natural fiber composites show that the performance of different orientations can depend on the specific fiber and matrix system. For instance ,Khan, et al. [223]developed poly lactic acid composites reinforced with plain-woven jute fabrics to evaluate how the weaving architecture influences their mechanical performance. The findings revealed that the woven configuration provided significantly enhanced mechanical properties compared to the nonwoven counterparts. In a study conducted by Khan, et al. [223]on carbon fiber-reinforced epoxy composites, three fiber arrangement patterns were adopted: unidirectional, bidirectional, and tridirectionally. The findings indicated that failure in the unidirectional and bidirectional patterns was associated with delamination, while the tridirectionally patterns exhibited enhanced resistance to this failure type, demonstrating more balanced behavior and closer to isotropic properties. Furthermore, mechanical tests showed that three-dimensional composites outperformed two-dimensional composites in terms of compression and bending resistance, while unidirectional composites reinforced with chopped fibers performed similarly to the unreinforced matrix. The research presented by Belliveau, et al. [204]addressed the development of discontinuous fiber composites by replacing unidirectional flakes with woven flakes, with the aim of reducing variability and improving mechanical performance. Tensile and flexural test results demonstrated that woven flakes offer enhanced stiffness and improved resistance, even with lower fiber content and tensile strength compared to unidirectional flakes. Furthermore, measurements demonstrated a substantial decrease in the coefficient of variation when using woven flakes, indicating the homogeneity of mechanical properties within the plane. The author concluded that this approach enhances the recyclability of composites without changing traditional manufacturing processes . Amir, et al. [205]conducted investigations on the development of banana fiber-reinforced polymer composites in order to evaluate the effect of different fiber configurations on mechanical properties. The results demonstrated that the banana yarn composition yielded the highest tensile and flexural strength, with increases of 294% and 72%, respectively, in comparison to unreinforced polypropylene. The other compositions (raw fiber and mat) yielded less efficient and varied results. Microscopic examinations confirmed the homogeneity of the bond between the banana yarn and the polymer matrix, indicating that the composition of the yarn is optimal for achieving the highest mechanical performance in these composites.

Fiber orientation is a key factor in improving the mechanical properties of composite materials; however, the performance of these materials does not depend completely on this variable, but is also influenced by a number of other factors, including fiber volume fraction, polymer matrix properties, and manufacturing techniques and processes. The interaction between these elements largely determines the structural and mechanical behavior of composites. Therefore, the choice of fiber orientation should be based on the specific application requirements, taking into account the optimal balance between tensile strength, stiffness, and other desired functional properties.

In addition to the influence of fiber orientation on the mechanical and physical properties of composite materials, the architecture of the weave and its different patterns represent another decisive factor in controlling their structural performance, particularly with respect to durability, interfacial strength, and resistance to multidirectional loading.

I.4 Woven structures

Woven structures in textile engineering are characterized by their high resistance to damage and stress, which gives them a clear advantage over alternative configurations. Since the 19th century, studies have shown that the use of natural fibers in a continuous woven form has improved mechanical properties by up to three or four times, coupled with a noticeable increase in durability and fracture resistance[213]. However, research developments in composite textiles in recent decades have gone beyond traditional forms to include advanced patterns and geometries such as two-dimensional (2D) woven, three-dimensional (3D woven) weaving, and hybrid woven composites, each of which has distinctive properties that allow for the adjustment of structural performance and the expansion of advanced industrial applications.

I.4.1 Basic concepts in weaving and loom structure

The steps of weaving can be summarized as follows:

- **Shedding:** The warp threads are moved up and down to divide them into two layers Figure I.13a, allowing the opening necessary for the weft thread to pass through.
- **Picking**: The shuttle or beater carrying the weft thread is passed through the opening from one side to the other Figure I.13b.
- **Beating up**: The newly inserted weft yarn is pushed toward the fabric using a reed to ensure tight packing of the horizontal threads and the formation of a compact fabric Figure I.13c.

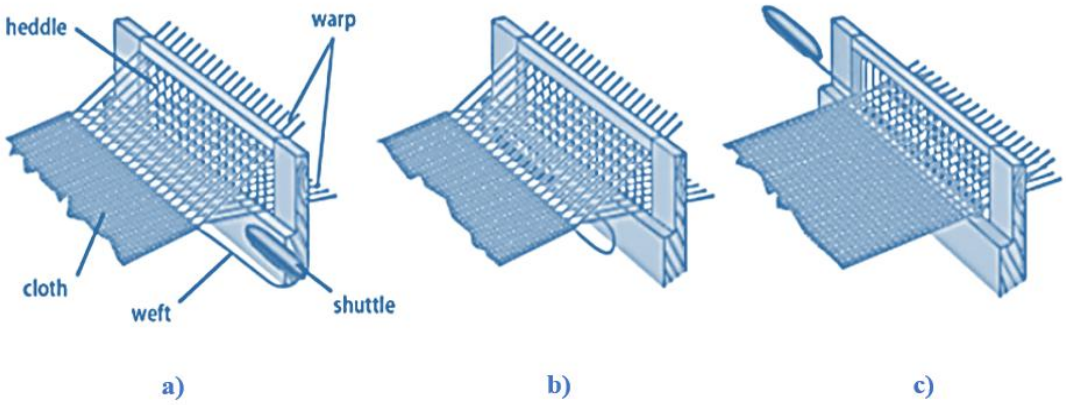


Fig I.13 The basic steps in hand weaving [213].

I.4.2 Weave pattern types in composite materials

Weave patterns are considered one of the key factors controlling the mechanical and physical properties of composite materials, as each pattern contributes to determining the stress distribution mechanism and the material's response to loads. These patterns vary according to the requirements of different industrial applications, the most prominent of which are the following:

I.4.2.1 Two-dimensional woven composites

Two-dimensional (2D) woven composites are defined as being based on perpendicular interlacing between two sets of threads: warp yarns in the 0° direction and weft yarns in the 90° direction. This provides a stable textile architecture. The fundamental principle of this structure lies in systematic interlacement patterns that control fiber crimp and yarn waviness. which directly influence load transfer efficiency and the overall mechanical performance of the composite. Despite the wide variety of modern weave designs, they essentially derive from three fundamental weave types: plain, twill and satin (Figure I.14) which form the foundation for more complex fabric designs. It is vital to understand these patterns if the right fabric is to be selected for different engineering applications. All other configurations represent modifications or combinations of these basic patterns[224].

I.4.2.1.1 Plain weave architecture

Plain weave is the simplest and most common basic weave pattern, relying on the warp thread passing over and under the weft thread alternately in a 1/1 configuration, giving it the highest density of interlocking points per unit area, this structure (Figure I.14a) in turn exhibits the least

shrinkage during the finishing process. and provides the fabric with high stability and a symmetrical structure, making it one of the most structurally balanced two-dimensional fabrics [225]. Despite its simple structure, plain weave in basalt/epoxy composites demonstrated mechanical superiority over basket weave, with increases of 12%, 10%, and 7% in stiffness, compression, and tensile strength, respectively [226]. Also, Pothan, et al. [227]investigated the mechanical behavior of different woven sisal fiber composites and reported that the plain weave configuration led to enhanced composite performance .This is consistent with the findings of Aisyah, et al. [228], who reported that plain-woven banana/kenaf hybrid polyester composites exhibited the highest mechanical strength when compared to both twill-woven and randomly oriented counterparts.. However, the density of the interlocks leads to increased fiber crimp, which reduces load transfer efficiency and negatively affects mechanical performance. A study by Torres, et al. [229] demonstrated that, relative to the jute laminate in plain weave, the twill weave pattern showed a 5.4% reduction in tensile strength. Whereas, the satin weave configuration exhibited a 28.13% increase in tensile modulus. However, its drapability is limited and it is fairly brittle. It can therefore be concluded that plain weave combines simplicity and stability, but its mechanical performance remains inferior to some other weave patterns such as twill and satin weaves.

I.4.2.1.2 Twill weave architecture

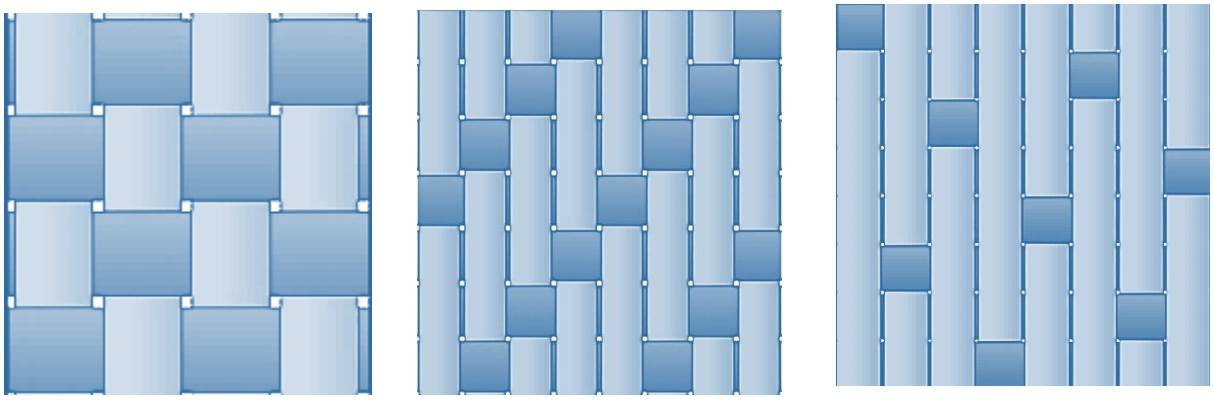
Twill weave is characterized by a diagonal structure resulting from an orderly sequence of warp and weft threads, which gives it better flexibility and shape compared to plain weave. The most common patterns are 2/1, 3/1(Figure I.14b), and 2/2, which reduce bending and increase fiber straightness, thereby improving load transfer efficiency[230]. In addition, the diagonal weave demonstrates a high impact energy absorption capacity (22.5 kJ/m²), attributed to the deflection of cracks across the diagonal paths [230, 231]. The low interlocking density allows for greater freedom of movement of the strands. This, in turn, results in higher tear resistance due to the participation of multiple strands in load-bearing at failure. Furthermore, the inclined weave has shown clear effectiveness in achieving a homogeneous distribution of the binder within the stacked layers of reinforcement fibers, resulting in stronger bonding and improved mechanical performance under tensile and bending forces [225].

I.4.2.1.3 Satin weave architecture

Satin weave is one of the most complex two-dimensional patterns, characterized by long floating threads (more than four threads) with few interlacing points(Figure I.14c), giving it a smooth surface and distinctive luster with high flowability in complex shapes [232, 233]. The low

waviness of the material has been shown to facilitate resin flow during processing, thereby reducing fiber damage caused by minimized crossing points. Research has demonstrated that satin composites exhibit a high flexural strength of 320 MPa, attributable to minimal fiber waviness [233, 234].From a thermal perspective, these composites exhibit distinctive properties, including low thermal conductivity (0.28 W/m·K), which is attributed to the discontinuous fiber paths (12), as well as good thermal cracking resistance, as demonstrated by Micro-CT tests [235]. However, satin weaves have clear limitations, most notably poor abrasion resistance (40–50% less than regular weaves) and the tendency of the threads to slip or break under load.

Thus, the morphological characteristics of this weave give it high thermal performance and distinctive surface flexibility, but it remains more suitable for decorative or non-structural applications where aesthetics and flexibility are preferred over mechanical strength.



a) b) c)

Fig I.14 Basic weave patterns: a) Plain [1/1], b) Twill [3/1], c) Satin [8-end].

I.4.2.2 Three-dimensional woven composites

Three-dimensional (3D) woven composites represent a significant engineering advancement due to the incorporation of vertical binding threads (Z-binders), which address the structural shortcomings of two-dimensional systems associated with weak interconnectivity and layer separability [236, 237]. The structural principle of these materials is based on the interlocking of warp, weft, and binding threads in three perpendicular directions, creating a cohesive monolithic structure capable of withstanding multidirectional loads and improving damage resistance. Their most notable advantage is enhanced separation resistance, as the Z-fibers contribute to inhibiting crack growth and altering their paths, which reduces the size of the separation compared to two-dimensional composites under the same impact conditions [238]. Research has demonstrated that the delamination threshold remains constant at 3.1 kN, as the matrix controls the initial cleavage, but crack propagation is inhibited by fiber interception and fiber bridges [239]. Furthermore, these

materials offer greater compressive strength exceeding 300 MPa through interfacial friction and damage propagation across levels. Research has demonstrated a fracture resistance enhancement of over 40% in Mode-I compared to two-dimensional systems [237, 240, 241]. Research has demonstrated that three-dimensional tissues maintain their structural integrity under cyclic loading conditions by utilizing additional energy dissipation mechanisms, such as fiber bridges, withdrawal, and fragmentation, thereby prolonging their fatigue life [236]. Three-dimensional weaving enables the development of multiple architectural configurations. The Figure I.15 presents schematic depictions of 3D woven structures, including the fully interlaced, angle-interlock, and orthogonal arrangements.

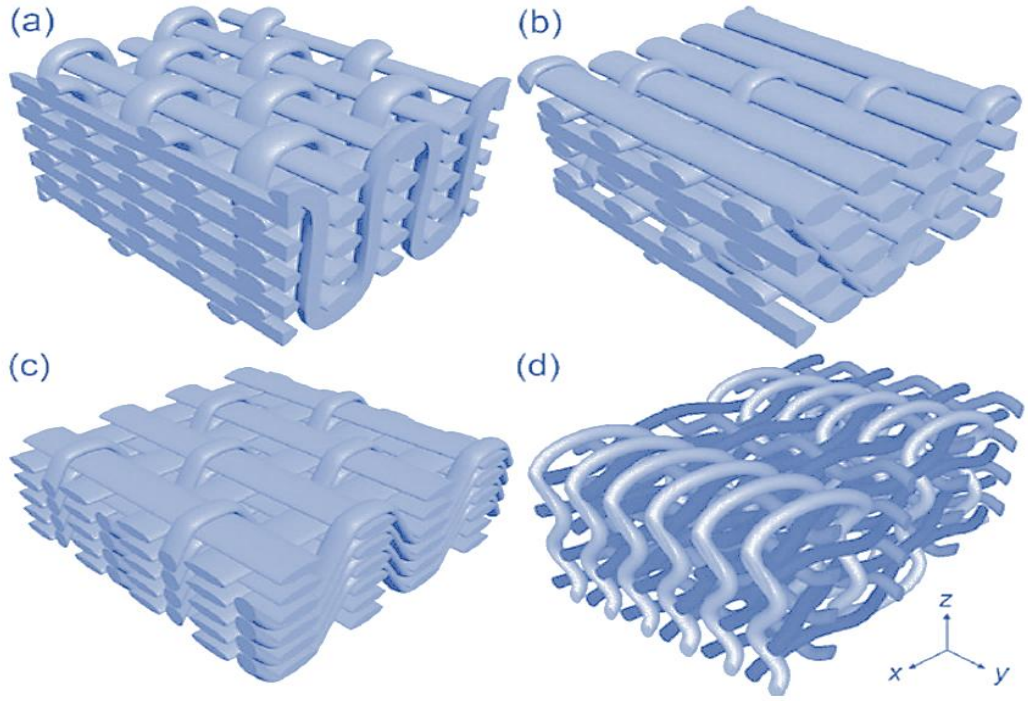


Fig I.15 Three-dimensional woven architectures: (a) orthogonal arrangement, (b) through-thickness angle-interlock, (c) layer-to-layer angle-interlock, and (d) completely interlaced configuration.

I.4.2.3 Hybrid woven composites

In recent years, researchers have increasingly focused on integrating two or more types of natural fibers within a single matrix in order to exploit the advantages of each while compensating for their individual limitations, thereby enhancing the overall performance of the composite. This strategy, commonly referred to as hybridization, has been widely studied due to its effectiveness in tailoring properties to meet specific requirements[242].

The main hybridization configurations most frequently discussed in the literature are illustrated in Figure. The first, known as intra-layer or yarn-by-yarn hybridization (Figure I.16a), consists of weaving different fiber yarns together within the same layer to produce a unified fabric. The second configuration, interlayer or layer-by-layer hybridization (Figure I.16b), arranges the fiber layers of distinct types sequentially, one above the other, offering a relatively cost-effective method for producing hybrid composites. The third approach, intra-yarn hybridization (Figure I.16c), places bundles of distinct fibers in parallel alignment, enabling mixing at the fiber scale.

Beyond these individual schemes, hybrid systems can also adopt more complex forms by combining elements of two or more configurations, thereby offering a wider range of design possibilities and property optimization[243, 244].

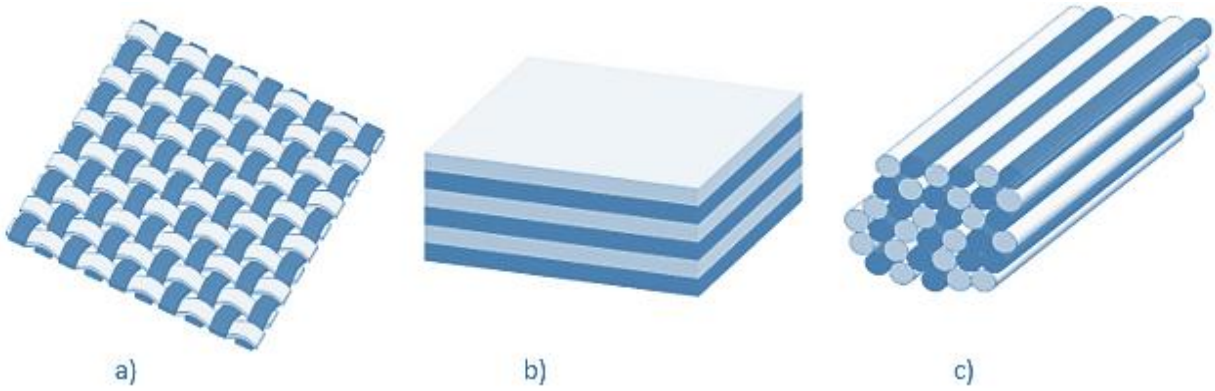


Fig I.16 primary hybrid configurations: a) intra-layer, b) inter-layer, and c) intra-yarn.

Scientific research in the composite materials context has seen growing interest in the hybridization of synthetic fibers with plant fibers, with numerous studies showing a marked improvement in the overall performance of these hybrid composites. Ferreira, et al. [245]examined the mechanical behavior of sisal/glass fiber hybrid composites reinforced with silica microparticles. Their findings revealed that the tensile and flexural strengths of the hybrid composite were enhanced by nearly 80% compared to the sisal-only composites. Moreover, the addition of 5% silica was particularly effective in further boosting the performance of composites with higher sisal fiber content. Giridharan [246]analyzed epoxy composites reinforced with hybrid ramie/glass fibers in two proportions, 20%/80 %and 30%/70%, through tensile, flexural, and impact testing. The study demonstrated that the 30%/70% ratio yielded markedly enhanced mechanical performance compared to non-hybrid counterparts. In a related work, Zin, et al. [247]investigated epoxy composites containing banana, pineapple, and glass fibers at different loadings (30%, 40%, and

50%). They reported that the formulation with 40 wt.% reinforcement achieved the highest flexural strength and exhibited greater thermal stability, as confirmed by TGA through an elevated onset degradation temperature.

However, recent research trends have focused on reducing dependence on synthetic fibers due to their negative environmental impacts. This has prompted researchers to develop sustainable composites based entirely on plant fibers. In this context, extensive research has been conducted on the integration of two or more types of plant fibers as reinforcements composite materials. These materials can be produced in various forms, including chopped fibers, woven fabrics, and through inter-layer, intra-layer, and even intra-yarn configurations. These studies have shown that plant-plant hybridization is a promising option for producing high-performance, environmentally friendly composite materials. Maslinda, et al. [248]reported that intra-layer laminates combining kenaf with hemp or jute displayed superior tensile and flexural strength compared to composites reinforced with fabrics of hemp, kenaf, or jute. This improvement was attributed to the interwoven architecture, which enhanced stress transfer among yarns and required higher loads to induce failure. Similarly, Cavalcanti, et al. [249]explored how intralaminar hybridization influences the mechanical response of jute-based epoxy composites. Their study demonstrated that combining jute with curauá fibers enhanced tensile strength by about 77 %, while hybridization with sisal fibers produced an improvement of nearly 68 % relative to pure jute composites. Dr.C.Senthamaraikannan1 [32]demonstrated that intra-ply hybrid composite combining jute/sisal and flax/sisal fibers exhibited higher tensile strength compared to composites reinforced only with woven jute, flax, or sisal fabrics. Yaisun and Trongsatitkul [250]incorporated bamboo leaf fibers (BF) and coir fibers (CF) into a polylactic acid matrix at a 1:2 BF:CF ratio. Intra-layer hybridization resulted in significant mechanical improvements, with tensile strength increasing by 16% compared to single-fiber composites, while impact resistance benefited from the combined rigidity of BF and the ductility of CF. Subsequent annealing enhanced crystallinity, leading to a 20 °C increase in thermal stability relative to unhybridized systems. This functional synergy between agricultural waste fibers not only reduces dependence on synthetic reinforcements but also offers a cost-effective and high-performance pathway for sustainable packaging applications. Chen, et al. [251] investigated corn stalk/sisal fiber hybrid composites, focusing on their mechanical response and water absorption behavior. The incorporation of sisal fibers notably improved performance, with flexural strength rising by 20 % and tensile strength by nearly 50 %, while water uptake remained largely unaffected. In a related study, Boopalan, et al. [252]explored jute/banana fiber epoxy hybrids at varying weight ratios . Their findings revealed that blends

containing up to 50 % banana fibers exhibited superior mechanical strength and thermal stability compared to nonhybrid-fiber composites. Parbin, et al. [253]developed hybrid composites by combining banana fibers and betel nut shell fibers at 20% by weight within a polylactic acid matrix. The incorporation of betel nut shell fibers enhanced hydrophobicity, reducing water absorption by nearly 50 % and increasing the flexural modulus by 28 %. In addition, thermal stability was enhanced as the decomposition onset temperature increased to 305 °C (approximately 30 °C higher than that of the banana fiber/polylactic acid composite). Meanwhile, tensile strength retention remained at 83 % following soil burial testing.

I.5 Factors affecting the properties of plant fibers

I.5.1 Introduction

The properties of plant fibers are subject to complex interactions between internal biological factors and external environmental and manufacturing conditions. These properties, whether mechanical, thermal, or morphological, are not constant but vary according to a complex set of influences that include genetic traits, climatic conditions, extraction and processing methods, and even measurement conditions. These influences can be systematically classified into two main categories: pre-harvest factors, which are related to the plant and its environment, and post-harvest factors, which include extraction, processing, and preparation methods [254].

I.5.2 post-harvest factors

These refer to the conditions and processes that influence the properties of plant fibers after harvesting. They include methods of collection, drying, storage, and primary processing, all of which can significantly affect the quality, structural integrity, and overall performance of the fibers in various applications.

- ✓ Extraction methods (mechanical, chemical, biological) and retting duration[255].
- ✓ Fiber surface treatments: physical, chemical, enzymatic, thermal, and environment-friendly methods such as steam explosion, acetylation, plasma, nanocoating, ultrasound, corona, and fungal treatments.
- ✓ Drying and storage conditions: temperature, humidity, ventilation, storage duration.
- ✓ Transport and supply conditions.
- ✓ Life expectancy of the fiber after storage.
- ✓ Natural degradation: is the loss of properties that occurs as a result of prolonged exposure to ultraviolet rays or moisture[256, 257].

- ✓ Measurement conditions: tensile speed, gauge length, Cross-sectional diversity of fibers at distinct points, moisture and temperature.

I.5.3 Pre-harvest factors

These factors are associated with the plant itself, its surrounding environment, and the conditions under which it grows. They include genetic characteristics, soil composition, climatic conditions, and agricultural practices, all of which play a crucial role in determining the morphological, chemical, and mechanical properties of the resulting fibers.

- ✓ Plant species and genetic traits: Differences in species and varieties are reflected in the chemical composition and cellular structure of the fibers[258].
- ✓ Physiological maturity and age at harvest: The rates of cellulose, hemicellulose, and lignin change as the plant ages[259].
- ✓ Biological cycle: Distinction between growth stages (spring/autumn) and dormancy stages (summer/winter)[260].
- ✓ Environmental conditions: These include soil quality and fertility, prevailing climate, temperature, rainfall, and atmospheric humidity[260, 261].
- ✓ Climate change: such as drought or heat waves, which can directly affect fiber quality[14].
- ✓ Mineral nutrition and fertilizers: the availability of major and minor elements (N, P, K, Si, Ca) contributes to the promotion of cell wall growth and fiber crystallization.
- ✓ Planting density and growing conditions: Related to planting distances, growth density, and their effect on fiber uniformity[262].
- ✓ Diseases and pests: Can weaken cell structure and negatively affect mechanical properties[263].
- ✓ Location of fibers in plant tissue: Fiber performance varies depending on its location within the stem or leaves.
- ✓ Harvest timing and method: It is important to note that variations in fiber quality can be significantly impacted by differences in harvesting time, method of harvesting [260].
- ✓ Microstructural characteristics: Such as cell wall thickness, fiber roughness, porosity, and microfibril angle, which largely determine the final properties[264].

I.5.3.1 Effect of pre-harvest factors on the properties of plant fibers

Many researchers have focused on studying the factors that influence the properties of plant fibers, particularly those related to the pre-harvest stage, which is considered a critical phase in determining the chemical composition, microstructural organization, and final mechanical

properties of the fibers. In this context, several studies have examined the effects of maturity degree, soil quality, plant age, and harvest timing, as well as environmental and genetic factors, fiber location within the plant, and growth stage, on the overall performance and structural and functional quality of plant fibers.

Asmare [11] investigated the influence of nettle maturity on the physical and mechanical properties of mechanically extracted fibers. The study analyzed samples from plants aged between three and five months. Results revealed that the period between the 14th and 16th weeks of stem age represents the optimal stage for obtaining high-quality fibers. At this maturity stage, the fibers exhibited a moisture content of approximately 12.5%, a fineness between 1.0 and 1.2 tex, and a dry tenacity ranging from 5 to 7 CN/tex. The elongation at break varied between 2.1% and 2.3%. This behavior was attributed to gradual changes in cellulose and lignin proportions as the plant physiologically matured. These findings confirm that selecting the optimal harvest time when the plant reaches full maturity is critical for producing fibers with superior physical and mechanical properties, making them suitable for diverse applications such as agricultural packaging, superabsorbent materials, high-strength and hygienic textiles, and eco-friendly paper and composite products. Figure I.17 illustrates the variations in the physicomechanical properties of nettle stem fibers with plant age.

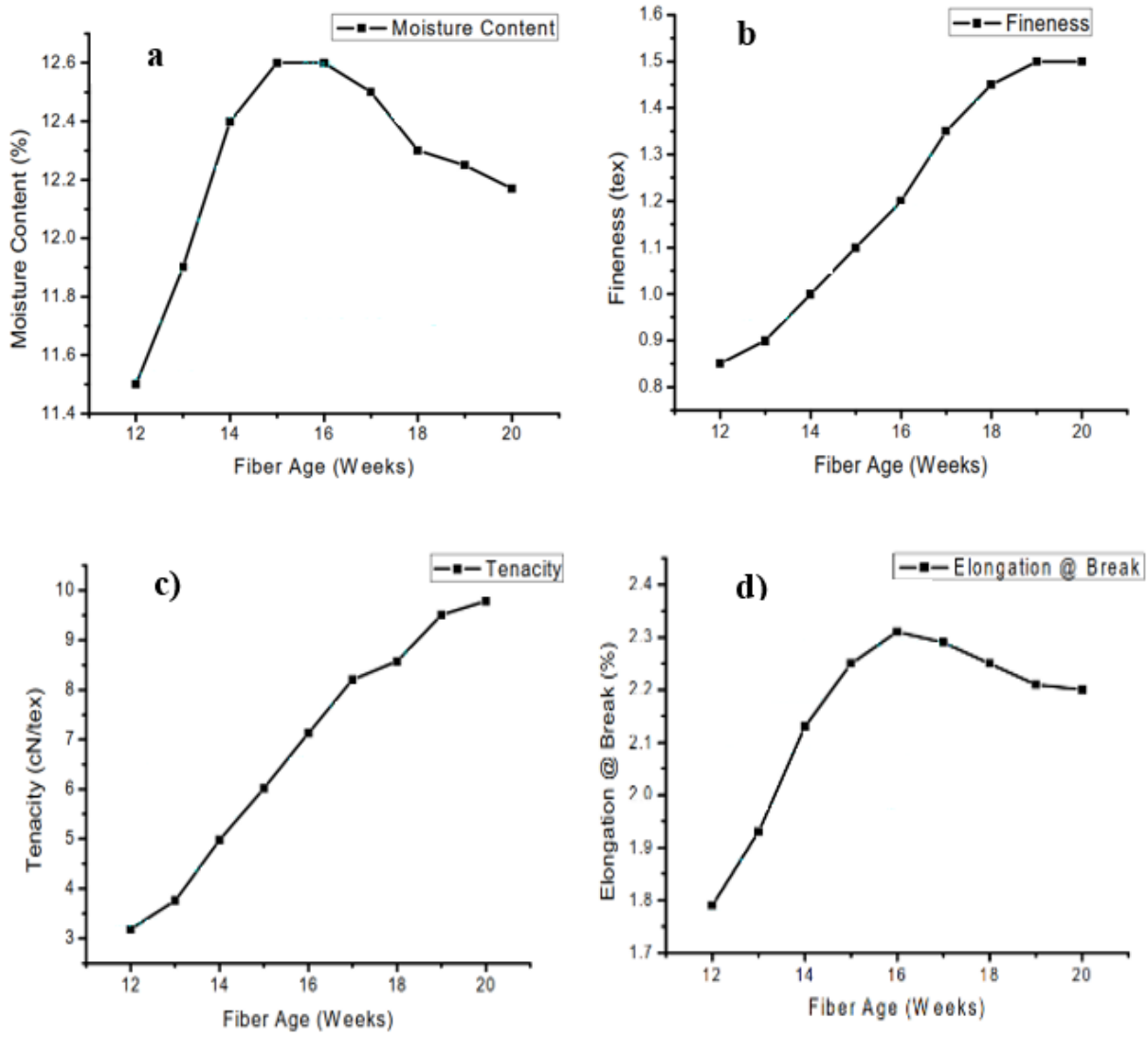


Fig I.17 The variation in the physical and mechanical properties of nettle stem fibers with plant age: a) Moisture content, b) Fineness, c) Tenacity, d) Elongation at break [11].

Liu, et al. [12] investigated the effect of growth stage and retting time on the mechanical and chemical properties of hemp fibers. with the goal of determining the optimal harvest stage and ensuring the suitability of the fibers for use in high-performance composites. The results demonstrated that plant maturity led to a significant decrease in the content of bast fibers and the thickness of the primary layer, which was offset by an increase in the proportion of secondary fibers, which reached approximately 10 % at the seed maturity stage. Furthermore, there was a significant decrease in cellulose deposition within the cell walls of the fibers as maturity progressed, while there was a significant increase in lignin content and a slight decrease in pectin (Figure I.18). Microscopic and histochemical analyses supported these findings, revealing distinct

changes in the morphological structure and spatial distribution of carbohydrates and lignin between the early and late stages of growth.

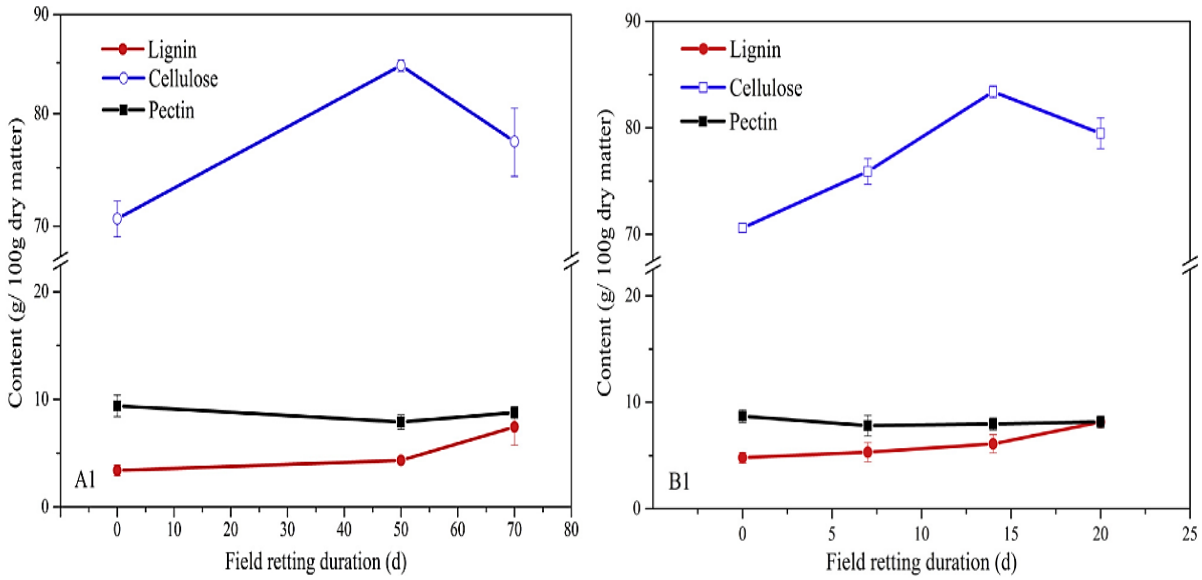


Fig I.18 Hemp stem content variations during field retting A1: early harvest, B1: late harvest [12].

The study demonstrated that fibers extracted at the onset of flowering exhibited superior tensile strength and elongation compared to fibers produced in later stages. This is attributable to the accumulation of secondary fibers and the decline in cellulose deposition, resulting in a deterioration in mechanical properties (Table I.5) .

Table I.5. effect of retting and harvest stage on hemp fiber mechanical properties[12].

Retting duration(days)	Early harvest			Late harvest				P* Value
	0	50	70	0	7	14	20	
Ultimate tensile strength (MPa)	954^{a} (162)	822^{b} (136)	816^{b} (128)	812^{ab} (105)	832^{a} (198)	697^{ab} (135)	683^{b} (107)	P< 0.05
Elongation at break (%)	6.2^{a} (1.2)	5.4^{ab} (1.5)	4.6^{b} (1.4)	4.6(1.3)	4.7(1.8)	4.5(1.5)	4.5(1.2)	P< 0.001
Stiffness (GPa)	34.9(4.4)	33.5(8.1)	31.3(4.0)	31.1^{ab} (5.6)	32.5^{a} (4.8)	30.7^{ab} (4.5)	27.5^{b} (4.6)	P< 0.05

Values marked with distinct letters within the same row for hemp harvested at the same time differ significantly (P< 0.0).

P* Value: Effect of harvest time on the mechanical properties of non-retted hemp fibers.

It was also noted that extended retting periods (70 days) resulted in a decline in fiber quality due to accelerated cellulose degradation by microbial activity, while no significant change was observed with short-term retting. Therefore, the study recommended harvesting at the onset of flowering to obtain fibers with high mechanical performance, suitable for applications in composite materials. It was also recommended that short retting periods be supported by auxiliary methods, such as enzymes or microbial and chemical treatments, to ensure efficient extraction and better quality.

Many studies indicate that plant species and genetic traits are decisive factors in determining the chemical and mechanical properties of plant fibers. Lignocellulosic components vary significantly between species, with cellulose content ranging from 20-60%, hemicellulose from 30-60 %, and lignin from 15-25 %, as in flax, hemp, jute, sisal, and coconut fibers[258]. Genetic improvements, particularly those targeting cellulose crystallinity and microfiber orientation, have shown remarkable improvements in fiber performance, with some hemp varieties recording Young's moduli of up to 70 GPa and tensile strengths exceeding 800 MPa. Genome-wide association studies in cotton (Gossypium hirsutum) have identified 31 quantitative trait loci (QTLs) associated with key traits such as fiber strength, length, and softness index. Furthermore, some genes have been shown to play a direct role in promoting fiber elongation by 15–20% in genetically modified strains[265]. Similarly, hemp varieties such as Futura 75 are characterized by a 12–15% increase in fiber yield thanks to photoperiod-insensitive flowering genes, which allows for greater flexibility in harvest times[266].

Razali, et al. [267]presented a detailed study on roselle fibers, investigating how maturity (3, 6, and 9 months) affects their chemical, physical, mechanical, thermal, and morphological properties. The results showed that the cellulose content was highest at three months (64.5 %), which was directly reflected in a tensile strength of 453.4 MPa, before gradually declining with the age of the plant due to a decrease in cellulose content and a relative increase in lignin content. This chemical change was accompanied by changes in physical structure, with fiber diameter and density increasing with maturity. These measurements ranged from 40-100 micrometers at 3 months to 90-150 micrometers at 9 months. While the density increased from 1.33 to 1.42 g/cm³. A decrease in water absorption from 320 % to 289 % was also observed, along with a reduction in the size of lumens. This improves dimensional stability and reduces the sensitivity of the fibers to moisture. Despite the occurrence of mechanical deterioration with age, TGA analyses demonstrated adequate thermal stability for all ages, with decomposition stages characterized by hemicellulose decomposition between 200-320 °C, cellulose at 315-400 °C, and lignin over a wide range at slow

rates. Morphological examination by scanning electron microscopy revealed increased surface roughness and a decrease in pectin volume with age. Tables I.6. and Tables I.7, along with Figure I.19, present the physical, thermal, mechanical, and chemical variations of roselle fibers at different maturation ages (3, 6, and 9 months).

Table I.6. Variation in the physical properties of roselle fibers with maturity age[267].

Physical properties	**Age of Roselle fiber (Months)**		
	3	**6**	**9**
Diameter (µm)	40-100	80-120	90-150
Density(g/cm^3)	1.33	1.14	1.42
Moisture content (%)	5.80	4.90	3.70
Water absorption	320	306	289

Table I.7. Thermal degradation characteristics of roselle fibers at different ages[267].

Months	**WL (%) at 30–110 °C**	**First degradation phase**			**Second degradation phase**			**Char residue(wt .%)**
		T_1 (°C)	**WL (%)**	**T_P (°C)**	**T_2 (°C)**	**WL (%)**	**T_P (°C)**	
3	10.28	220-350	-	346	350-400	76.36	364.05	10.31
6	8.25	200-315	14.16	298.01	315-390	62.27	363.24	14.21
9	4.10	210-320	15.21	298.58	320-390	63.69	366.08	12.24

WL: weight loss, **T_P**: peak temperature, **T_1**: First degradation temperature, **T_2**: Second degradation temperature.

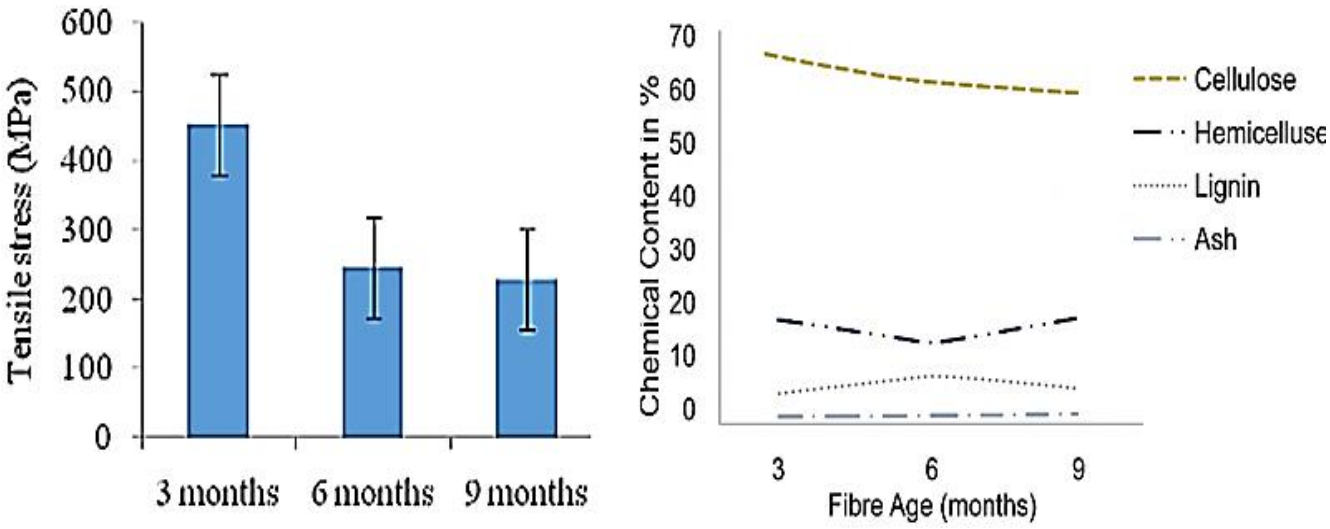


Fig I.19 Effect of maturation age on: a) tensile strength and b) chemical composition of roselle fibers[267].

I.5.3.1.1 Effect of seasonal changes and growth sites on plant fibers properties

Despite the limited number of studies investigating the effects of seasonal variations and growth locations on the properties and overall performance of plant fibers, several relevant research works have been gathered. These studies will provide a valuable foundation of knowledge to inform future strategies that aim to enhance our understanding of natural fiber behavior, improve their quality, and promote their sustainable use in various industrial applications.

Tarchi, et al. [268] conducted a comprehensive study on the Alfa fiber plant in the Sbeitla region of Tunisia, an area characterized by a semi-arid climate. The study focused on the chemical, morphological, and mechanical properties of the fibers, with particular emphasis on seasonal variations. The analyses included the measurement of several plant parameters such as length, diameter, density, and vegetation cover across different seasons. In addition, chemical analyses of Alfa leaves revealed seasonal fluctuations in cellulose, ash, and protein contents (Table I.8). The qualitative and mechanical characteristics of Alfa pulp used in papermaking were also evaluated, including dry matter content, basis weight, whiteness, breaking length, elongation, and ash content. The results further demonstrated that the particle size distribution of Alfa fibers plays a crucial role in determining their suitability for diverse industrial applications. These findings indicate that Alfa fibers exhibit distinct seasonal dynamics that significantly influence their quality and mechanical performance.

Table I.8. Physical properties of stipa tenacissima with seasons [268].

Components \ Season	Autumn	Winter	Spring	Summer
Cellulose (%)	42.8±2.61a	34.3±1.47d	40.26±1.25bc	41.16±0.86ab
Ash (%)	2.46±0.07a	2.36±0.34b	2.12±0.03c	2.41±0.08ab
Protein (%)	3.24±0.4c	4.47±0.97a	2.82±0.54d	3.55±0.02b

a-d: significant variation ($p < 0.05$).

The optimal harvest period was identified as summer, owing to the higher dryness, weight, and consistency of pulp properties recorded during this season. Conversely, fibers collected in spring and autumn exhibited superior whiteness, breaking length, elongation, and ash content attributes that are particularly advantageous for specific paper and industrial applications. Overall, these results confirm that controlling the harvesting season and the corresponding fiber characteristics can enhance the industrial potential of Alfa fibers in papermaking, composite manufacturing, and rope production, thereby promoting the efficient and sustainable utilization of this strategic natural resource.

The morphological analysis of Stipa tenacissima fibers conducted by Belkhir, et al. [269], which examined parameters such as fiber length, width, and surface roughness, revealed notable variations depending on both the growth location and the harvesting season .The study covered seven local sites in the Kasserine region (western central Tunisia) and spanned the four seasons of the plant's annual cycle. The results of the variance analysis demonstrated statistically significant variations in all the characteristics examined, influenced by factors such as location, season, and leaf level. The lowest values were found to occur in winter, with the highest values being recorded at full maturity in autumn. The analysis demonstrated a close correlation between fiber differentiation and the plant's biological cycle. Fibers are shortest and widest at the base of the leaf, gradually increasing in length and decreasing in roughness towards the upper parts. The results for raw leaf fibers were consistent with those for Alfa pulp, confirming the reliability of the analysis. As halfa leaves represent the sole source of fiber for paper production in Tunisia, these results are of practical importance in developing a harvesting strategy that is consistent with the specific biology of the plant and preserves the sustainability of the resource.

In order to fill the knowledge gap regarding spatial and seasonal effects on fiber characteristics, Istipliler, et al. [260]conducted a comprehensive study on cotton fibers in the Aegean region of Turkey. To achieve this, the researchers analyzed more than 368,000 fiber samples collected over seven years from three main regions (Bergama, Söke, Menemen), using High volume instrument) technology to measure basic characteristics such as length, strength, micronaire, and uniformity coefficient. The results showed clear variations between regions related to climatic conditions, with high temperatures during flowering and boll formation contributing to increased tensile strength, while excessive heat limited fiber length development. Differences in micronaire values were also associated with the combination of temperature and humidity during the boll development stages. The study concluded that fiber quality in the three regions remains within acceptable standards, but that environmental factors such as maximum and minimum temperatures, along with atmospheric humidity and north winds, directly affect fiber performance. These findings highlight the importance of considering the interaction between plant phenology and climatic factors to better understand changes in cotton quality, as well as the potential for using artificial intelligence tools for early market forecasting and identifying promising regions under climate change scenarios.

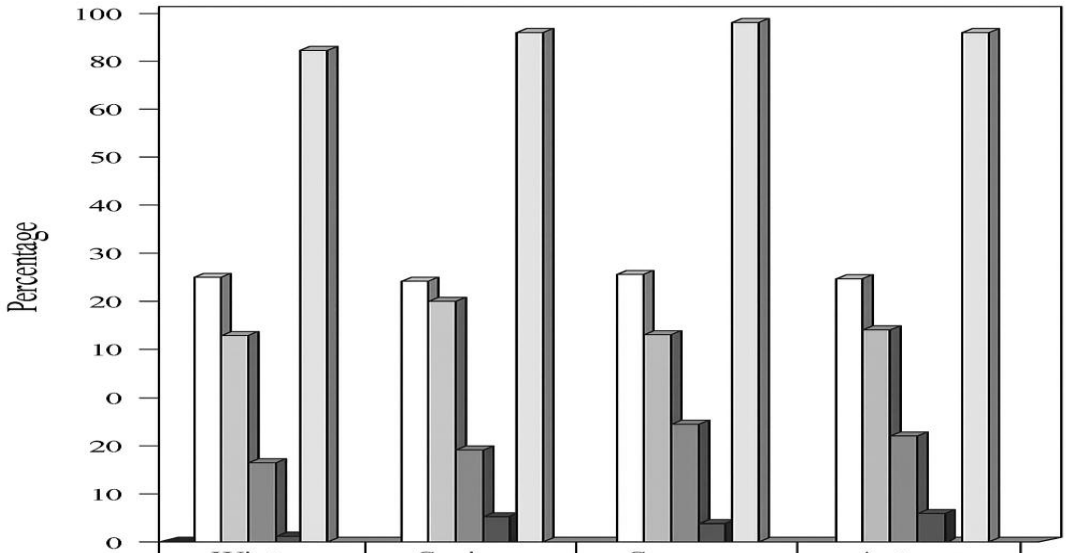
Pettolino, et al. [270]conducted a study to track the compositional changes in the cell walls of cotton fibers throughout their developmental stages in three species (*Gossypium barbadense, Gossypium hirsutum,* and *Gossypium arboreum*). The aim was to determine the influence of genetic and seasonal factors on polysaccharide biosynthesis and the resulting fiber properties. The three species were grown under controlled conditions during both summer and winter to assess the effect of variations in light radiation and temperature on cell wall development and the timing of secondary wall deposition .The results demonstrated that cell wall composition evolves progressively through the growth stages. Initially, the primary cell wall is rich in pectin and hemicellulose, while cellulose content is limited. As growth progresses, the non-cellulosic components gradually decrease, while there is a clear increase in crystalline cellulose and a decrease in pectin esterification, leading to wall hardening and elongation cessation.

In terms of seasonal effects, light intensity and duration were found to be the decisive factors in the timing of secondary cell wall deposition. Fibers harvested during the winter months exhibited a delay in secondary wall formation, enabling greater elongation. This process resulted in fibers that were thinner, longer, and less rigid. Conversely, the stronger light exposure during the summer months accelerated deposition, leading to fibers that were shorter, thicker, and more

rigid. Statistical analyses confirmed an inverse relationship between radiation intensity and fiber growth rate, highlighting the regulatory role of light in secondary wall synthesis.

The study concluded that the timing of secondary wall formation is the key determinant of fiber quality and that genetic or environmental control of this timing is an effective means of improving fiber properties and adapting them to climate change.

Mehdadi, et al. [15] conducted a study to assess seasonal changes in the cell wall composition of Stipa tenacissima L. leaves throughout the biological cycle in an arid region characterized by severe thermal and hydrological fluctuations. The results showed that the cell wall's chemical composition is clearly affected by seasonal changes (Figure I.20). It was found that the leaves of Alfa grass are low in pectin (1.05-1.09 %), while the percentage of hemicellulose was higher in spring (41.83 %), and the cellulose content peaked in summer (49.17 %). This occurred alongside an increase in lignin content, which rose from 8.40% in autumn to 12.97 %. Total fiber (the cumulative sum of the four components) also showed its maximum value in summer (97.39 %), reflecting the leaves' fibrous structure dominance during this dormant phase. Mineral analysis showed that major elements also vary seasonally.



	Winter	Spring	Summer	Autumn
Pectins	1,05	1,09	1,03	1,05
Cellulose	48,29	45,60	49,17	48,40
Hemicelluloses	3,84	41,83	34,22	36,19
Lignins	7,80	7,90	12,97	8,40
Total fibers	92,98	96,42	97,39	94,04

Fig I. 20 Seasonal variation in the parietal fiber composition of stipa tenacissima leaves[15].

The highest concentrations of potassium (5.13 %) and copper (0.45 %) were recorded in summer, while the maximum levels of calcium (3.75 %), iron (1.23 %), nitrogen (1.12 %) and

phosphorus (0.24 %) were reached in spring. This variation indicates a change in the palisade structure according to stages of activity, growth, and seasonal dormancy. These results demonstrate that seasonal changes drive the dynamics of wall component synthesis in algae in accordance with growth requirements and the dry environment.

I.6 Conclusion

A review of the literature has shown that plant fibers, as renewable cellulosic materials, are a promising alternative to synthetic fibers in composite materials due to their balanced chemical composition and unique morphological structure. This provides them with a combination of strength, lightness and flexibility. These properties are reflected in their physical, mechanical, and thermal performance, suitable for various industrial applications ranging from textiles and packaging to advanced materials such as building components and automotive parts.

The literature also shows that the performance of plant fibers is determined by the complex interaction of various factors, including environmental and climatic growing conditions, soil quality, plant age, harvest cycle and degree of maturity (pre-harvest factors), as well as post-harvest factors. Furthermore, various treatments, particularly chemical ones, play a pivotal role in modifying the fiber surface and enhancing interfacial bonding with the polymer matrix. The orientation of the fibers within the matrix has also been shown to be a key determinant of the final properties of the composites due to its direct influence on stress transfer efficiency and the material's resistance to failure under various loads. Therefore, a comprehensive understanding of these factors is essential for developing advanced strategies that can enable plant fibers to compete with synthetic fibers, particularly in high-performance applications that consider environmental sustainability.

However, the literature reveals key research gaps that warrant further attention. There is a lack of studies that systematically link growth factors, including seasonal climatic variations, growth locations and local environmental conditions, to the structural and functional properties of fibers. Although the chemical composition and mechanical and thermal properties of North African alfa fibers have received considerable attention, the impact of climatic conditions and growth factors on them, especially in Algeria, remains under-explored despite its considerable importance. Understanding this is essential for determining the optimal harvest time to ensure the desired characteristics are obtained and for accurately directing their use towards advanced industrial and engineering applications.

Chapter II: Materials and methods

Part I: Effect of growth conditions

Part II: Effect of chemical treatment and fiber architecture

II.1 Introduction

This chapter details the materials, tools and analytical techniques employed in this study. Given the diversity of analyses and experiments carried out, and in order to present them in a systematic, organized, and clear manner, the chapter has been divided into two main parts.

- In the first part (Effect of growth conditions), the raw material, Stipa tenacissima L, was collected from five different regions across Algeria during the four seasons of the year. These sites were carefully selected to represent diverse climatic environments, with the aim of studying how geographical factors and seasonal changes affect the characteristics of plant fibers.

The collection process was followed by the extraction of fibers from the Stipa stems, using a precise methodology that ensured the fibers obtained were pure and suitable for various analyses. Following extraction, the fibers were subjected to a series of morphological, physical, chemical and thermal studies. This included the use of a scanning electron microscope (SEM) to study the surface structure, Fourier transform infrared spectroscopy (FTIR) to identify functional groups, and X-ray diffraction (XRD) to assess the degree of crystallinity. Quantitative chemical analysis was also performed according to a rigorous scientific protocol based on sequential steps to identify the basic components of the Alfa plant, along with density, diameter, and tensile strength tests to evaluate the physical and mechanical properties of the fibers. These procedures aim to determine the extent to which climatic factors and geographical location affect the structural and mechanical performance of Alfa fibers.

- In the second part (Effect of chemical treatment and fiber architecture), the focus was placed on enhancing the performance of Stipa tenacissima. Alfa fibers harvested from the M'sila region through chemical surface modification. Three types of fibers were used: Untreated (A_{Un}), treated with sodium hydroxide (A_N), and treated with potassium permanganate (A_P) Alfa fibers.

The prepared samples underwent a series of physical, mechanical, and morphological analyses to evaluate the influence of each treatment on the fiber properties. The tensile strength and interfacial shear strength (IFSS) between the treated and untreated Alfa fibers and the polyester resin were determined using the Micro bond test, supported by Weibull statistical analysis to assess variability and treatment effects. Additional analyses included density measurements, surface morphology observation via Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FTIR) for functional group identification, and X-ray Diffraction (XRD) for structural evaluation.

Subsequently, plain-woven intra-ply hybrid fabrics were prepared by combining treated and untreated Alfa fibers with jute yarns. In parallel, unidirectional mats were produced using untreated Alfa fibers. These reinforcements were then used to fabricate polyester-based composite materials following a controlled manufacturing process designed to ensure homogeneous fiber distribution and proper resin impregnation.

The resulting composite samples were subjected to comprehensive mechanical and physical tests, including: Three-point bending test, Tensile strength test, Density and void content measurement, along with microscopic examination of cross-sections using SEM to assess fiber–matrix interfacial bonding quality.

Finally, an additional statistical evaluation was carried out to validate the reliability of the tensile strength data for both fibers and composites. This analysis provided a deeper understanding of the correlation between chemical treatment type, fabric architecture, and the mechanical performance of the resulting hybrid composites.

Part I: Effect of growth conditions

II.2 Materials

II.2 .1 Alfa plant

Alfa plant (Stipa tenacissima L.), more commonly known as esparto grass, is a perennial grass that grows in dense clumps. It is native to the semi-arid regions of the Mediterranean basin, particularly North Africa (e.g., Tunisia, Algeria, and Morocco) and southern Europe[271]. It belongs to the Poaceae family and grows in steppe ecosystems characterized by low rainfall (200-400 mm per year), high temperatures and nutrient-poor soil. The plant is a dense grass (Figure II.1) characterized by narrow, fibrous leaves (25–120 cm long) containing high amounts of cellulose and hemicellulose .making it suitable for use in the manufacture of paper and fiber-reinforced composites. The plant forms dense clusters called (Alfa grass plains), which stabilize the soil against erosion and support biodiversity in fragile ecosystems[272].

Figure II.1 Alfa grass plant.

II.2 .2 Sampling regions and environmental characteristics of Alfa growing sites

In this study, Alfa plants were harvested in 2022 from five representative sites in Algeria, namely Tiaret, Laghouat, Djelfa, Boussaada, and Maadid. These sites are located within the natural distribution area of Stipa tenacissima L. The geographical location of these sites is illustrated in Figure II.2. These regions were specifically selected owing to the high abundance of Alfa grass and the recognized quality of its fibers, which makes them particularly suitable for systematic sampling.

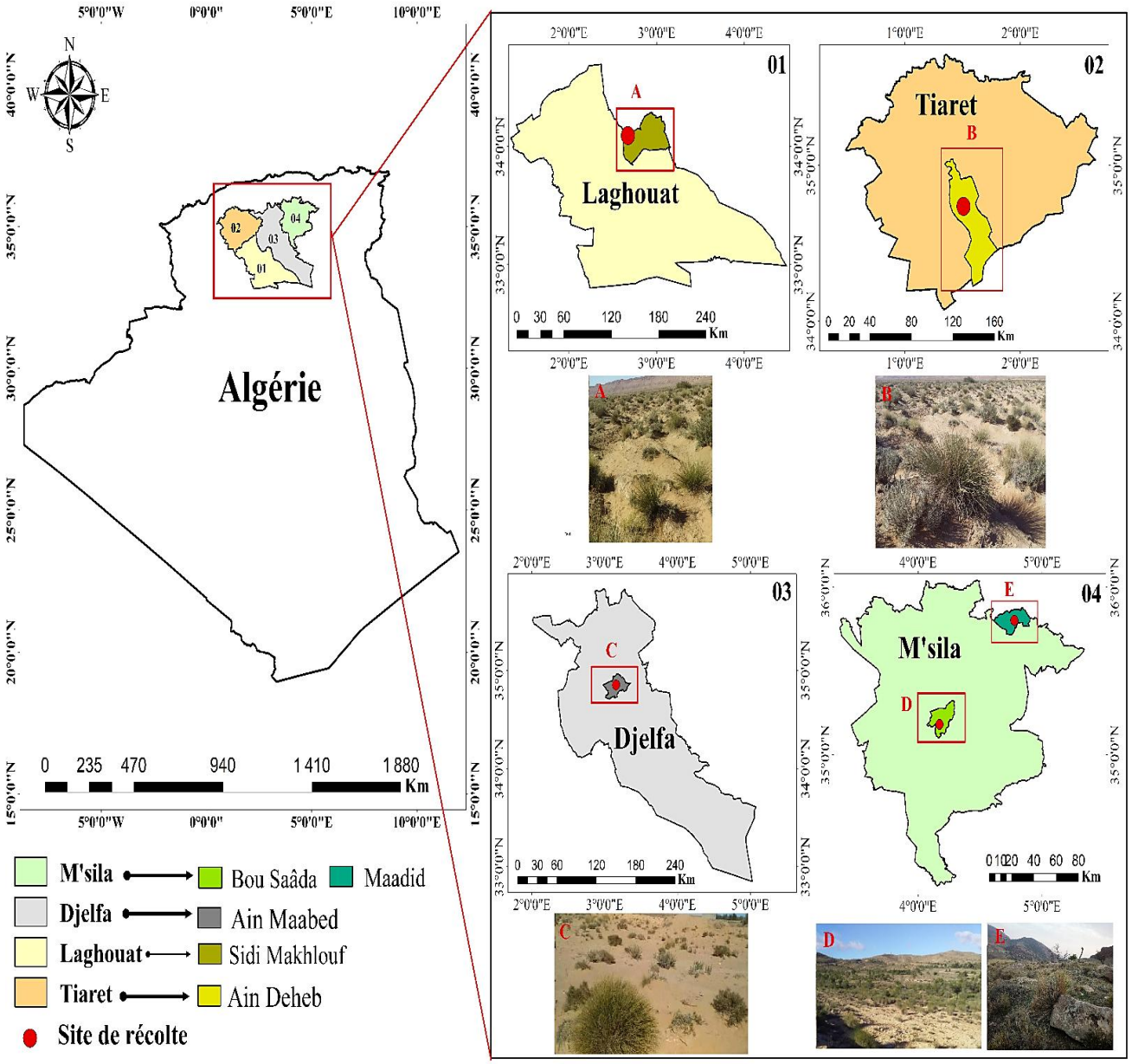


Fig II.2 Geographical map of the selected growth sites of Alfa plant samples.

To account for seasonal variability, plant materials were collected during the four main phases of the biological cycle: winter (mid-January), spring (mid-April), summer (mid-July), and autumn (mid-October).

This approach ensured that both spatial and temporal variations were adequately represented. The main environmental characteristics of the selected sites, including climatic and edaphic parameters, are summarized in Table II.1.

Table II. 1. Climatic characteristics of the studied growth sites in 2022 (data extracted from NASA/POWER database).

Seasons / Sites	Winter				Spring				Summer				Autumn (Fall)			
	AT (°C)	ARH (%)	AWS (m/s)	AP (mm/d)	AT (°C)	ARH (%)	AWS (m/s)	AP (mm/d)	AT (°C)	ARH (%)	AWS (m/s)	AP (mm/d)	AT (°C)	ARH (%)	AWS (m/s)	AP (mm/d)
Tiaret	9.36	66.46	13.15	16.82	15.18	62.1	16.93	71.00	26.65	36.41	13.46	6.73	18.42	54.21	13.92	28.87
Laghouat	10.51	58.37	14.48	6.90	14.98	62.15	16.74	82.13	29.20	23.26	12.27	3.34	19.2	47.68	12.52	24.04
Djelfa	9.92	61.57	14.65	6.96	14.63	54.92	16.46	37.29	28.04	25.42	11.7	1.10	18.97	50.22	12.16	26.3
Bousaada	11.85	64.82	14.90	17.21	16.41	57.69	15.25	39.97	30.58	29.75	12.25	0.96	21.84	51.65	12.04	37.92
Maadid	11.85	64.82	14.90	17.21	16.41	57.69	15.25	39.97	30.58	29.75	12.25	0.96	21.84	51.65	12.04	37.92

AT: Average temperature, ARH: Average relative humidity, AWS: Average wind speed, AP: Average precipitation.

II.2 .2 Extraction of Alfa fibers

Following harvesting, the stalks of Alfa plant were carefully separated by hand, focusing on selecting specimens that were straight and free of structural defects that could affect fiber quality. The selected stalks then underwent an initial cleaning stage using a 2% cleaning solution to remove surface dirt and impurities, including dust, soil, and other plant debris. This stage is an essential step to ensure the purity of the samples before they undergo further processing.

After cleaning, the stems were soaked in water for one month at room temperature. This step is necessary to break down non-cellulosic components, particularly hemicellulose and lignin, facilitating the separation of the fibrous fibers from the surrounding plant tissue[273]. At the termination of the soaking stage (water retting), the stems were washed repeatedly to remove dissolved or decomposed materials, and then a mechanical extraction process was carried out using a metal brush to gradually separate the fine fibers without damaging their structural integrity. The resulting fibers were then placed in an oven at 60 °C for six hours to dry them and ensure their structural stability by reducing their moisture content. The extraction process yielded fibers ranging in length from 50 to 60 cm, which were then ready for various subsequent tests. The basic stages of the extraction process are shown in Figure II.3.

Fig II.3 Steps of Alfa fiber extraction.

II.3 Fiber analysis

II.3 .1 Anatomical analysis of Alfa leaves

The anatomical study of Stipa tenacissima leaves was conducted through the preparation of transversal sections using a rotary microtome. The section thickness ranged from 250 to 300 µm, allowing adequate observation of the internal tissue organization. To enhance the contrast and facilitate the identification of cellular structures, the sections were subjected to a double-staining technique with safranin and fast green.

Following staining, the sections were mounted on glass slides using glycerin as a mounting medium to ensure sample preservation. The prepared slides were then examined under a light microscope (OPTIKA B-350), where multiple micrographs were taken. The most representative images were selected to illustrate the anatomical features of Alfa leaves.

II.3 .2 Chemical analysis

The objective of this analysis was to quantify the main chemical constituents of Stipa tenacissima leaves, specifically cellulose, hemicelluloses, lignin, extractives, and mineral residues (ash content). Alfa Plant was collected from five distinct growth sites and across different seasons

to account for potential environmental variations. The determination of these components was carried out according to the standardized protocols described by Rabouhi, et al. [274] Rowell, et al. [275], and Bulut and Aksit [276].For each growth site and season, three independent samples were analyzed, and the mean values of the replicates were calculated to ensure accuracy and reproducibility.

The main steps involved in the extraction of the chemical components of Alfa fibers (Figure II.4) include sample collection and drying at controlled temperatures (105 °C) to determine the moisture content, followed by cutting the fibers into small segments (3 to 5 mm). Soluble compounds (extractives) are first removed using a Soxhlet apparatus with an appropriate solvent system, such as an ethanol/acetone mixture. Subsequently, alkaline treatment (NaOH) or mild oxidation ($NaClO_3$/acetic acid) is performed to isolate cellulose from hemicellulose and lignin. The ash content is determined by combustion in a muffle furnace under controlled temperature conditions. Throughout these procedures, precision instruments such as analytical balances, glass condensers, Büchner funnels, rotary evaporators, and drying ovens are employed, with repeated washing using distilled water and careful control of experimental parameters (temperature, duration, etc.).

II.3 .2 .1 Cellulose rate

For the determination of cellulose content, one gram of chopped sample was subjected to chemical treatment using a 1.72 % sodium chlorite solution (98 %), to which a few drops of concentrated sulfuric acid (98 %) were added in order to initiate the reaction. The mixture was then refluxed for one hour, after which the excess liquid was carefully removed. The remaining solid residue was neutralized by the addition of concentrated ammonia solution and subsequently washed several times with distilled water to ensure the complete removal of residual reagents. The purified fraction was finally dried under controlled conditions and weighed. The cellulose content was expressed as the mass of the recovered residue relative to the initial dry sample weight (II.1), providing a reliable measure of cellulose concentration[277].

$$\text{Cellulose } \% = \frac{M_R}{M_d} \times 100 \qquad \text{(II.1)}$$

where M_R is the weight of residue (g) and M_d is the weight of dry sample (g).

II.3 .2 .2 Hemicellulose rate

To quantify the hemicellulose fraction, one gram of sample was first oven-dried at 100 °C for four hours to ensure complete removal of moisture. The dried material was then subjected to alkaline extraction using a 4% sodium hydroxide solution (MW = 40 g/mol) at room temperature for one hour. The resulting residue was subsequently neutralized with a 5% hydrochloric acid solution (98 %), thoroughly rinsed with distilled water, and dried under controlled conditions. The final dried residue was weighed, and the hemicellulose content was calculated according to the established formula(II.2), providing a reproducible estimation of this structural polysaccharide[276].

$$\text{Hemicellulose } \% = [(m_0 - m_f) / m_0] \times 100 \qquad \text{(II.2)}$$

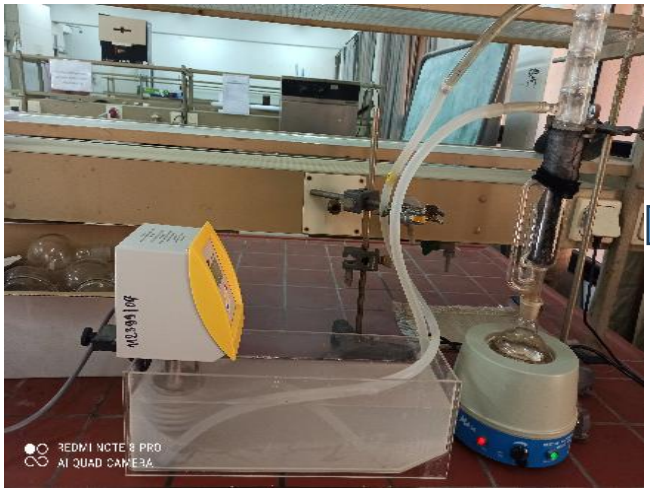
Where m_0 and m_f represent the initial and final weights of Alfa fibers, respectively.

II.3 .2 .3 Lignin rate

To determine the lignin content, 1 g of sample was soaked in 12.5 ml of 72 % sulfuric acid (98 %) for 1 hour at room temperature, with gentle stirring every 15 minutes. The solution is then diluted by adding 300 ml of distilled water. After 2 hours, the solution is filtered under a vacuum and rinsed with distilled water until a neutral pH is achieved. Finally, the sediment is dried to obtain a constant mass, representing the lignin amount[278].

II.3 .2 .4 Extractives rate

To evaluate the extractives content, a representative mass of Alfa leaves was placed in a Soxhlet extraction cartridge lined with cotton. The extraction was carried out using a binary solvent system of ethanol (96 %, MW = 46.07 g/mol) and acetone (MW = 58.08 g/mol) under reflux for 24 h to ensure the exhaustive recovery of soluble compounds. After completion, the solid residue was carefully rinsed several times with fresh solvent, followed by sequential drying, firstly under ambient conditions, then in a ventilated oven at 105 °C for 12 h to achieve constant weight. The dried sample was finally weighed on a high-precision analytical balance (±0.00001 g). The proportion of extractives was subsequently determined according to the following equation[277]:



Extractives extraction process

Extraction of hemicellulose

Extraction of lignin

Vacuum filtration of cellulose

Obtained Cellulose content

Fig II.4 Main stages and equipment used in the extraction and determination of the chemical components of Alfa fibers.

$$Extractives\ \% = \frac{m_i - m_{fi}}{m_i} \times 100 \qquad (II.3)$$

Where m_i and m_{fi} represent the initial and final weights of Alfa fibers, respectively.

II.3 .2 .5 Mineral matter

The determination of mineral content (Ash) in Stipa tenacissima fibers was carried out by combusting a pre-weighed amount of sample at 800 °C for a duration of four hours. The remaining inorganic residue, referred to as ash content, corresponds to the total mineral matter present in the fibers. The mineral fraction was quantified by applying equation (II.4), which relates the mass of the residual ash to the initial dry weight of the analyzed sample[278].

$$Ash\ \% = \frac{m_R}{m_0} \times 100 \qquad (0.4)$$

Where m_0 and m_R represent the initial and residue (ash) weights of Alfa fibers, respective

II.3 .3 Structural analysis

II.3 .3 .1 Fourier Transform Infrared Spectroscopy

Fourier Transform Infrared Spectroscopy (FTIR) is a prominent non-destructive technique used to identify the functional groups in materials based on their absorption of infrared radiation at specific wavelengths, corresponding to molecular vibrations. In plant fiber studies, particularly bast and leaf fibers, FTIR provides qualitative and quantitative information on major constituents such as cellulose, hemicellulose, lignin, and extractives. Spectra in the 4000-400 cm^{-1} range reveal characteristic absorption bands of –OH, –CH, and C=O groups, reflecting the chemical structure of the fibers. This technique also enables monitoring of structural changes induced by treatments like delignification or alkaline modification, making it essential for characterizing and optimizing natural fiber composites.

In this study, the powdered fiber samples were analyzed using Attenuated Total Reflectance - Fourier Transform Infrared Spectroscopy (ATR-FTIR) . Each sample was placed directly on the ATR crystal and gently pressed with a dedicated clamp (FigureII.5) . The spectra were recorded using an Agilent Technologies Cary 630 spectrometer equipped with a KBr beam splitter, at a spectral resolution of 2 cm^{-1}.

Fig II.5 ATR-FTIR device used for fiber spectroscopic analysis.

II.3 .3 .2 X-ray Diffraction

X-ray diffraction (XRD) is a widely used, non-destructive analytical technique for characterizing the crystalline structure of materials. Based on the principle of elastic scattering of X-rays by regular atomic planes, it results in characteristic diffraction patterns that allow accurate information about the internal structure to be extracted. In the case of natural fibers such as Stipa tenacissima L. the crystallinity index (CrI) is one of the most important structural parameters affecting physical, chemical, mechanical and thermal properties. High values are typically associated with increased tensile strength, stiffness and improved thermal stability.

In this study, XRD analysis of Alfa fibers was performed using a Philips X'Pert Pro SW device (Figure II.6) with a wavelength of 0.154 nm. The crushed samples were analyzed under operating conditions of 40 kV and 30 mA. The spectra were recorded at room temperature (24 °C) in the (2θ) angle range between 10° and 70° with a step of 0.02°. To determine the crystallinity index, the empirical approach of Segal [279] was adopted, based on the mathematical relationship between crystalline peak intensity (I_{002}) and amorphous phase intensity (I_{am}), as shown in equation (0.5). Furthermore, XRD enables the tracking of structural changes resulting from chemical or physical treatments, such as alkali treatment or bleaching. This provides a valuable insight into improving fiber performance in industrial and composite applications.

$$\mathrm{CrI}\% = \left(\frac{I_{002} - I_{am}}{I_{002}}\right) X100 \qquad (0.5)$$

Where, I_{002} represents the maximum intensity of the crystalline peak, while I_{am} denotes the peak corresponding to the amorphous phase.

Fig II.6 X-Ray Diffraction (XRD) Machine.

II.3 .4 Thermal analysis

II.3 .4 .1 Thermogravimetric analysis

Thermogravimetric analysis (TGA-DTG) is among the most widely applied techniques for characterizing the thermal behavior of materials. It provides information on thermal stability, moisture content, activation energy, maximum decomposition temperature, ash residue, as well as the reactions that occur during heating. In this study, the TGA-DTG characterization of Alfa fibers was carried out using a TA-60WS thermal analyzer (Shimadzu Corp., Kyoto, Japan) (Figure II.7). The experiment was conducted at a constant heating rate of 10 °C/min, under an inert nitrogen atmosphere (80 mL/min), within a temperature range of 30-700 °C.

II.3 .4 .2 Activation energy (Ea)

Several methods are available for deriving kinetic parameters from TGA data, one of which is the determination of the activation energy (Ea), representing the minimum energy required to initiate fiber degradation. Evaluating the thermal response of natural fibers and predicting their degradation pathway is an essential step in assessing their suitability as reinforcements in polymeric composites. In this work, the activation energy corresponding to the main degradation stage (cellulose decomposition) was calculated following the Broido method [280]. The Ea value

is obtained from the slope of the straight line (-Ea/R), derived from the linear relationship between ln[ln(1/y)] and 1/T and 1/T1/T, as expressed in the following equation:

$$ln\left[ln\frac{1}{y}\right] = -\frac{Ea}{R}\left[\left(\frac{1}{T}\right) + K\right] \quad (0.6)$$

where y = wt/w0, with wt being the residual sample mass at time t, and w0 the initial mass. Ea refers to the apparent activation energy; R is the universal gas constant (8.314 KJ/mol.K); T is the absolute temperature in Kelvin, and K is a constant.

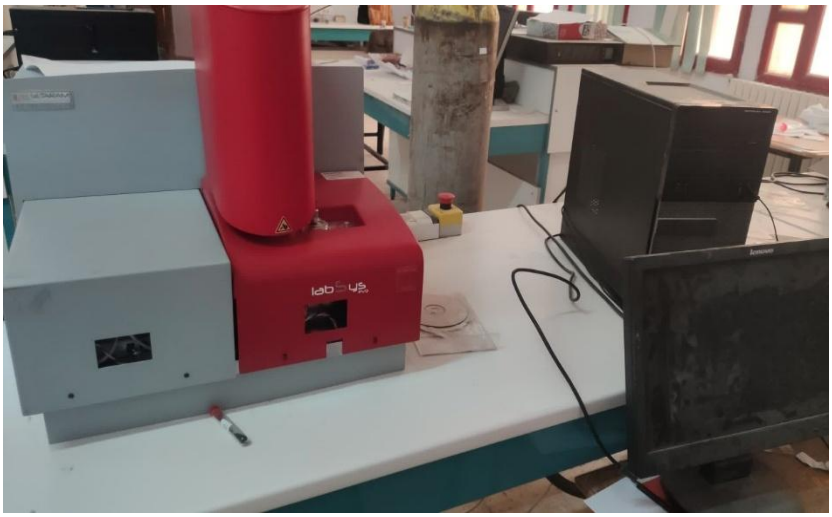


Fig II.7 TGA: Thermogravimetric Analyzer.

II.3 .5 Physical characterization

II.3 .5 .1 Moisture Content

The determination of moisture content (MC) was carried out in accordance with ASTM D4442. Approximately 5 g of Alfa fiber samples were oven-dried at 105 °C, with the heating process repeated until a stable mass was achieved. The percentage of moisture was then calculated based on the weight loss of the samples, using the following relationship[278] :

$$mc\ (\%) = \frac{m_0 - m_f}{m_0} \times 100 \quad (II.7)$$

where m_0 is the initial mass of the fibers before drying, and m_f corresponds to their final constant dry weight.

II.3 .5 .2 Density measurement

Density constitutes a critical physical property in the characterization of plant fibers,

The density of Alfa fibers harvested from five different regions during the year was assessed using the pycnometer technique in accordance with ASTM D2320-98. In this method, the pycnometer with a capacity of 50 ml was first filled with methanol (ρ=0.791g/cm3 at 25 °C) (Figure II.9). About 1 g of fiber fragments, cut to lengths of 5 to10 mm and pre-dried at 60 °C until their moisture level was reduced by 5% [281] , was then introduced. The arithmetic mean of five measurements of the apparent fiber density was calculated using equation (0.8)[282]:

$$\rho_{AF} = \frac{\Delta m_1}{\Delta m_2 - \Delta m_3}\rho_M \qquad (0.8)$$

where ρ_{AF} is the apparent density of Alfa fibers, Δm_1 denotes the mass difference between the pycnometer containing chopped fibers and the empty one, Δm_2 is the mass difference between the pycnometer filled with methanol and the empty pycnometer, and Δm_3 represents the mass difference between the pycnometer containing both methanol and chopped fibers and the one filled with methanol only. ρ_M refers to the methanol density (0.791 g/cm³ at 25 °C).

Fig II.8 Pycnometer used for density measurement.

II.3 .5 .3 Linear density

The determination of the linear density of Alfa fibers was performed by weighing more than 30 individual fibers, each cut to a length of 200 mm, in order to obtain a representative mean value. Measurements were carried out using a high-precision analytical balance with an accuracy of 0.00001 g. The linear density, expressed in Tex, was evaluated according to the following relation:

$$D = \frac{M}{L} \qquad (0.9)$$

Where M is the fiber mass (mg) and L is its length (m).

The cross-sectional area of the fibers was determined based on the equation (0.10) [20]:

$$S_f = \frac{M}{\rho_{AF} \cdot L_f} \qquad (0.10)$$

Where M represents the fiber mass, L_f the fiber length, and ρ_{AF} is the apparent density of Alfa fibers.

II.3 .5 .4 Diameter of fiber

Fiber diameter is a fundamental property of plant fibers, as it directly influences their mechanical behavior, such as tensile strength and flexibility. It also determines porosity and interfacial bonding with matrix materials in composites, thereby affecting the overall mechanical performance of the final product.

Measuring the diameter of plant fibers is a crucial step in material characterization, although it remains challenging due to the significant variability in the shape and size of the extracted fibers. To achieve a more accurate estimation, a computational approach was adopted, assuming that the fibers have a cylindrical geometry. Based on this assumption, the fiber diameter was determined using equation (0.11):

$$D_{\mathrm{AF}}(\mathrm{cm}) = \sqrt{4 * \frac{M}{\pi \rho_{\mathrm{AF}} L_f}} \qquad (0.11)$$

Where: D_{AF}: Nominal Alfa fiber diameter (cm), L_f: Alfa fiber length (cm), M: Alfa fiber mass (g) and ρ_{AF}: Alfa fiber apparent density (g/cm³) .

We can also derive the equivalent mean diameter from the apparent density (ρ_{AF}) and linear density values, according to the following expression [283]:

$$D_e(\mu m) = \sqrt{4 \cdot \frac{D(mtex)}{\pi} \cdot \rho_{AF}\left(\frac{g}{cm^3}\right)} \qquad (0.12)$$

II.3 .6 Mechanical characterization of fiber

II.3 .6 .1 Tensile test

A universal testing machine (Zwick/Roell Z100) was used to evaluate the mechanical behavior of Alfa fibers, focusing on tensile strength, Young's modulus, and elongation at break. The preparation of specimens followed the methodology reported by Bouchareb et al. [25]. To ensure proper fixation of each Alfa fiber between the clamps, the samples were mounted on a paper frame using an adhesive, following the preparation method illustrated in Figure II.9. Tensile experiments were performed under static loading at a crosshead speed of 2.5 mm/min, employing a 5 kN load cell and a gauge length of 40 mm. Since the mechanical response of plant-based fibers is strongly influenced by parameters such as extraction technique, fiber maturity, experimental setup, diameter irregularities, and surface imperfections, a set of 35 representative Alfa fibers was tested. The tensile strength (σ) was determined according to ASTM D3379-75, using Equation (II.13).

$$\sigma = F/S_f \qquad (II.13)$$

where F is the breaking load (N) and S_f is the mean cross-sectional area of the fiber (mm²).

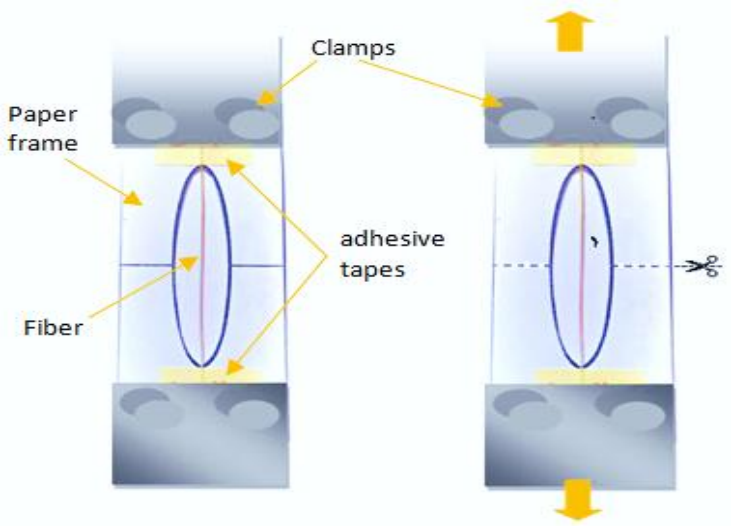


Fig II.9 Specimen Preparation for Single-Fiber Tensile Tests.

II.3 .7 Scanning electron microscopy

The scanning electron microscope (SEM) is one of the most prominent modern technologies in materials science, allowing for the analysis of surfaces and microstructures with nanometer-level precision. The device utilizes a beam of electrons to generate high-resolution images that reveal the morphological and microstructural properties of a sample. SEM is a highly accurate analytical tool that is employed in a variety of fields, including materials science, biology, engineering, and nanotechnology.

This research aims to examine the influence of seasonal variation and collection sites on the morphological characteristics of Alfa fibers. To evaluate the surface morphology, the fibers were analyzed using a Prisma E Scanning Electron Microscope (SEM). This device is notable for being the first SEM to integrate a tungsten source with advanced chemical analysis technology, allowing simultaneous acquisition of topographical and elemental information. During the analyses, the microscope was operated with an emission current of 2 µA, an analytical working distance of 10 mm, and an accelerating voltage adjustable between 200 V and 30 kV. Table II.2 summarizes the designation of Alfa fibers as a function of their collection sites and harvest seasons.

Table II.2 Designation of various Alfa fibers according to growth sites and season.

	Season			
growth sites	Winter	Spring	Summer	Autumn (Fall)
Tiaret	ATW	ATSP	ATS	ATF
Laghouat	ALW	ALSP	ALS	ALF
Djelfa	ADW	ADSP	ADS	ADF
Bousaada	ABW	ABSP	ABS	ABF
Maadid	AMW	AMSP	AMS	AMF

Part II: Effect of chemical treatment and fiber architecture

II.4 Materials

II.4.1 Alfa fiber

In this second part, Alfa fibers collected from the M'sila region were used. The same extraction procedure described previously was employed during the fiber extraction process. The resulting fibers are referred to as untreated Alfa fibers.

II.4.2 Chemical treatment

The extracted Alfa fibers were subjected to two distinct chemical treatments (Figure II.10): the first using sodium hydroxide (NaOH) and the second employing potassium permanganate ($KMnO_4$).

II.4.2.1 Alkali treatment Alfa fibers

Alfa fibers were immersed in an aqueous solution containing 5 % sodium hydroxide (NaOH) for 24 hours. The purpose of this process was to remove impurities and non-cellulosic substances such as lignin and hemicellulose, thereby enhancing fiber purity and increasing cellulose accessibility. Following the immersion period, the fibers were thoroughly rinsed several times with distilled water to eliminate any residual alkali. Surface neutralization was then carried out using a dilute acidic solution of 2 % sulfuric acid for 10 minutes in order to completely remove traces of NaOH. These steps were integral to the process of ensuring that the obtained fibers were chemically stable and sufficiently purified. This, in turn, made them suitable for subsequent characterization and analysis. The resulting fibers are referred to as alkali-treated (NaOH) Alfa fibers (A_N).

II.4 .2.2 Permanganate treatment

Following the completion of the preliminary alkaline treatment of the fibers with a sodium hydroxide solution (NaOH 5 %), the fibers underwent a subsequent treatment stage using a potassium permanganate solution ($KMnO_4$). The fibers were immersed in a low-concentration aqueous solution (0.033 %) for a maximum of three minutes. Following treatment, the fibers were washed several times with distilled water until the medium stabilized at a pH of approximately 7, to ensure that any residues of the oxidant or reaction by-products were removed. In the final stage, the fibers were placed in a drying oven at a constant temperature of 60 °C for six hours. The obtained fibers are designated as potassium-permanganate-treated Alfa fibers (A_P).

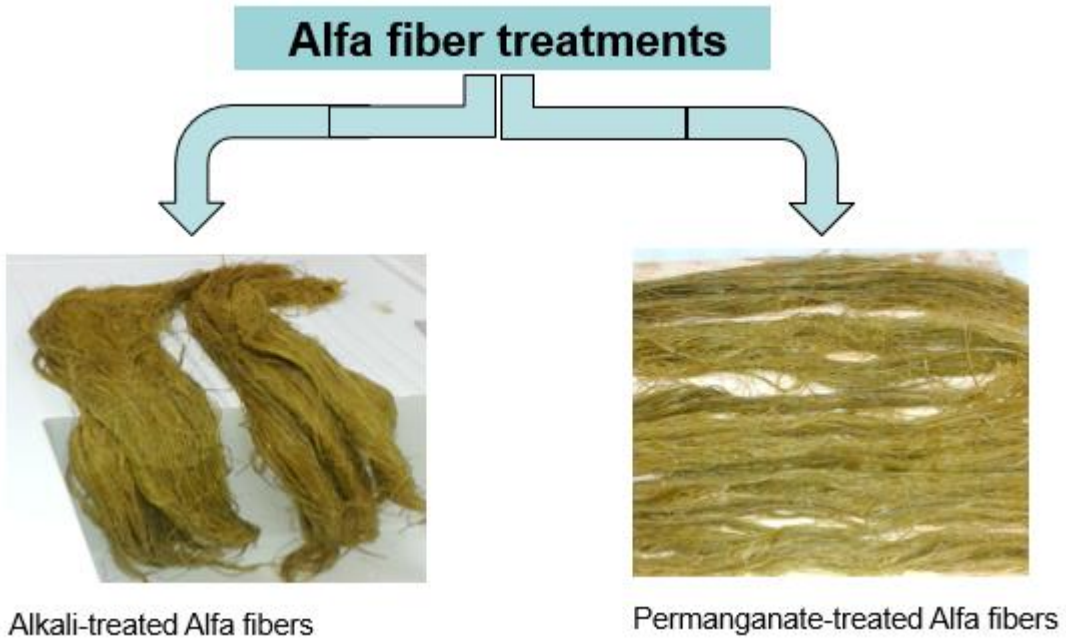


Fig II.10 Treated Alfa fibers.

II.5 Fiber characterization methods

In this section, a comprehensive characterization of three types of Alfa fibers was conducted: untreated, treated with sodium hydroxide, and treated with permanganate. This study aimed to examine the impact of chemical treatments on the structural and functional properties of these fibers. This characterization relied on the same analytical protocols as indicated in the first part, where identical preparation procedures, measurement tools, and operating conditions were followed to ensure methodological consistency among the different samples and to avoid any variability arising from differences in treatment or measurement techniques. The characterization included the determination of apparent density, linear density, and fiber diameter, as well as attenuated total reflectance Fourier-transform infrared spectroscopy (ATR-FTIR) to identify the characteristic functional groups, and X-ray diffraction (XRD) to evaluate crystallinity index. Furthermore, tensile tests were performed to measure tensile strength, Young's modulus, and elongation at break. Scanning Electron Microscopy (SEM) was also employed to investigate the surface and internal morphology of the fibers, providing high-resolution imaging that reveals the microstructural features, pore distribution, and surface regularity. These observations supplied essential data to link the mechanical and chemical properties with the structural behavior of the fibers. Altogether, this set of analyses enabled the establishment of a precise and comprehensive database regarding the differences induced by each treatment, thereby allowing a coherent comparative approach that supports the objectives of the present study.

II.5 .1 Interfacial characterization test

II.5 .1 .1 Micro-droplet test

The micro-gouttelette test is a widely used micromechanical technique for characterizing the interfacial properties between a polymeric resin matrix and single filaments[284]. In this method, a microdroplet of resin is deposited on an individual fiber and subsequently cured. The fiber is then subjected to a controlled tensile loading, during which the embedded droplet is pulled off. From the force required to detach the droplet and the embedded length, the interfacial shear strength (IFSS) can be determined, providing a quantitative measure of the adhesion quality between fiber and matrix. This test is particularly suitable for evaluating the influence of fiber surface treatments or chemical modifications on the fiber–matrix bonding, and it offers valuable insight into the micromechanical performance of composite materials.

In this work, focus was given to evaluating the influence of chemical treatments on the interfacial behavior of Alfa fibers treated with alkali and permanganate solutions. The microdroplet test was carried out on a minimum of thirty specimens for each fiber category (A_{Un}, A_N, A_P). Small resin droplets were deposited onto individual Alfa filaments (Figure II.11), allowed to dry under ambient conditions, and then cured in an oven at 70 °C for 2 hours.

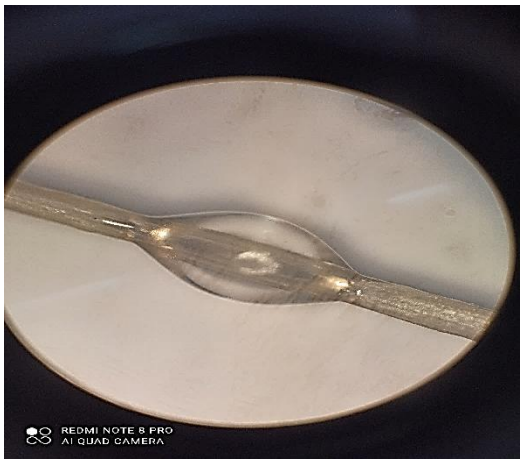


Fig II.11 Preparation of polyester microdroplets on treated and untreated Alfa fibers.

The droplets that adhere most closely to the fiber surface are selected, ensuring that the fiber is well-centered within them and that their volume is evenly distributed across the contact area This configuration guarantees uniform stress transfer during the micro-droplet test. Controlling the droplet dimensions is crucial, as the study comparison of Methods for the Measurement of fiber-matrix Adhesion in composites [285] reported an optimal droplet diameter ranging between 80

and 200 µm. Before conducting the mechanical tests, the fiber diameter and the embedded droplet length were precisely measured using an optical microscope (MOTIC). The interfacial experiments were then carried out on a Zwick Z005 universal tensile testing machine. To avoid undesired displacement of the resin droplet during the test, two sharp blades were carefully placed on either side of the fiber adjacent to the droplet. Finally, the fiber was subjected to tensile loading at a constant crosshead speed of 0.5 mm/min until complete debonding occurred between the fiber and the resin matrix, as shown in Figure II.12.

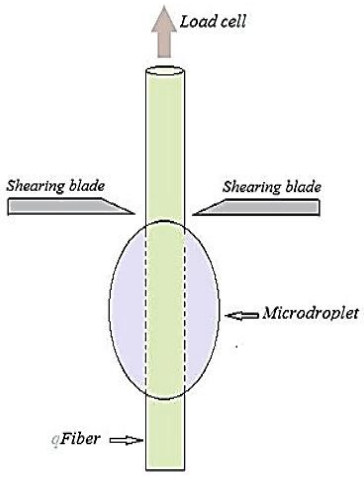


Fig II.12 Preparation of micro-droplet test specimens.

The interfacial shear strength (IFSS), denoted as τ, was determined using Equation (II.14)[286]:

$$\tau = \frac{F_{max}}{D_f \cdot \Pi \cdot L_e} \qquad (0.14)$$

where, F_{max} represents the highest pull-out load recorded, L_e corresponds to the length of the fiber embedded within the resin droplet, and D_f refers to the mean diameter of the Alfa fiber.

II.6 Composite materials manufacturing

Two kinds of composite materials are developed in this study:

- Bidirectional composite materials.
- Unidirectional composite materials.

Both composites are primarily based on a UP matrix combined with Alfa fiber reinforcements. Two reinforcement architectures were employed: unidirectional plies and intra-layer woven fabrics.

II.6 .1 Bidirectional composite materials

II.6 .1.1 Polyester resin

For both composites, unsaturated polyester resin (UP) was used. It is one of the most widely used thermosetting polymers in composite manufacturing. It is produced through the polymerization of unsaturated polyester monomers, commonly in combination with styrene. When an organic initiator such as methyl ethyl ketone peroxide (MEKP) is added, a chain polymerization reaction begins, transforming the resin from its liquid state into an irreversible solid form.

The curing time depends on the proportion of hardener, which typically ranges between 1-2 % of the resin mass. Such concentrations ensure controlled polymerization and prevent the occurrence of defects or undesired deformations in the final structure. Styrene acts as a reactive diluent to reduce viscosity and maintain resin fluidity, while the resin must be stored in sealed metallic containers, away from light and in a cool environment to preserve its stability.

In this study, UP resin was selected due to its cost-effectiveness and high performance. It demonstrates strong interfacial adhesion with natural fibers in addition to favorable thermal and mechanical properties. Moreover, its ability to cure efficiently at room temperature simplifies processing, making it an ideal choice for composites intended for large-scale applications. The physicomechanical properties of the unsaturated polyester (UP) resin are presented in Table I.1.

II.6 .1.2 Intra-layer woven fabrics

In the composite materials industry, woven and nonwoven fabrics are among the most commonly used types of fabric for commercial purposes [155]. Woven fabrics have a regular structure consisting of warp and weft threads that are interlaced in a crisscross pattern. This gives them an orderly, architectural form. In contrast, nonwoven fabrics are characterized by a random arrangement of fibers within their structure. These structural differences directly affect their mechanical properties: the repeating patterns in woven fabrics, such as plain, twill and satin, improve stiffness, fracture resistance and damage tolerance compared to nonwoven fabrics. The weaving process itself is a traditional textile technique involving the perpendicular interlacing of warp and weft threads to form the fabric. This gives the final product distinctive structural and mechanical properties, qualifying it for use in advanced composite applications.

In this study, three distinct types of plain-woven intra-layer hybrid fabrics were manufactured by altering the composition of the auxiliary Alfa fibers (A_{Un}, A_N, A_P), while preserving the utilization of jute yarns as warp yarns in all fabrics, as illustrated in Figure II.13. The use of Alfa

fibers in the weft direction and jute fibers in the warp direction results in a balanced combination of stiffness and elasticity, thereby ensuring the optimal performance of the hybrid fabric. This design is consistent with the primary objective of the study, which is to improve the interconnection between the fibers and the matrix through chemical treatments. The well-branched nature of Alfa fibers contributes to better resin penetration within the fabric structure, which has a positive impact on the performance of composite materials [287].

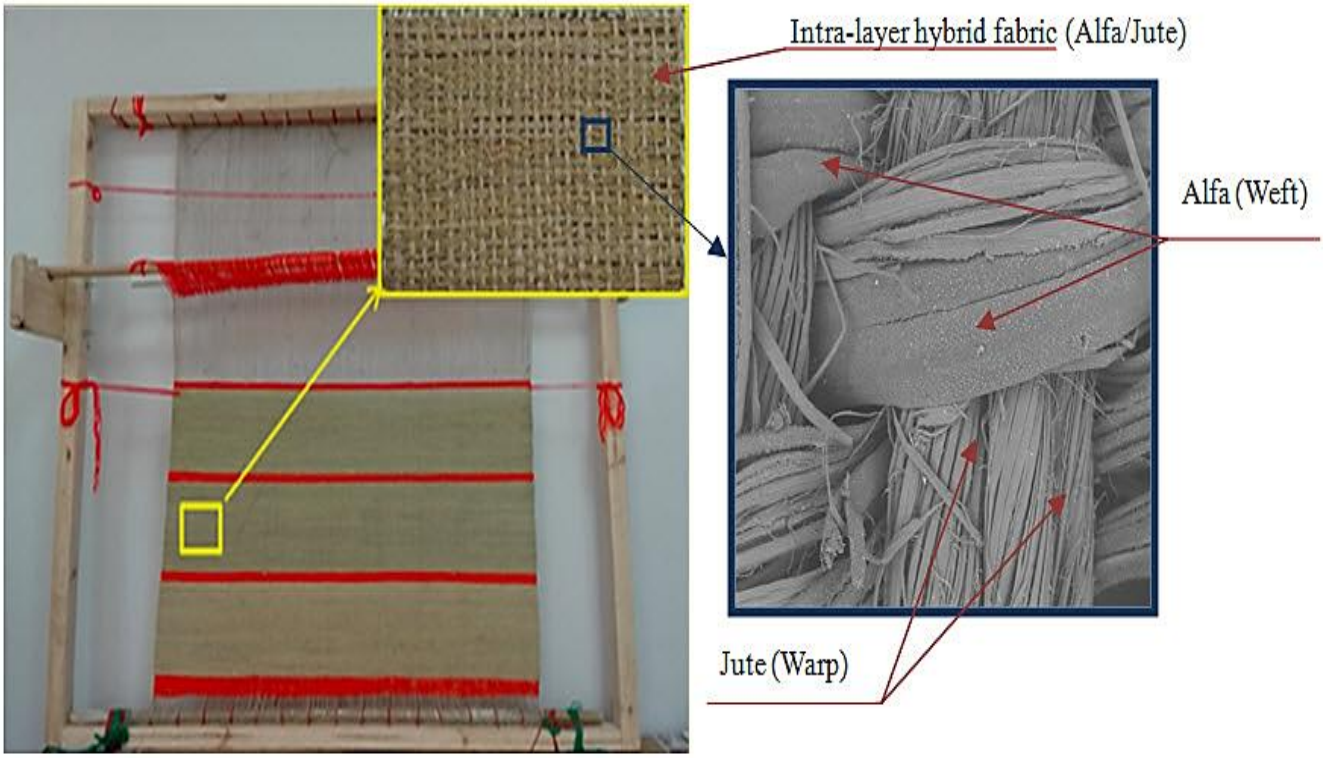


Fig II.13 Production of plain-woven intra-layer hybrid fabrics[34].

Each plain-weave hybrid fabric was woven by passing the weft fibers (weft threads) horizontally over and under the warp threads (warp threads), which were arranged and secured vertically on a wooden frame using a special tool to guide the warp threads. The process of interlacing warp and weft threads is fundamental to the creation of diverse patterns and textures, including plain, twill, and satin, as well as more complex designs. However, the present study focused on plain weave for several reasons, the most significant of which is that this configuration provides good tensile properties [33], in addition to being easy t to prepare [288], which contributes to accelerating the process of manufacturing samples with high precision. Plain weave is also characterized by its structural stability, which reduces the variable factors that may affect the interactions of the fibers with the polymer matrix. Furthermore, it ensures a homogeneous

distribution of fibers within the layers, thereby enhancing the homogeneity of the composite material. It is noteworthy that red woolen threads were incorporated during the weaving process to visually distinguish between different fabrics, as shown in Figure II.14.

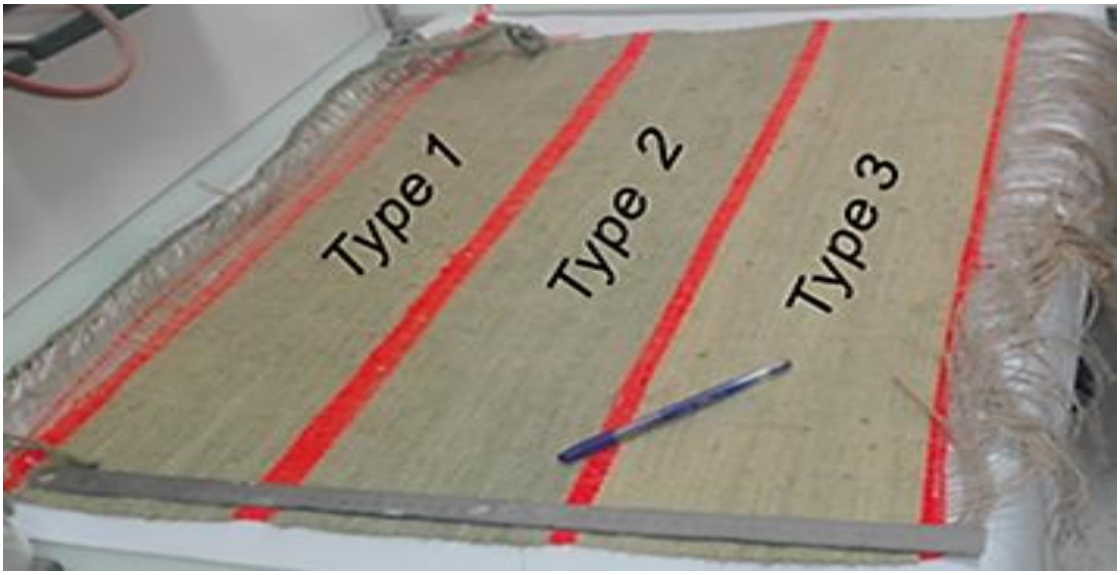


Fig II.14 Types of plain-woven intra-layer hybrid fabrics: type1 (A_{Un}/J); type 2 (A_N/J); and type 3 (A_P/J).

II.6 .1.3 Preparation of bidirectional composites

in this research, four types of composite laminates are manufactured using vacuum infusion molding process. This method has significant advantages over traditional methods, such as ensuring complete fiber impregnation, reducing internal defects (air bubbles), limiting resin loss, and lower equipment costs. Before the manufacturing process, the fabric folds were subjected to heat compression at 80°C for 10 minutes under 5 bar pressure (Figure II.15), in order to remove wrinkles and reduce thickness, ensuring uniform sheets.

Fig II.15 Simple manual compression press.

The process commences with the preparation of the mold. This involves cleaning the surface of the mold and applying a thin layer of wax to facilitate the subsequent removal of the sheets. Two rectangular layers (160 × 180 mm²) of fabric reinforcements (woven fabrics), are then carefully placed on the glass surface. The mold is then closed with a plastic film secured around its perimeter with adhesive tape (Figure II.16), with special tubes installed for the inlet and outlet of the resin, ensuring the system is airtight.

When the vacuum pump is activated, air is drawn from within the mold, thereby generating pressure that enables the flow of ortho-phthalic polyester resin. This resin is notable for its cost-effectiveness and ease of use, as well as its ability to be processed at room temperature [289]. The resin gradually spreads between the fiber layers, ensuring complete saturation and preventing the formation of air bubbles that could affect the quality of the composite panel. Following injection molding, the mold is then left under vacuum throughout the initial polymerization period until it has hardened over 24 hours at room temperature. Additional heat treatment in an oven at 60°C for 6 hours then ensures the rigidity of the board. Figure II.16 illustrates the vacuum infusion setup used in this study. For comparative purposes, the composite material was reinforced with two plies of jute arranged in a taffeta weave, having a surface density of 150 g/m². The fiber weight fractions, volume fraction and the corresponding designations of each composite formulation are presented in Table II.3.

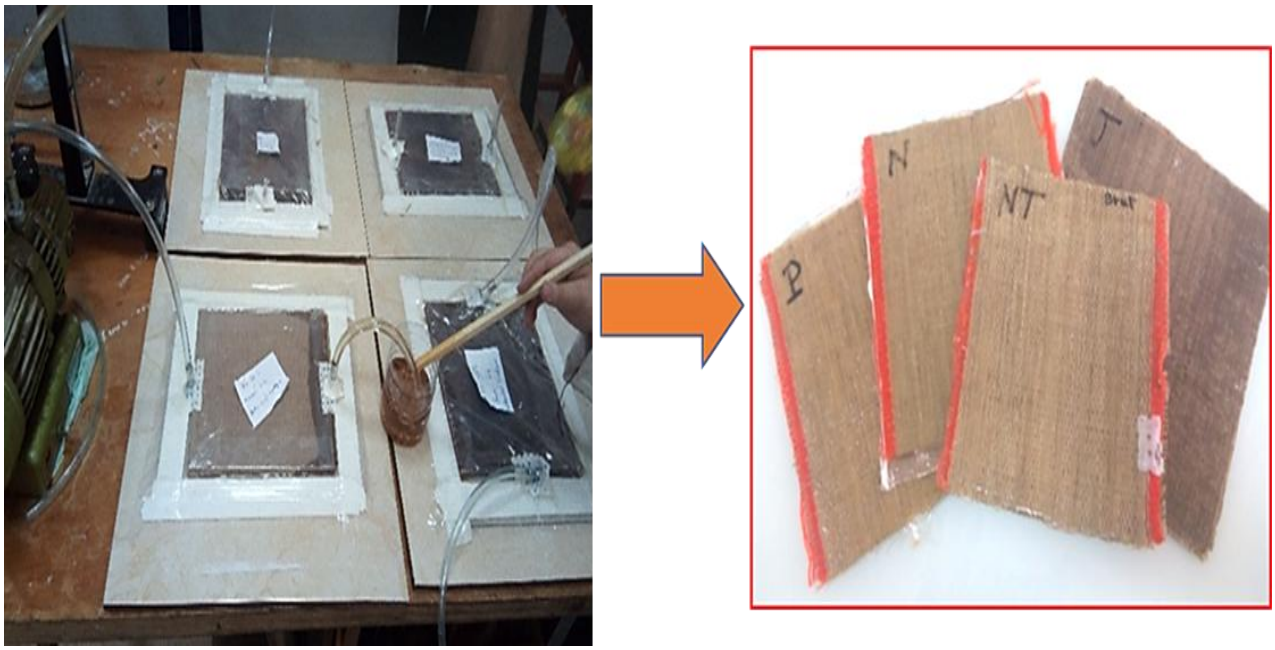


Fig II.16 vacuum infusion process for various composites.

Table II.3. Designation of different types of prepared bidirectional composites.

Intra-ply woven fabrics	Weight fraction (%)	volume fraction (%)	Designation of composites (*)
Intra-ply woven fabric (A_{Un} / J)	18.219	12.112	Comp-A
Intra-ply woven fabric (A_N / J)	17.666	8.942	Comp-B
Intra-ply woven fabric (A_P / J)	20.217	20.221	Comp-C
Woven fabric (J / J)	25.901	30.412	Comp-D

(*) Resin: Polyester

II.6 .1.4 Preparation of composite samples

The sample preparation process begins by cutting the composite sheets to predetermined dimensions, in accordance with the standard specifications approved for each mechanical test (such as the relevant ASTM or ISO specifications). Cutting is performed using a saw equipped with a diamond disk, with continuous water lubrication to reduce friction and heat build-up, ensuring dimensional accuracy, surface quality, and preservation of the composite's properties. This is followed by careful smoothing of the edges and surfaces to remove any imperfections or burrs that could adversely affect the test results. The dimensions are then measured using high-

precision standard measuring instruments, such as a vernier caliper, to verify that they comply with the required specifications. Finally, the samples are marked with unique serial numbers to facilitate tracking and documentation during the various stages of testing. Four samples are prepared for each type of compound, as shown in Figure II.17, thereby enhancing the reliability and validity of the results.

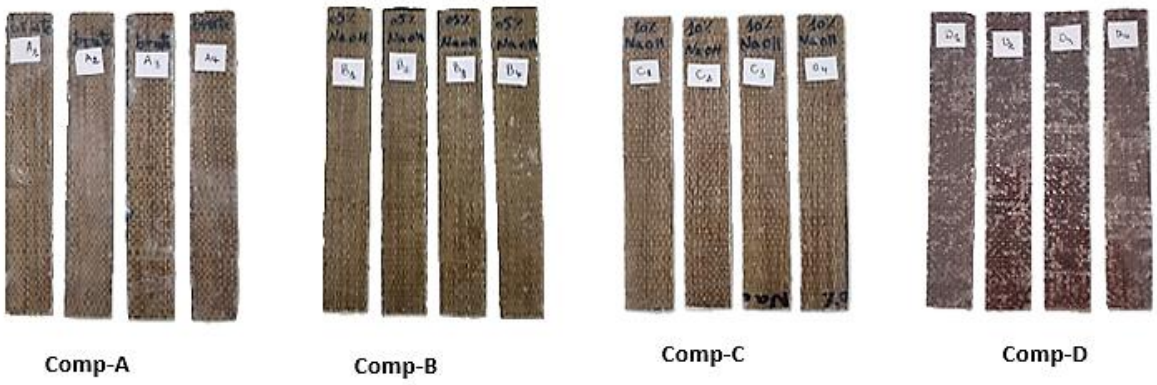


Fig II.17 Representative bidirectional composite samples.

II.6 .2 Unidirectional composite materials

II.6 .2 .1 Unidirectional Alfa ply

Unidirectional Alfa ply was prepared prior to the composite manufacturing process. First, defect-free fibers were carefully selected and cut into uniform lengths of 250 mm. They were then precisely weighed using a high-sensitivity electronic balance to determine the required amount in relation to the fiber-to-matrix ratio. Subsequently, the fibers were manually aligned in a single direction, ensuring parallel and homogeneous orientation for an even fiber density distribution. Finally, the bundles were fixed on both sides with adhesive tape, resulting in stable and coherent plies ready for the subsequent manufacturing stage (Figure II.18).

Figure II.18 Unidirectional Alfa fiber ply.

II.6 .2.2 Preparation of unidirectional composites

For unidirectional composites, UP was used as the matrix, while unidirectional Alfa plies served as the reinforcement. It is important to note that the Alfa plies were subjected to thermal pressing to remove wrinkles and ensure a uniform thickness, as previously applied to the different intra-ply fabrics. The unidirectional composite material was fabricated following the same protocol previously applied for the bidirectional material composites as illustrated in Figure II.19. The fiber weight fraction, volume fraction and the corresponding designation of unidirectional composite formulation are summarized in Table II.4.

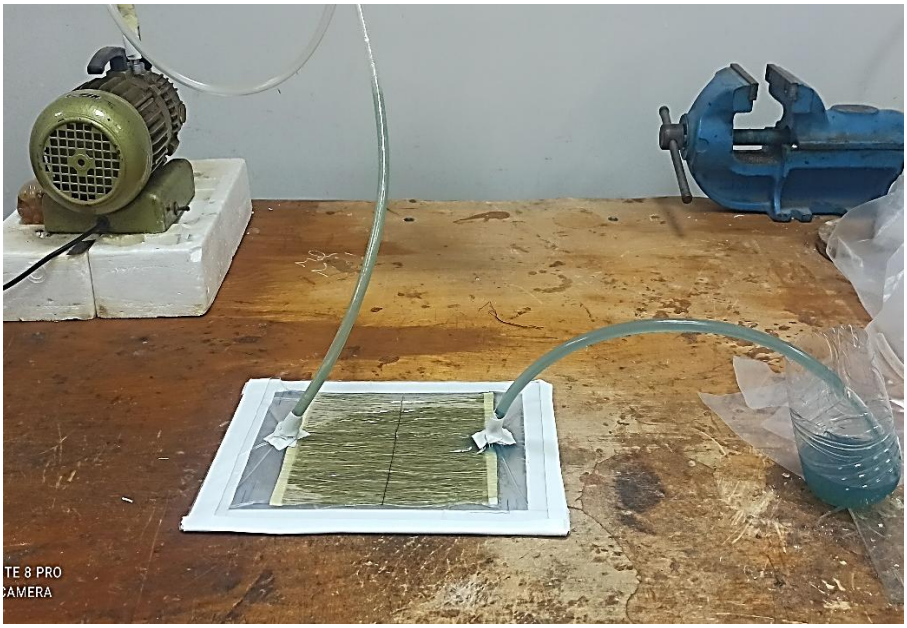


Fig II.19 Fabrication process of the unidirectional composite laminate.

II.6 .2.2 Preparation of composite samples

Unidirectional composite samples were prepared following the same procedure previously adopted for the bidirectional composite samples. The fabrication was carried out under identical experimental conditions and according to the same preparation standards. Four unidirectional composite specimens (FigureII.20) were produced for subsequent physicomechanical characterization.

Fig II.20 Representative unidirectional composite samples.

Table II.4. Designation of prepared unidirectional composites.

Reinforcement architecture	Weight fraction (%)	volume fraction (%)	Designation of composites (*)
unidirectional Alfa ply	15.731	12.435	Un-Comp

(*) Resin: Polyester

II.7 Characterization of bidirectional and unidirectional composites

II.7 .1 Density and void content

The apparent density of the different composite materials was measured in accordance with ASTM D-792 [290]. Rectangular specimens with dimensions of 30 mm × 20 mm were used and immersed in distilled water at room temperature as the reference fluid. The mass of each specimen was recorded using a high-precision digital balance with a resolution of 10^{-5} g, ensuring the accuracy and reliability of the measurements. To improve the representativeness of the results, six specimens from each type of composite both intra-ply woven fabric and unidirectional ply reinforcement composites were tested ,and the average value was calculated.

The void content was determined following ASTM D-2734 [290], using Equations (II.15)and (II.16):

$$Void\ contents\ (\%) = \frac{\rho_{theoretical} - \rho_{experimental}}{\rho_{theoretical}} \times 100\% \qquad (0.15)$$

$$\rho_{\text{theorethical}} = \frac{1}{\left(\frac{w_f}{\rho_f} + \frac{w_m}{\rho_m}\right)} \qquad (0.16)$$

Where W_f and W_m represent the weight fractions of the fiber and the matrix, respectively, while ρ_f and ρ_m denote their corresponding densities.

This analysis is fundamental for evaluating the internal porosity of composites, which is a key indicator of manufacturing quality and has a direct influence on the mechanical and physical properties of the materials.

II.7 .2 Tensile test

Tensile tests were conducted to determine the mechanical properties of various composite materials. These tests were performed on five categories of standard samples. For the purposes of this study, parallelepiped samples with dimensions of (150×25×3) mm^3.

The different specimens were subjected to tensile tests to evaluate their tensile strength, elongation at break, and elastic modulus, in accordance with ASTM D3039 [291] . The tests were performed using an INSTRON 5969 universal tensile/bending testing machine with a maximum load capacity of 50 kN, operating at a constant crosshead speed of 1 mm/min, as shown in Figure II.21.

The different samples of each type of composite were stretched to failure along the Alfa fibers direction. The arithmetic mean of the measured values was taken as an indicator to represent the mechanical properties of each composite.

II.7 .3 Three-point bending test

The three-point bending test is a standardized method used to evaluate the mechanical properties of materials subjected to flexural loads, particularly composites and polymers. In this test, a concentrated load is applied at the midpoint of a beam supported at both ends, using a universal testing machine of the type Instron 5969, operating at a constant crosshead speed of 1 mm/min. The support span was set at 50 mm, allowing the measurement of both flexural strength and sample deformation. The geometry and dimensions of the test specimens were defined according to ASTM D3039 [51], with rectangular specimens of (60 × 10 × 3) mm^3. These dimensions were mainly based on the specimen thickness h, while maintaining the standard span to thickness ratio L/h = 16. To ensure the reliability of the results, four specimens were tested for each condition, and the average values were recorded.

Fig II.21 Tensile/bending machine. INSTRON 5969 50 kN.

The flexural fracture stress in the three-point bending test was calculated using the following equations, in accordance with ASTM D790:

$$\sigma_f = \frac{3\,F\,L_s}{2\,b\,h^2} \qquad (0.1)$$

$$E_f = \frac{{L_s}^3\,m_s}{4\,b\,h^3} \qquad (0.2)$$

$$\varepsilon_f = \frac{6\,w\,h}{{L_s}^2} \qquad (0.3)$$

Where σ_f: Flexural strength at fracture (MPa), E_f: Flexural modulus of elasticity (GPa), ε_f : Flexural strain (%), F: Applied load (N), L_s: Support span length (mm), b and h: Width and thickness

of the specimen, respectively (mm), *w*: Maximum mid-span deflection mm (Maximum deflection at the center of the beam), m_s: Slope of the linear elastic region of the load-deflection curve (N/mm).

II.7.4 Morphological analysis of composites

To investigate the morphological characteristics of the fiber-matrix interfacial region, cross-sectional samples of the different composite materials were analyzed using the same scanning electron microscope previously employed in the morphological study of the various Alfa fibers.

II.8 Statistical analysis

As is common with natural fibers, the measurements exhibited considerable variability. Therefore, a Weibull statistical analysis was performed to assess the reliability and scatter of the data obtained from both the micro-droplet interfacial tests and the tensile tests of intra-ply hybrid fabric composites[292].

The data were fitted using the two-parameter Weibull distribution [293], expressed by the following relation:

$$F(\sigma) = 1 - \exp\left(-\frac{L_{ref}}{L_0}\left(\frac{\sigma}{\sigma_0}\right)^m\right) \quad (0.20)$$

Which can be linearized as:

$$ln\left(ln\left(\frac{1}{1-F(\sigma)}\right)\right) - ln\left(\frac{L_{ref}}{L_0}\right) = m(ln\sigma - ln\sigma_0) \quad (0.4)$$

Where $F(\sigma)$ is the probability of fiber failure, L_0 is the initial length, L_{ref} the reference length, σ_0 the characteristic strength, σ the applied stress, and m the Weibull modulus (shape parameter), which reflects the variability in strength values.

The distribution parameters were estimated by linear regression using the median rank method, defined as:

$$F(\sigma) = \frac{i-0.3}{N+0.4} \quad (0.5)$$

With N being the total number of samples and i the rank of the i_{th} data point.

II.9 Conclusion

In this chapter, which was devoted to presenting the materials, techniques, and experimental methods adopted in this research work, a comprehensive and coherent scientific methodology has been outlined in direct alignment with the study's objectives.

The first part focused on the investigation of Alfa grass collected from five different regions across Algeria during the four seasons of the year, with the aim of analyzing the influence of climatic and geographical factors on the properties of the extracted fibers. This was achieved through precise extraction protocols and the use of advanced analytical techniques such as Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FTIR), and X-ray Diffraction (XRD).

The second part examined the effect of chemical treatments—using sodium hydroxide and potassium permanganate—on improving the performance of Alfa fibers harvested from the M'sila region. Mechanical, structural, and morphological tests were carried out, in addition to the fabrication of hybrid polymeric composites to assess the efficiency of treated fibers and their suitability for composite applications.

Through this systematic organization, this chapter provides the experimental and scientific foundation for the discussion and interpretation of results in the following chapter, paving the way for a comprehensive understanding of the natural and chemical factors influencing Alfa fiber properties and their potential for the development of sustainable, high-performance composite materials.

Chapter III: Results and discussions

Part I: Effect of growth conditions

Part II: Effect of chemical treatment and fiber architecture

III.1 Introduction

This chapter presents the results of the various analyses and experiments carried out during this research work, with a focus on their discussion and interpretation in light of the scientific objectives of the study and in comparison, with relevant previous research. Given the diversity and large number of results obtained, and in order to ensure a systematic, organized, and clear presentation, this chapter has been divided into two main sections.

The first part provides a detailed presentation and analysis of the results concerning the effect of growth conditions, including climatic factors, seasonal variations, and geographical location on physical, chemical, mechanical, and morphological properties of Alfa fibers collected from five different regions in Algeria during the four seasons.

This analysis allows for a deeper understanding of how the Alfa plant adapts to different environmental conditions and how these factors influence the quality and suitability of its fibers for industrial and composite applications. Such insights represent a key step toward optimizing the utilization of this natural resource and promoting its integration into sustainable and advanced engineering materials.

The second part of this chapter presents and analyzes the results related to the effect of chemical treatment on the performance of Alfa fibers plant harvested from the M'sila region, focusing on their physical, mechanical, and morphological properties through tensile, interfacial shear, spectroscopic, microscopic, and structural analyses.

It also includes the presentation and discussion of the mechanical, physical, and morphological properties of the composite materials manufactured from various hybrid intra-ply fabrics and unidirectional plies reinforced with unsaturated polyester, in order to determine the influence of fiber architecture within the matrix on the overall composite performance.

Part I: Effect of growth conditions

III.2 Anatomical analysis of Alfa plant

In cross-section of esparto (Alfa) leaf a well-organized tissue architecture is evident, as shown in Figure III.1. The leaf is surrounded by a compact epidermal layer that functions as a protective barrier, beneath which lies parenchymatous tissue composed of large, rounded cells that envelop the vascular bundles. Each vascular bundle consists of xylem, oriented toward the inner side, and phloem, oriented toward the outer side, and these bundles are surrounded by a distinct sheath of supportive fibrous (sclerenchymatous) cells. The fiber cells exhibit thick primary and secondary walls and a narrow central lumen, while the middle lamella remains as an intercellular linkage between cells ,features that distinguish them from the parenchymatous cells, which have thin walls and wide lumina. As shown in panel (b), the fibers are aggregated into longitudinal, parallel bundles that run along the length of the leaf; this structural arrangement provides the leaf with the stiffness and flexibility required for mechanical support. The distribution of fibers around the vascular bundles and within the cortex directly contributes to the mechanical reinforcement of the leaf and enables the plant to withstand environmental stresses, particularly drought conditions. This anatomical organization of the Alfa stem aligns with observations reported in previous studies on other natural fiber-producing plants [294].

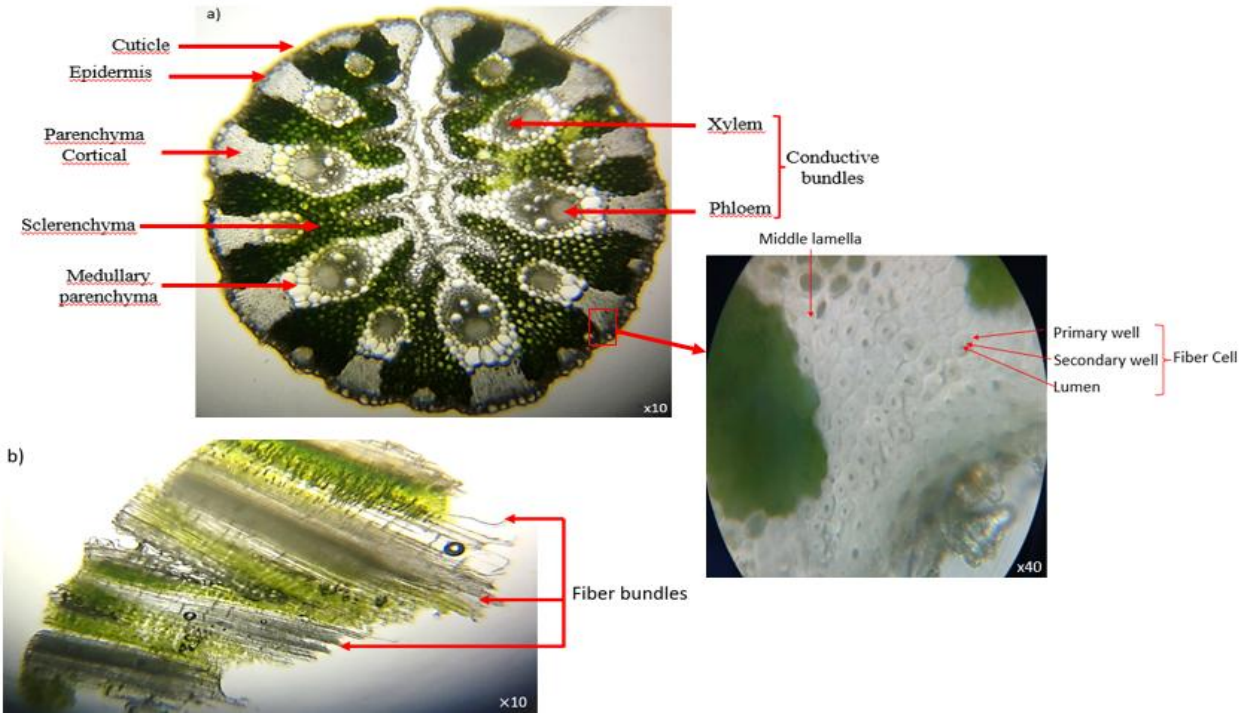


Figure III. 2 Anatomy of Stipa tenacissima L leaf: (a) magnified view showing vascular bundles and fibrous tissues; (b) overall structure showing fiber bundles.

III.3 Chemical characterization

In this section, the chemical composition of Alfa fibers was determined according to the previously established protocol. Table III.1. presents the average relative proportions of the chemical components of Alfa leaves, collected from five different locations in Algeria (Tiaret, Laghouat, Djelfa, Boussaada, and Madid) during four seasons.

The structural components of Alfa plant such as cellulose, hemicellulose and lignin play a key role in determining the quality and performance of the fiber. Cellulose, as the most prominent component, is the main supporting structure of the fiber, giving it strength and durability. Hemicellulose, due to its amorphous nature, improves its flexibility [295] and lignin acts as a natural binder between the fibers, making them highly resistant to heat and various environmental factors [296]. Accurate quantitative analysis of these components, in addition determining the proportions of secondary compounds such as extractives and ashes, is an essential procedure for assessing the quality of fibers and their suitability for industrial use, whether in manufacturing of composite materials, textile products, or other advanced applications.

The results of Table III.1 show that a significant variation in the chemical composition of Alfa leaves, which appears to be influenced by seasonal changes and differences in growth sites. This observation aligns with findings from studies such as those by Mehdadi, et al. [17] and Belkhir, et al. [16], which reported notable seasonal variations in the biochemical composition of Esparto leaves.

III.3.1 Cellulose content

As shown in Table III. 1, there is a marked discrepancy in the cellulose content of Alfa leaves, which can be attributed to geographical location and seasonal variations. Notably, the highest cellulose content was recorded during spring across the five studied regions, with the Laghouat region, situated in the semi-arid superior bioclimatic stage, exhibiting the peak value at Laghouat 51.42± 1.35 %. This was followed by Tiaret at 50.95 ±0.73 %, Boussaada at 50.03 ±0.92 %, Djelfa at 49.52 ±0.67 %, and Maadid, also in the semi-arid superior stage, at 49.18 ± 1.33 %. These findings are consistent with the findings of Belkhir, et al. [14], who observed higher cellulose content in Alfa leaves from semi-arid upper bioclimatic zones.

Table III. 1. Seasonal variations in the chemical composition of Alfa leaves (% dry weight) from five different growth sites.

Sites / Seasons	Tiaret	Laghouat	Djelfa	Boussaada	Maadid
	Cellulose (%)				
Winter	39. 45±0.51	40.72 ±0.22	39.19 ±1.03	40.01 ±1.73	40.08 ±0.56
Spring	50.95 ±0.73	51.42± 1.35	49.52 ±0.67	50.03±0.92	49.18 ± 1.33
Summer	42.87±1.12	41.79±0.33	41.01±0.27	41.32±0.57	42.63±1.78
Autumn (Fall)	42.51±1.50	41.93±0.45	42.79±1.02	43.89±0.34	41.32±0.24
	Hemicellulose (%)				
Winter	23.83±0.5	22.77±0.51	23.05±0.31	22.23±1.43	22.43±1.55
Spring	20.29±0.29	20.54±0.63	20.97±1.45	19.31±1.09	21.25±1.33
Summer	20.15±1.23	20.12±1.12	20.01±0.22	20.11±0.50	21.08±0.72
Autumn (Fall)	22.87±1.15	21.85±0.41	23.16±1.32	21.18±0.46	22.15±0.25
	Lignin (%)				
Winter	24.54±1.03	23.90±0.53	24.07±1.17	25.26±0.42	24.34±1.90
Spring	19.96±0.42	20.17±0.22	19.08±1.12	19.03±1.26	19.42±0.36
Summer	26.01±0.57	27.08±0.89	26.19±0.31	26.54±1.15	26.11±1.38
Autumn (Fall)	23.25±1.06	24.10±1.33	23.40±0.52	24.61±0.27	23.46±0.62
	Extractives (%)				
Winter	9.30±0.35	9.57±0.13	11.06±0.41	9.93±0.04	10.22±0.72
Spring	5.20±0.91	4.07±0.03	7.90±0.15	8.80±0.50	7,33±0.43
Summer	8.12±0.9	8.39±0.36	9.37±0.23	9.08±0.49	8.02±0.53
Autumn (Fall)	9.93±0.14	10.46±0.56	8.98±0.12	8.12±0.27	10.28±0.17
	Ash (%)				
Winter	2.82±0.12	2.24±0.06	2.45±0.24	2.44±0.09	2.81±0.04
Spring	2.06±0.11	2.24±0.09	2.23±0.08	2.69±0.05	2.13±0.02
Summer	2.70±0.08	2.44±0.13	3.41±0.09	2.85±0.19	2.02±0.05
Autumn (Fall)	2.10±0.07	2.26±0.02	2.12±0.23	2.48±0.17	2.39±0.19

However, they contrast with the study of Ghrab [297], which associated higher cellulose levels with arid regions. This discrepancy suggests that cellulose synthesis in Alfa leaves is not only dependent on bioclimatic conditions but is also influenced by other factors such as soil properties [298], physiological maturity, seasonal distribution of rainfall and annual temperature patterns, which affect plant metabolic activity and cell wall composition [269]. Wang, et al. [299]provide a comprehensive review on the effects of osmotic (salt), photoperiodic, and thermal conditions on cellulose synthesis in wild plants. Research on hemp (Cannabis sativa L.)[300] has also shown that seasonal variation, geographical origin and soil quality have a significant effect on the final cellulose content of fibers.

From Table III. 1, the highest cellulose content in spring seems to be due to several factors, including:(i) Increased metabolic activity of the plant due to higher temperatures and abundant light, which enhances photosynthesis and cellulose synthesis Balaguer, et al. [301]; (ii) improved soil properties after the winter rainy season, providing a nutrient-rich environment for plant growth and the formation of strong cell walls[269, 297, 302]; (iii) the active growth phase of the plant during spring, when cells are in a state of division and expansion, requiring greater cellulose production to strengthen cell walls[269].

These findings corroborate earlier studies by Mehdadi, et al. [15], Belkhir, et al. [14], and Tarchi, et al. [13], Each emphasizes that cellulose biosynthesis is controlled by diverse and complex environmental factors. Mehdadi, et al. [15] observed that leaves collected during the summer exhibited the highest cellulose percentages, a trend attributed to the maturation and dehydration of leaves in this period. Conversely, Belkhir, et al. [14]and Tarchi, et al. [13] found that the fall season represents the optimum period for the highest cellulose content in Alfa leaves.

Compared to the elevated cellulose levels recorded in spring, the results revealed a marked decrease in cellulose content across all five study sites during the winter season. The average values were relatively close: 40.72 ± 0.22 % in Laghouat, 40.08 ± 0.56 % in Maadid, 40.01 ± 1.73 % in Boussaada, 39.45 ± 0.51 % in Tiaret, and 39.19 ± 1.03 % in Djelfa. This reduction in cellulose content, ranging approximately from 20.80 % to 22.57 %, can be attributed to the plant's reduced metabolic activity during its dormant phase in winter. During this period, most leaves are in the early stages of growth and begin to elongate, but their full development is slowed or even halted by extreme weather conditions [269, 303] , adversely affect the synthesis of cellulose molecules in the fiber walls [304]. These findings align with the results of Belkhir, et al. [14], which reported that the cellulose content of Alfa fibers leaves is lowest in winter.

Although our study did not record the highest cellulose content in the studied areas during the fall season, cellulose levels remained high compared to the summer and winter seasons. The highest level was recorded in Boussaada (43.89±0.34 %) followed by Djelfa (42.79±1.02 %), Tiaret (42.51±1.50 %), Laghouat (41.93±0.45 %) and finally Maadid (41.32±0.24 %). It should be noted that the value recorded in Boussaada (43.89±0.34 %) exceeds the highest cellulose content recorded by Tarchi, et al. [13] in the autumn season at (42.8 %). Such considered proportions of cellulose content in autumn may be due to the dual growth pattern of the Alfa plant, with an active growth phase in spring and autumn and a dormant phase during winter and summer, according to MERIEM [305]on the biological cycle of the Alfa plant. During the vegetative phases, leaves undergo rapid elongation, while growth rates drop dramatically during dormant periods due to extreme temperatures and low photosynthetic efficiency, cold in winter and drought in summer. This might explain the slight decrease in cellulose content in summer compared to autumn in most regions, except the Tiaret region, where cellulose content was higher in summer than in autumn.

Studies show that higher cellulose content in Alfa plant correlates with faster cell growth and greater biomass during the growing season. Nedjimi [306]. found that the aerobic biomass of Stipa tenacissima L peaks in spring, reaching a maximum dry weight of 55.31 ± 2.45 %, then declines significantly in summer (dry season). These seasonal variations are closely linked to environmental factors, particularly temperature and soil moisture, as Slimani, et al. [307]demonstrated. Additionally, Haase, et al. [308]reported in Almeria, southeastern Spain, that carbon fixation rate, maximum photosynthesis, and leaf elongation in Stipa tenacissima L plant increase with soil moisture.

III.3.2 Hemicellulose content

As know, Hemicellulose is the second most abundant component of the cell wall of lignocellulosic plants, the study of the seasonal variation in this compound seems to be important. The Table III.1 presents the seasonal variation in the hemicellulose content of the fiber of Alfa leaves collected from five different regions. The data indicate that the percentages of hemicellulose in fibers harvested during winter and fall are similar, ranging from 21 % to 23 %. In spring and summer seasons the low percentages were recorded in all studied regions, with the highest value in Boussaada at 19.31 ± 1.09 % in spring followed by Djelfa at 20. 1± 0.22 % in summer.

The observed decrease in hemicellulose content during spring may be attributed to improved environmental conditions, such as mild temperatures and nutrient availability [306],which enhance the metabolic activity of Alfa grass during the active growth phase [309]. At this stage, the plant exploits the carbon stored in hemicellulose as a rapid energy source to meet the needs of cellular expansion and accelerate growth (Hoch [310], Schädel, et al. [311]) . On the other hand, hemicellulose content is higher during fall and winter, acting as a reserve compound and a mobile carbon reservoir that is accumulated during periods of metabolic slowdown or dormancy, when energy demand is lower and the rates of degradation of sugars and proteins are reduced [310].

This seasonal variation on hemicellulose content is also explained by the influence of environmental factors such as lower temperatures and less light in winter, which reduce carbon consumption and promote the accumulation of hemicellulose in cell walls [310] .Furthermore, higher hemicellulose content during winter may be indicative an adaptive physiological response intended to strengthen cell wall rigidity and maintain their structural integrity, helping the plant to cope with extreme cold conditions [312]. This balance between consumption and storage reflects the physiological role of hemicellulose as a reserve compound that regulates energy and carbon flow in the plant (Volponi, et al. [313],Schädel, et al. [311]). Thus, the decline in hemicellulose content in spring and its increase in winter and fall represents an integrated regulatory response to environmental and physiological factors associated with the plant life cycle.

Whereas the decrease in hemicellulose content during the summer may be attributed to several factors, the most important of which are:(i) Extreme climatic conditions such as high temperatures and low rainfall (Table II. 1), as studies have shown that hemicellulose decreases with increasing temperature[314], while it is less related to precipitation changes, and delayed harvesting time decreases its content [315]. (ii) a plant enters summer dormancy, where metabolic activity is reduced and resources are directed to strengthen cell wall components (survival) rather than growth [316]. (iii) Alfa plant has a summer adaptation strategy that is manifested by an increase in lignin and cellulose content, which enhances cell wall rigidity and reduces water loss, to support survival under climatic stress[317].

Our findings are consistent with the biochemical analysis of Stipa tenacissima L. leaves by Mehdadi, et al. [15], who found that summer shows a marked decrease in hemicellulose content, reflecting the plant's adaptation to seasonal climatic stress (Arid conditions). Additionally,

different plant species show distinct responses to seasonal changes in hemicellulose composition. For example, cotton fibers from multiple species showed different temporal patterns in hemicellulose composition, influenced by the growing season [270]. Similarly, in lignocellulosic herbaceous crops, hemicellulose composition varied significantly depending on plant species and harvest cycles, highlighting the importance of plant type in seasonal biochemical changes [318] .Furthermore studies suggest that increased altitude, accompanied by lower temperatures, may reduce soil microbial activity, which may lead to reduced uptake of Phosphorus, Kalium and Nitrogen which is related to hemicellulose synthesis by Brassica napus[319] , while the hemicellulose and lignin content of the grasses studied by Koutsoukis and Akrida-Demertzi [320] may decrease as a result of changes in environmental conditions associated with altitude.

III.3.3 Lignin content

The experimental results of lignin content across the studied sites and throughout four seasons (Table III.1) revealed that the highest concentrations were observed during winter and summer, notably in Laghouat and Boussaada sites, with values of 27.08±0.89 % and 26.54±3.15 % respectively, during summer. Similarly, high lignin level was recorded in Boussaada during winter at 25.26±0.42 %. In contrast, the lowest lignin contents appeared in spring, particularly at the Boussaada (19.03±1.26 %) and Djelfa (19.08±3.12 %), indicating a strong seasonal influence on lignin biosynthesis. It is clear that these seasonal variations in lignin content are closely related to environmental factors. as high summer temperatures and low winter temperatures (Table II. 1) trigger lignin biosynthesis within the plant's abiotic stress defense response[321], especially drought and cold. Han, et al. [322]confirm that lignin accumulation is promoted under stress conditions to strengthen cell walls and improves toughness against solar radiation and wind [323]. It is also deposited at sites of injury or pathogen attack to prevent the invasion of harmful microorganisms [317, 324]. This mechanism is also supported by the research of Belkhir, et al. [14], who reported that during periods of dormancy, especially in summer, leaf growth slows down while tissue lignification increases. This physiological adaptation enhances structural rigidity and helps minimize water loss. Study of Henderson and Robinson [314],indicate that temperature significantly affects the concentrations of fiber components, with higher temperatures increasing cellulose and lignin levels, while hemicellulose concentrations tend to decrease.

Conversely, spring is characterized by active plant growth and reduced environmental stress, which favors the growth of primary walls rather than structural support as observed by Boerjan, et al. [325]Furthermore, the observed discrepancies in lignin content among sites, such as the variation between Laghouat (23.90±0.53 %) and Maadid (25.26±0.42 %) during winter, may be indicative of regional variations in soil properties, local climatic conditions, and levels of sunlight exposure. These fluctuations between sites and within seasons suggest that lignin polymerization is a tightly regulated, this regulatory mechanism enables plants to modulate the mechanical flexibility of vascular tissues in response to developmental and environmental stresses.

Based on these results, natural fiber utilization can be guided by seasonal lignin content, when lignin content is high (as in winter or summer), fibers are better suited for applications that demand rigidity and resistance, such as wood panels, thermal insulation. In such materials, elevated lignin content enhances microbial resistance and dimensional stability, although it reduce compatibility with certain polymer matrices [34]. For instance, recent studies have shown that composites reinforced with lignin-rich wood fibers retained higher tensile strength after microbial exposure (40 % strength loss) compared to those using low-lignin jute fibers (57 % loss), confirming lignin's protective role[326].

When lignin content is low (typically in spring), the fibers are more suitable for applications requiring high cellulose accessibility, such as composite materials, papermaking, chemical pulping, or bioethanol production. Their low lignin content facilitates enzymatic and chemical breakdown, making them ideal for processes that rely on depolymerization and sugar conversion.

III.3.4 Extractives content

The results of the current study presented in Table III.1 indicate a clear seasonal variation in ethanol extractives content of Alfa leaves across the five studied sites. The lowest values were recorded during spring, especially at Laghouat (4.07 ± 1.03 %), while values peaked in winter (Djelfa at11.06 ± 0.41 %) and fall (Laghouat at10.46 ± 0.56 %). While Site-to-site differences were relatively small (standard deviations ±0.3–3.7 %). The values were also relatively high in summer at most sites.

These results are partially consistent with the study of Belkhir, et al. [14], which showed higher extractive percentages during winter (12.9 ± 0.5 %), while Alfa leaves collected in spring showed lower content (4.6 ± 0.3 %) due to the physiological activity associated with leaf growth, which reduces the accumulation of secondary compounds as the papermaking characteristics

were optimal at that time. In other hand, studies on non-woody herbaceous plants (Panicum virgatum and Arundo donax) [327, 328], also showed an increase in the content of phenolic compounds under heat and drought conditions, suggesting a role for these compounds in the plant's response to environmental stress. This variation in extractives content of Alfa fibers may be influenced by biological factors, particularly plant growth cycles and environmental stressors [329].

In winter, low temperatures contribute to the accumulation of defensive compounds against cold stress, while summer drought stimulates the production of waxy and phenolic compounds to reduce water loss and protect tissues [330, 331]. In spring, increased vegetative growth reduces the production of these compounds in favor of building primary walls [76].

Seasonal extractive variations in Alfa leaves determine their optimal industrial use: Higher levels in winter, fall and summer are suitable for the production of insulation materials or antimicrobial applications, while lower content in spring is preferred for pulp and paper and bioconversion processes. Aligning harvesting with these biochemical properties enhances efficiency and sustainability in various industries.

III.3.5 Ash content

The ash content in plant fibers is a measure of the concentration of metals and inorganic salts remaining after combustion and directly affects the efficiency of industrial processes such as sintering, papermaking, and biofuels [110] . The values reported in the Table III.1 demonstrate that the lowest ash percentages were recorded in the spring across all sites, Notably in Laghouat at 2.24 ± 0.09 % and Boussaada at 2.69 ± 0.05 %. with the highest values in summer (mean ≈ 2.68 %) and/or winter (mean ≈ 2.72 %). Djelfa Site shows a clear superiority in summer (3.41 ± 0.09 %), this could be attributable to influence of water stress on the concentration of minerals in tissues [332] and high organic matter at this site [14].

In our study, ash content of Alfa leaves generally ranged from 2.02 % to 3.41 %, which are higher than those found in wood residues such as Eucalyptus sawdust 0.6 %, Chestnut shavings (0.4 %), Beech wood(0.6 %), Fir wood (0.1 %), but much lower than non-woody plants such as Bamboo leaves (7.0 3 %), Cynara grass leaves (29.6 %) , Cynara grass leaves (29.6 %),Tobacco stalks (22.5 %),Sugar cane leaves (7.7 %) according to Vassilev, et al[110] .

The observed increase in ash content during summer may be linked to the concentration of minerals and salts resulting from intense evapotranspiration and continuous root uptake under drought stress conditions[332]. In contrast, the elevated ash levels in winter could be associated

with reduced vegetative activity and the subsequent accumulation of inorganic solutes within plant tissues.

During spring, a marked decline in ash content is likely due to enhanced vegetative growth, where minerals are predominantly directed toward the biosynthesis of primary cell walls. Additionally, seasonal rainfall and potential topsoil erosion might contribute to the depletion of mineral availability in the upper soil layers, thus lowering their accumulation in leaf tissues[333] . Therefore, it is preferable to harvest Stipa tenacissima L leaves in the spring season with the lowest ash (and silica) content, which improves the efficiency of foliar and chemical sintering processes and reduces the wear of milling and cutting tools, while fibers harvested in summer or winter, -with higher ash, can be directed to the production of biofuels and less refractory materials because the excess minerals enhance combustion properties[111, 334].

III. 4 Thermal analyses

As illustrated in Figure III (2-5), the TGA and DTG curves were used to investigate the influence of seasonal variations and growth sites on the thermal behavior of Alfa fibers. It is clear that in all TGA curves the thermal decomposition consists of three stages of weight loss. Generally, the first phase between [50 °C –150 °C] corresponds to the vaporization of moisture in Alfa fibers[335] . The presence of absorbed water is evidenced by a characteristic ATR-FTIR absorption band at approximately 1640 cm^{-1} [336].

Afterward, the sample weight stabilizes within a thermal range that varies between samples. The temperature at which weight loss resumes ,corresponding to the initial decomposition of hemicelluloses, can serve as an indicator of thermal stability, known as the maximum stability temperature (T_{ms}), or alternatively referred to as the onset decomposition temperature [337]. The second phase ranged between [200 °C–400 °C], which is related to the thermal degradation of cellulosic compounds, specifically the depolymerization of hemicellulose and cellulose [338]. Reddy, et al. [339] noted that a small percentage of lignin undergoes thermal degradation during this stage. It is important to note that, based on the DTG curves, the maximum peak represents the temperature at which the maximum thermal degradation of cellulose occurs, i.e., the point at which the highest rate of mass loss at Max decomposition temperature (T_{max}) [295].

Table III.2. Seasonal variation in thermal analysis parameters of Alfa fibers from the selected growth sites.

Season / Sites	Winter			Spring			Summer			Autumn (Fall)		
	T_{ms}(°C)	T_{max}(°C)	E_0(kJ/mol)	T_{ms}(°C)	T_{max}(°C)	E_0(kJ/mol)	T_{ms}(°C)	T_{max}(°C)	E_0(kJ/mol)	T_{ms}(°C)	T_{max}(°C)	E_0(kJ/mol)
Tiaret	217.63	308.89	86.44±1.05	231.80	329.09	121.26±2.60	234.93	328.81	117.37±2.30	198.87	316.85	87.41±3.33
Laghouat	229.19	320.96	88.58±0.70	237.85	320.70	120.35±1.40	211.88	324.70	115.25±3.37	202.02	312.23	108.85±4.27
Djelfa	203.83	311.14	83.55±0.94	229.77	320.36	118.41±3.17	183.08	319.54	109.75±3.12	218.87	321.61	113.15±5.57
Boussaada	207.54	315.48	85.68±2.38	213.57	321.95	109.75±3.17	210.08	322.22	110.27±3.17	234.08	322.51	115.75±3.4
Maadid	197.33	303.69	75.77±1.49	226.86	315.30	119.25±3.37	224.37	327.73	115.47±4.27	213.19	309.72	113.65±3.27

T_{ms}(°C): Maximum stability temperature, T_{max}(°C): Maximum decomposition temperature, E_0(kJ/mol): Activation energy.

Lignin may also undergo depolymerization at this stage [340]. The third phase [400 °C–600 °C], corresponding to the degradation of non-cellulosic materials such as pectin and lignin, as well as the oxidative degradation of the charred residue [146, 341], may exhibit multiple peaks in some DTG curves. The thermal degradation characteristics of the different Alfa fibers, including the maximum stability temperature (T_{ms}), activation energy (E_o), and maximum decomposition temperature (T_{max}) are summarized in Table III.2. As illustrated in the table, noticeable variations in these thermal parameters are observed among the studied Alfa fibers, mainly due to seasonal effects and differences in growing regions. First, in terms of thermal stability, the highest values of initial weight loss temperatures were recorded during spring in most growth sites, especially for AL and AT samples at 237.85 °C and 231.80 °C, respectively, respectively.

This was attributed to the previously reported low hemicellulose content in this season's samples. Hemicellulose is an amorphous component that mainly contributes to the initial thermal degradation and is correlated with moisture content[342]. This was supported by the lower moisture content recorded in the same samples (ATSP, ALSP, ADSP, ABSP, AMSP) reflecting higher thermal resistance and slower degradation during spring compared to other seasons.

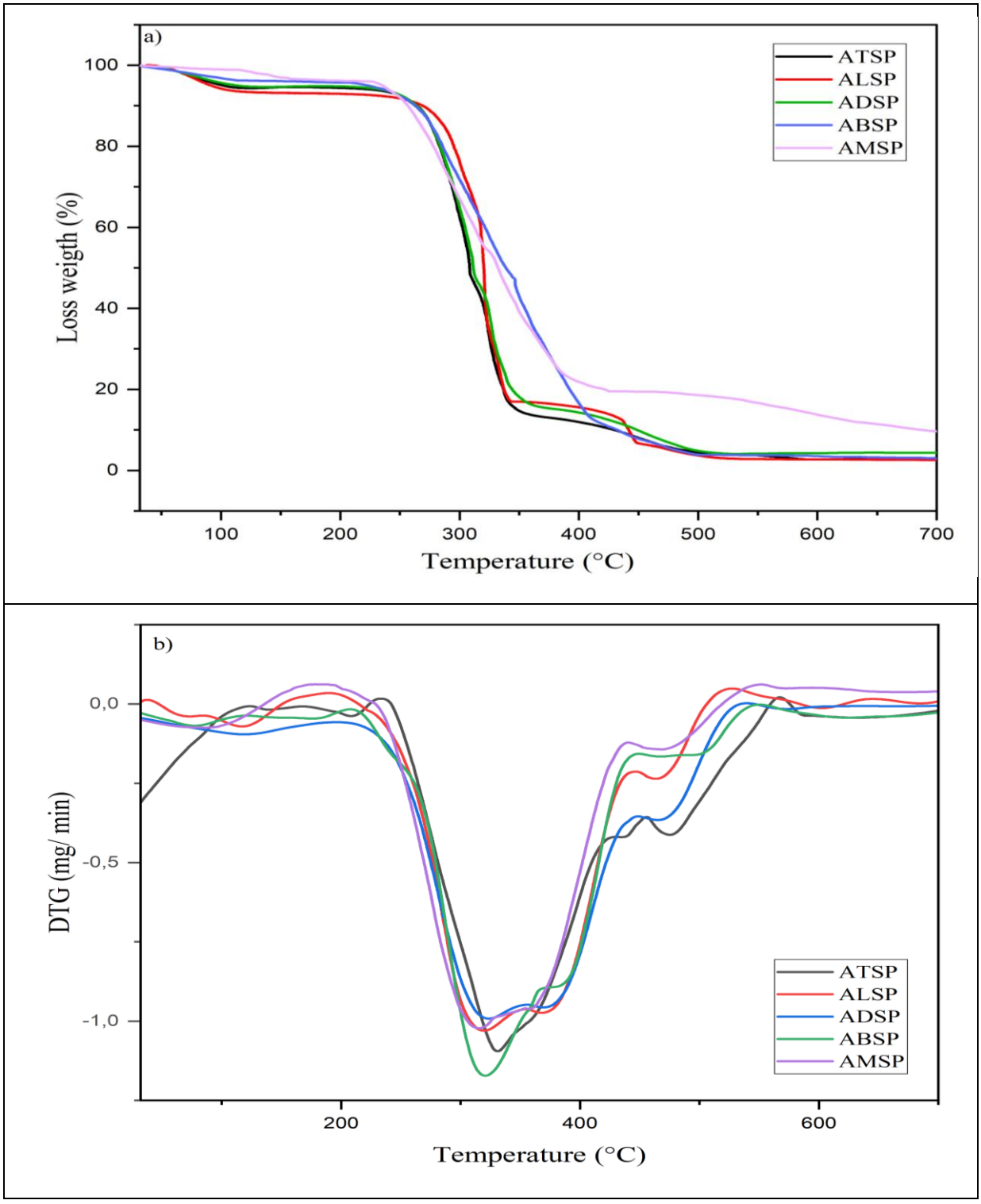


Fig III.2 Thermogravimetric curves (TGA-DTG) of Alfa fiber during spring.

On the other hand, most samples collected during summer showed greater thermal stability, which is consistent with their higher values of initial loss weight temperature or maximum stability temperature (T_{ms}). This could be a reflection of the lower hemicellulose and moisture

content of this season's samples shown in Table III.1 and Table III. 6, With the exception of the ADS sample, in which the maximum stability temperature decreased to 183.08 °C, which may be attributed to its high extractives content (9.37±0.23 %) compared to the rest of the samples of the same class. This is consistent with the findings reported by Poletto, et al. [343], that substantial quantities of extractables may accelerate the degradation of fibers at low temperatures. In contrast, the decrease in T_{ms} was observed for most of the fall and winter samples, likely due to the high moisture content, as well as the high extractives content previously recorded during these two seasons. This is also supported by the findings of Poletto, et al. [344], suggested that both bound water and extractives may contribute to the initiation of the pyrolysis process at relatively low temperatures, accelerating the degradation of the rest of the fiber components and ultimately leading to reduced thermal stability, as observed in sisal and buriti fibers.

Second, in term of maximum decomposition temperature (T_{max}) and activation energy(E_0), based on the results of Table III.2, it should be noted that most of the Alfa fiber samples harvested during spring and summer showed higher thermal degradation compared to other seasons. This is evidenced by the high values of maximum degradation temperature T_{max} recorded in all samples of these seasons, especially ALSP, ATS and AMS, which reached 329.09 °C, 328.81 °C and 327.73 °C respectively. This may be attributed to : (i) elevated cellulose content, as it is known that the maximum thermal degradation degrees of plant fibers often correspond to the degradation of cellulose, which in turn requires sufficient activation energy to decompose it[295], (ii)higher crystallinity index, as well as the lower proportion of hemicellulose noted during that phase. This aligns with the findings of Indira, et al. [345],who emphasized that the thermal degradation behavior of hemp fibers is primarily influenced by the structural characteristics of cellulose and the proportion of non-cellulosic constituents within the fiber. A higher crystallinity index reflects a more regular crystal structure, which in turn enhances the thermal resistance of the fiber [344]. Previous studies have shown that this crystal structure requires a higher activation energy to initiate the thermal degradation process, as a result of a lower proportion of irregular or easily degradable regions (Jonoobi, et al. [346]; Poletto, et al. [343]). This can be explained by the crystalline structure of cellulose, which enhances its resistance to thermal degradation, resulting in a higher activation energy for its degradation compared to hemicellulose and lignin[347], which explains the higher maximum degradation temperature recorded for Alfa fibers with higher crystallinity.

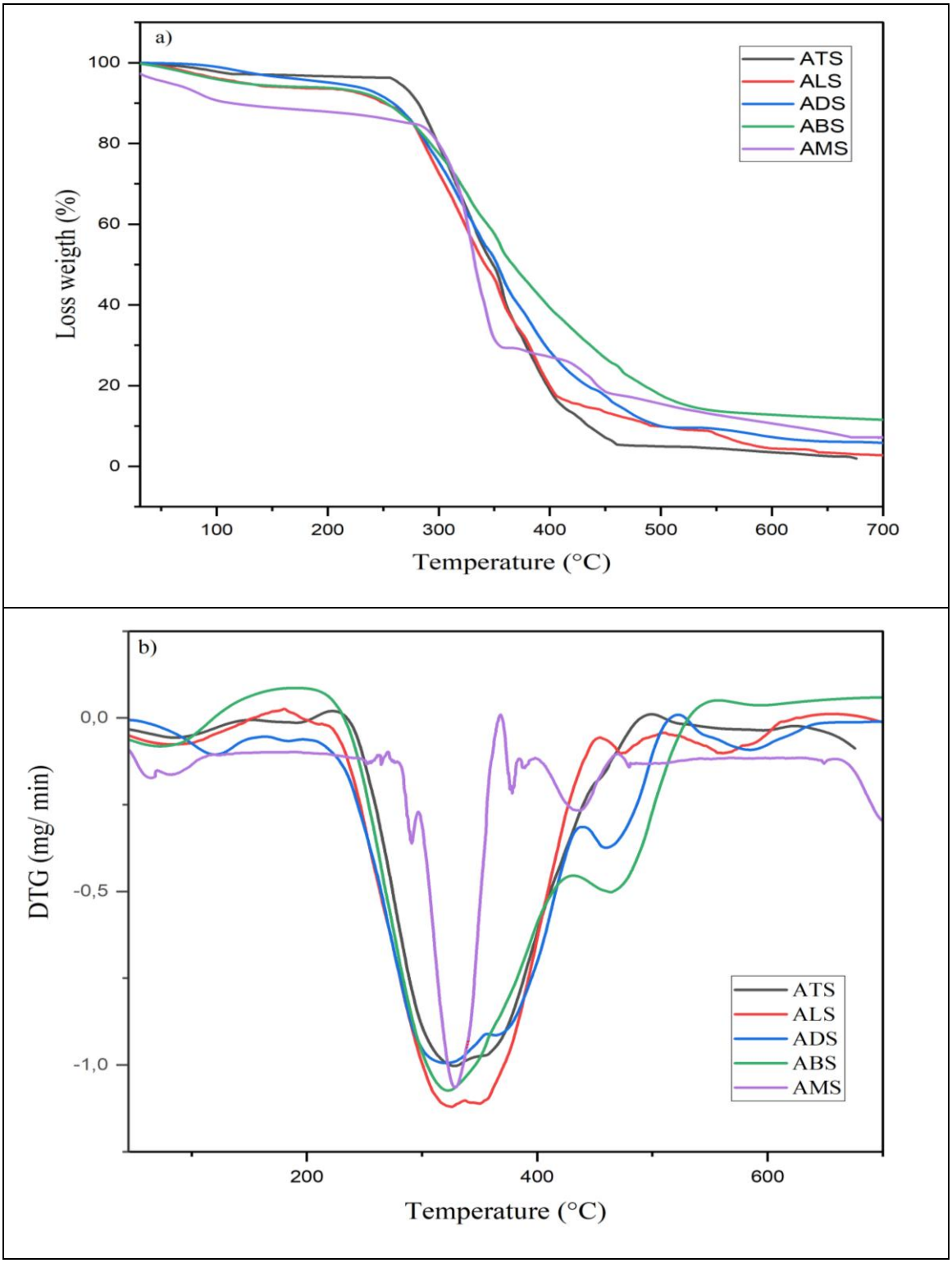


Fig III.3 Thermogravimetric curves (TGA-DTG) of Alfa fiber during summer.

This finding is supported by the activation energy values associated with cellulose degradation (Table III.2), with the highest values recorded in most of the spring and summer samples, ranging from 105 to 121 kJ/mol, followed by the fall samples. In contrast, the lowest

values were observed in samples harvested during winter, indicating that the thermal behavior of Alfa fibers is directly influenced by their chemical composition and seasonal crystallinity.

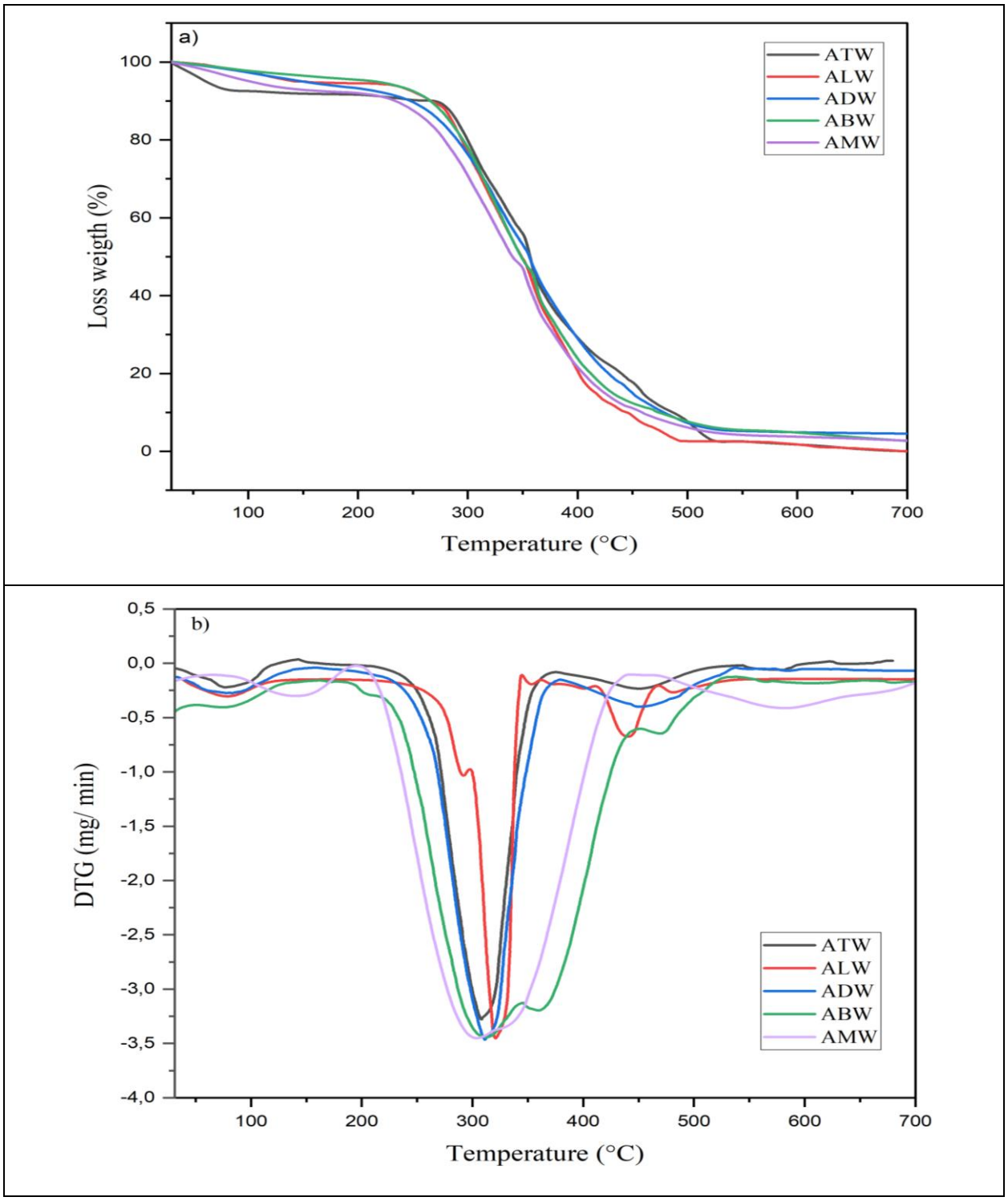


Fig III.4 Thermogravimetric curves (TGA-DTG) of Alfa fiber during winter.

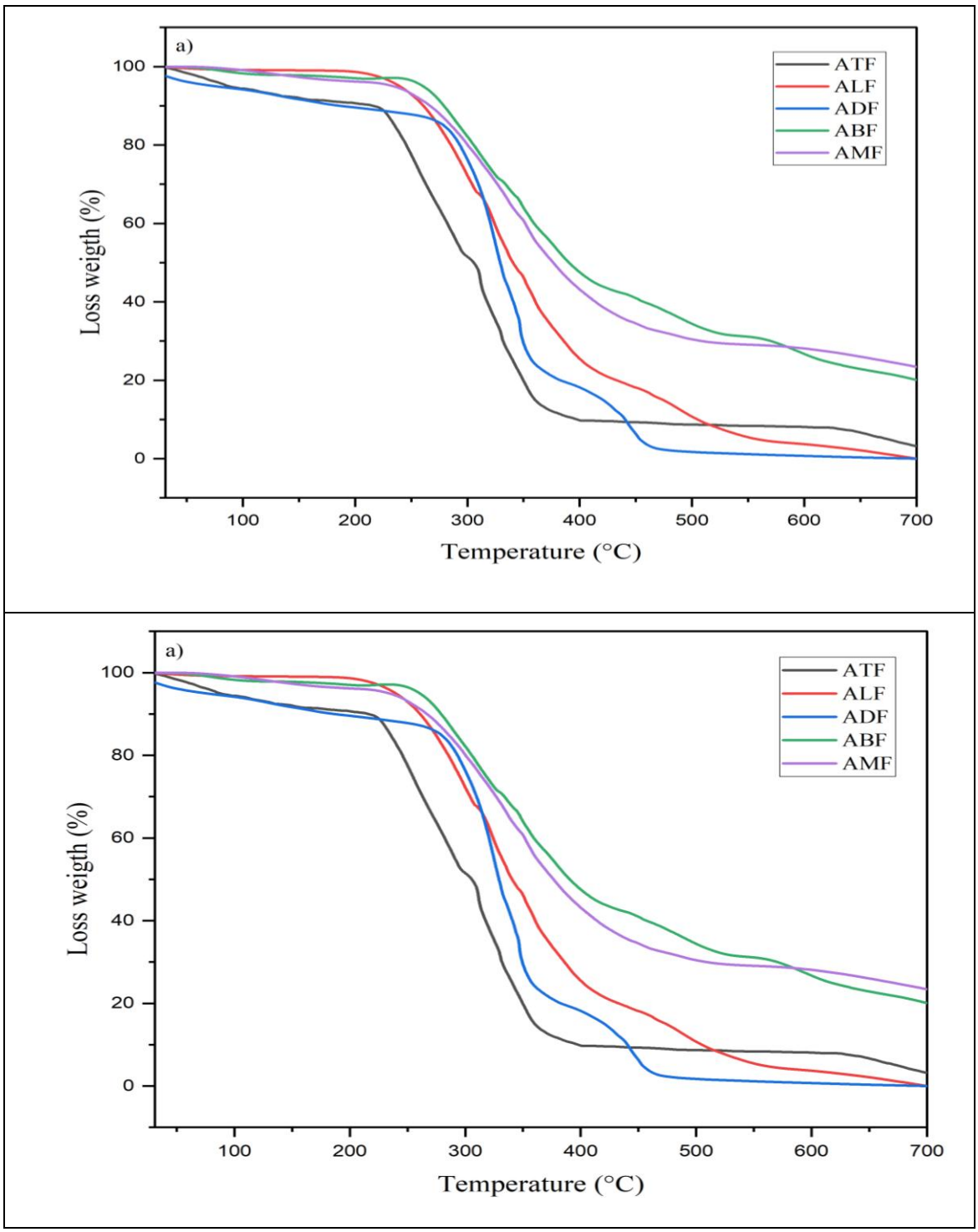


Fig III.5 Thermogravimetric curves (TGA-DTG) of various Alfa fibers during fall.

III. 5 X-ray diffraction analysis

The crystallinity index (CrI), determined by X-ray diffraction, indicates the degree of structural arrangement of cellulose chains in plant fibers and is directly related to the mechanical stiffness, thermal stability and other quality attributes of natural fibers [279] .

The X-ray diffraction (XRD) curves for all samples show the same general trend Figures III (6,7) characterized by two clear peaks: The first is broad and low-intensity within the range 2θ≈16°-18° and refers to the non-crystalline regions, while the second is sharp and intense at about 2θ≈22°, attributed to the crystalline structure of cellulose, The different intensities of these peaks among the samples reflect variation in the degree of crystallinity, expressed in this work by the crystallinity index, as shown in Table III. 3.

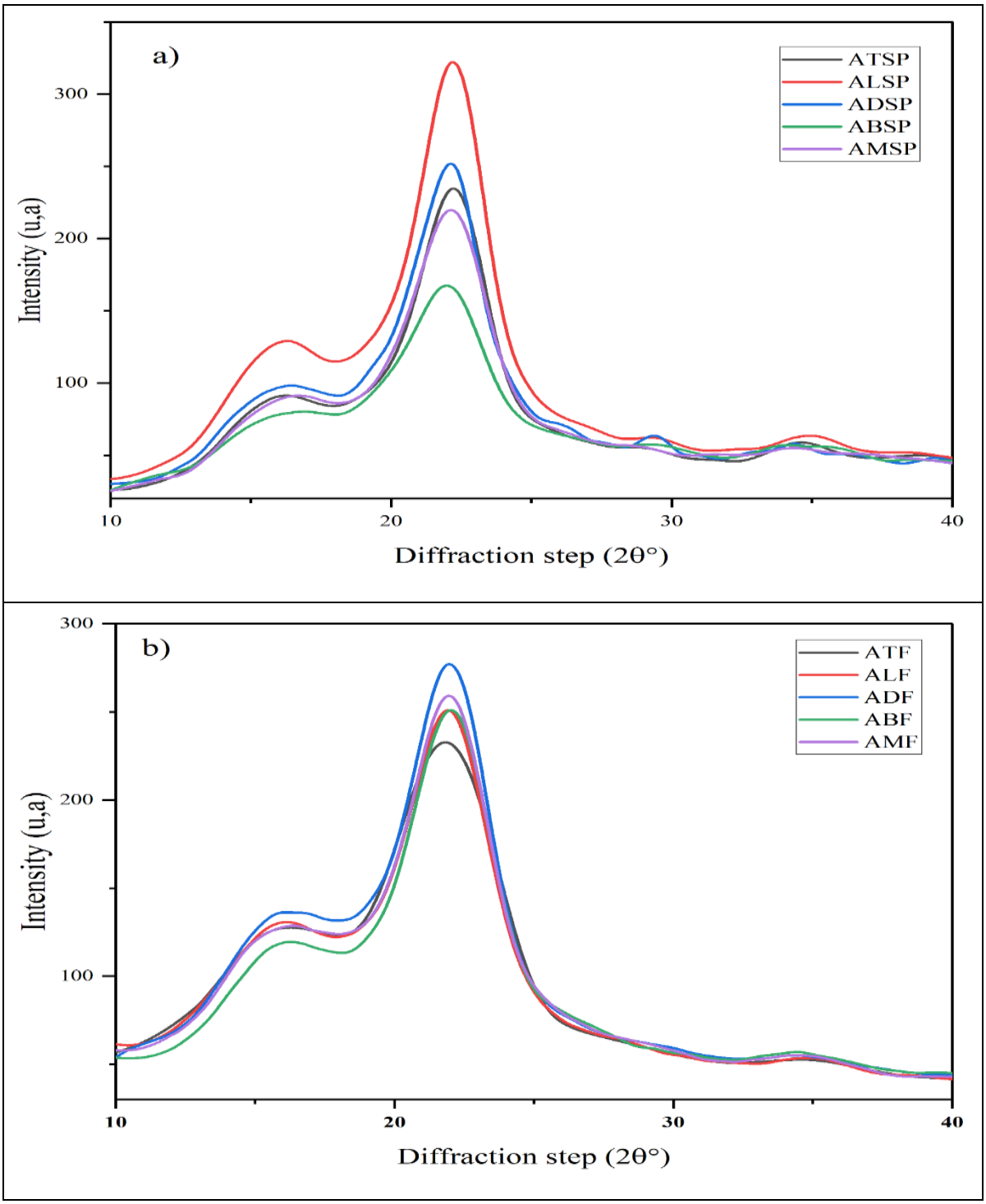


Fig III.6 X-ray diffraction curves for selected Alfa fibers during (growth phase): a) spring and b) fall.

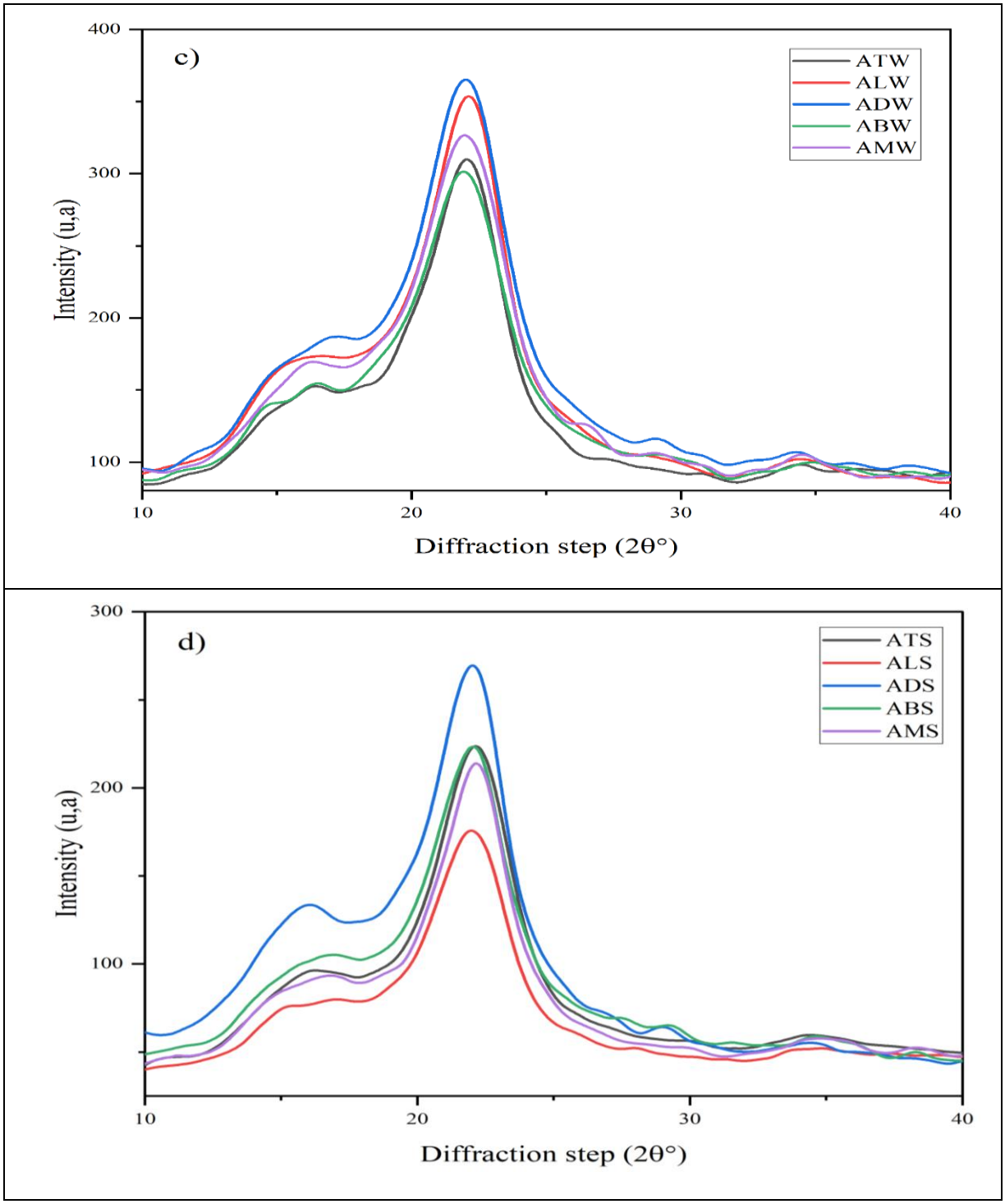


Fig III.7 X-ray diffraction curves for selected Alfa fibers during (latency phase): c) winter; d) summer.

The results illustrated in the histogram (Figure III.8) reveal that the crystallinity index (CrI) of Alfa fibers reached its maximum during spring in most of the studied regions, especially in AT (65.95 %) and AL (65.84 %). This is probably related to the high cellulose content recorded in the same samples in this season compared to the rest of the samples (chemical characterization

part), which is the most crystalline component of the plant cell wall. Cellulose forms a highly ordered structure through intense hydrogen bonding, which enhances the mechanical stiffness and thermal stability of the fibers. These results support studies of [9, 34, 80, 348], which demonstrated that an increase in highly ordered cellulose chains is associated with a higher crystallinity index.

Table III. 3. Seasonal variation in crystallinity index of Alfa fibers from the selected growth sites

	Crystallinity index [CrI %]			
Seasons / Sites	**Winter**	**Spring**	**Summer**	**Autumn (Fall)**
Tiaret	49.63	65.95	60.06	53.94
Laghouat	51.84	65.84	56.59	53.09
Djelfa	49.74	63.64	54.14	55.22
Boussaada	50.25	64.15	54.76	56.76
Maadid	49.38	61.73	58,.15	52.01

In contrast, the lowest values of the Crystallinity Index (CrI) were observed during the winter season, followed by fall, and then summer. Specifically, CrI values in winter ranged between 49.38 % in AT and 51.84 % in AL. During fall, values varied from 52.01 % in AM to 56.76 % in AB, whereas in summer, they ranged from 54.14 % in AD to a maximum of 60.06 % in AT. The low CrI of the fall samples compared of summer samples is likely due to high hemicellulose content leads to an increased moisture content recorded during fall season. This is consistent with the findings of Jiang, et al. [349]and Karimah, et al. [82] , as their results showed that high moisture disrupts the hydrogen bonds between cellulose chains, which weakens the crystal network structure and reduces the stability of materials [349]. In addition, a high hemicellulose content contributes to fiber flexibility and facilitates swelling, which negatively affects structural regularity. Conversely, low hemicellulose content improves the regularity of cellulose chains in non-crystalline areas and enhances fiber crystallinity[82].

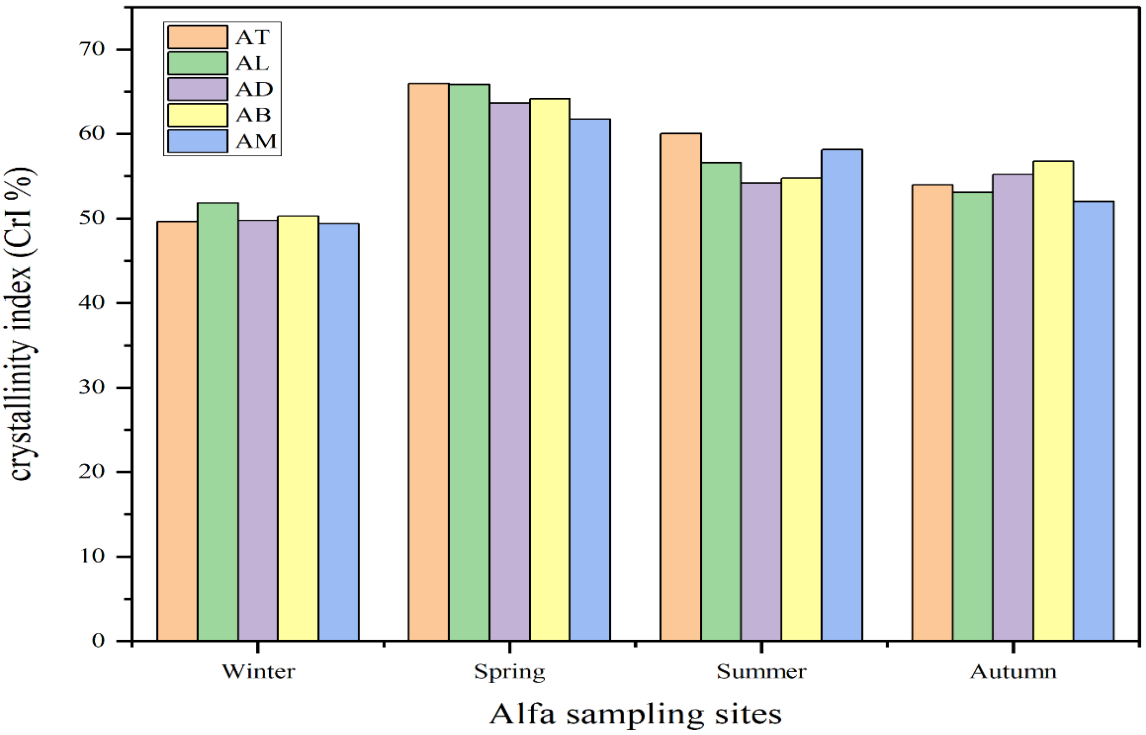


Fig III.8 Evolution of Alfa fibers crystallinity index (CrI) during seasons and growth sites.

However, the decrease in crystallinity index (CrI) in summer samples relative to the spring ones, along with the noticeable decline in cellulose content, which is directly related to this index, may be attributed to the impact of harsh environmental conditions, such as heat stress, which in turn leads to the breakdown of hydrogen bonds between cellulose chains and hence a decrease in the regularity of the crystalline arrangement. This is consistent with the findings of [350, 351], where Wei, et al. [351]demonstrated that elevated temperatures break down internal hydrogen bonds, thereby promoting the dissolution of cellulose chains and weakening its crystalline structure. On the other hand, The study conducted by Lu, et al. [350] on cell wall remodeling under heat stress demonstrated that plants exposed to heat stress tend to reorganize their cell wall components, including polysaccharides and lignin, promoting crosslinking of cellulose chains as a defense mechanism to maintain structural homeostasis and reduce water loss. In addition, the high content of non-crystalline compounds such as Lignin and hemicellulose, due to their amorphous and irregular structures can interrupt the regular packing of cellulose chains and thus reduce the total CrI in biomass.

This result supports the studies of Wang, et al. [352]and Karimah, et al. [80] which showed that a high content of amorphous compounds negatively affects the crystalline structure of cellulose Such findings may also provide a plausible explanation for the notably low crystallinity

index observed in the winter samples, which exhibited the highest recorded levels of hemicellulose and lignin compared to those collected in other seasons.

The crystalline arrangement of plant fibers is also likely to be influenced by several factors, most notably moisture content[4, 353] and fiber maturity; the degree of crystallinity decreases as fiber maturity decreases[9, 348]. In contrast, moderate hydration stabilizes the hydrogen bonds between cellulose chains and may even induce annealing in amorphous regions. However, exposure to severe water stress or flooding leads to crystal structure disruption and reduced CrI [354]. Moreover, spatial variation shows a clear impact on CrI, which has been found to vary from one region to another during the same season, with differences ranging from 1 % to 11 %. This variation is likely due to differences in soil texture, nutrient availability, pH, and elevation, which are formative factors that directly affect cellulose deposition and its structural organization within cell walls.

III. 6 Spectroscopic characterizations

The comparative analysis of ATR-FTIR spectra of Alfa fibers collected from five regions across the four seasons revealed a generally consistent spectral pattern, similar to that observed in other plant fibers. Absorption peaks appeared at comparable wavelengths, with variations in intensity among the samples (Figures III.9-10), indicating that all samples share the same functional groups and primary chemical constituents, including cellulose, hemicellulose, and lignin, albeit in varying proportions depending on the season and growth site. This observation corroborates previous chemical composition analyses and highlights the clear influence of seasonal changes and growth locations on the chemical structure of Alfa fibers.

The Table III. 4. highlights Characteristic absorption bands in ATR-FTIR spectra of various Alfa fiber samples, along with associated functional group assignments and molecular vibrational modes, as reported in the scientific literature. ATR-FTIR spectra of summer Alfa fiber samples showed a high degree of similarity, with almost identical absorption bands, reflecting a great convergence in the chemical composition of the different samples of this season. This similarity is clearly evident in the content of cellulose, hemicellulose, lignin and extractives, as the differences between the components of the samples belonging to the same season did not exceed 1%, indicating a high degree of chemical homogeneity within the seasonal group. A similar similarity was observed in the spectra of the spring samples, which also indicates a remarkable chemical homogeneity among the samples, except for the AL sample that recorded a high

cellulose content (51.42 ± 1.35%), which explains the prominent peak at 1418 cm^{-1}, which is attributed to cellulose-related vibrations [355].

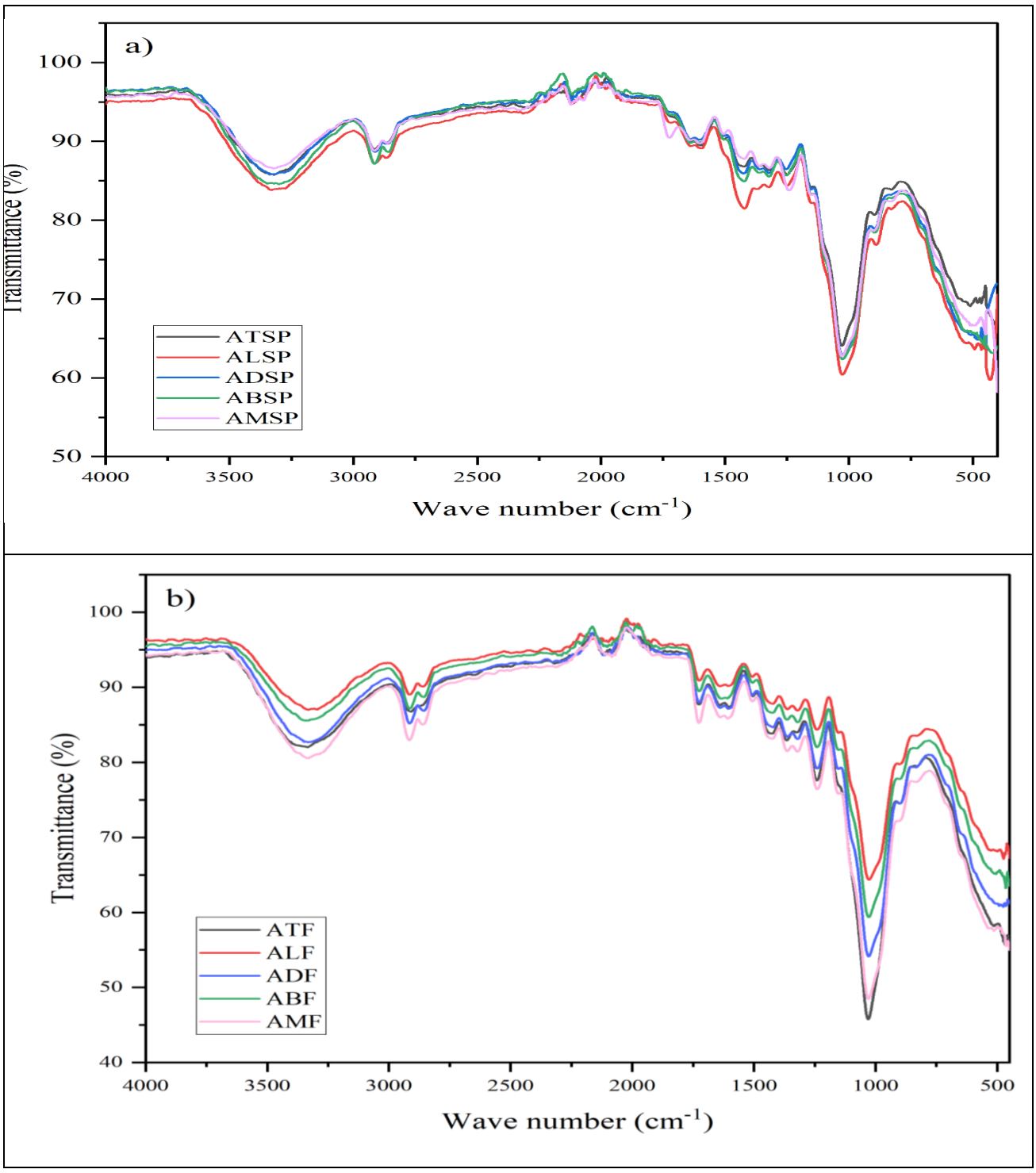


Fig III.9 ATR-FTIR spectra of various Alfa fibers during spring and fall (growth phase): a) spring and b) fall.

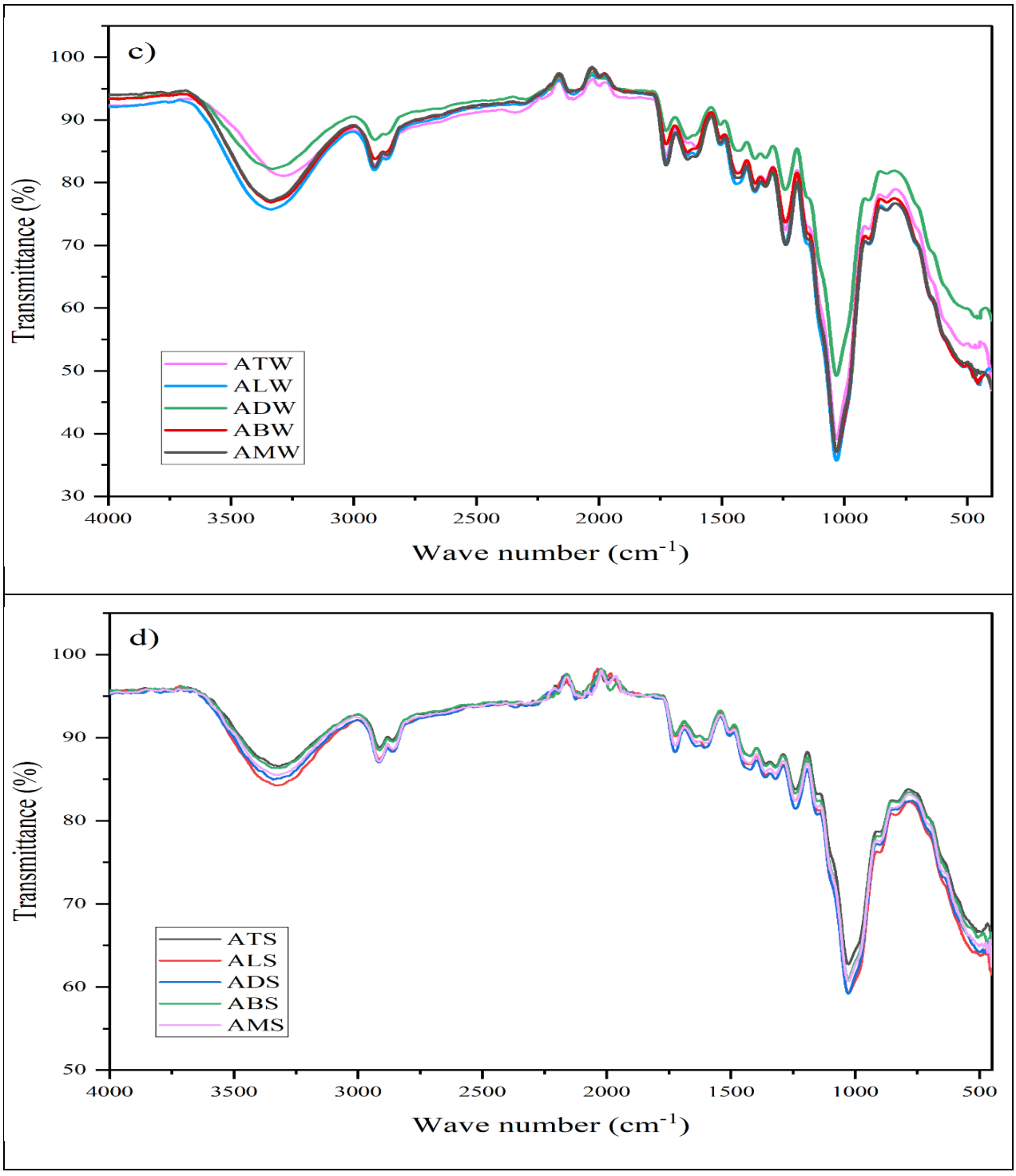


Fig III.10 ATR-FTIR spectra of various Alfa fibers during winter and summer (latency phase): c) winter; d) summer.

In contrast, greater variability was observed in the spectra of the winter samples, particularly in the broad peaks around 3334 cm^{-1} and 3277 cm^{-1}, which appeared significantly lower in AD and AT samples, possibly indicating their lower cellulose content compared to the other samples. In addition, there was a slight decrease in the intensity of the peak at 1242 cm^{-1} for the AD

sample, a peak usually attributed to vibrations of the -COO stretching bonds in hemicellulose, which could be an indication of the low concentration of this component in the sample. The peak at 1036 cm^{-1}, associated with C–O–C bridge stretching coupled vibrations in cellulose and hemicellulose[336], also appeared relatively low in the same sample, which in turn supports the possibility of low content of both components (cellulose and hemicellulose) in AD. There was also a noticeable difference in the spectra of the fall samples, especially at the broad peaks around 3339 cm^{-1} and 3324 cm^{-1}, where these peaks appeared with significantly lower intensity in AL and AB samples.

Table III. 4. ATR-FTIR spectra of various Alfa fiber samples and the corresponding functional group assignments.

Wave number range (cm^{-1})	**Origin**	**References**
3800-3100	O-H stretching (Cellulose, hemicelluloses)	[18]
2910- 2854	C-H stretching in CH and CH_2 (cellulose, hemicellulose)	[10]
1724-1718	C = O stretching carbonyl groups (hemicellulose, lignin and extractives)	[34]
1650-1600	H–O–H bending of absorbed water	[336]
1440-1420	CH_2 and CH_3 group symmetric bending from cellulose	[355]
1243-1235	-COO stretching in hemicelluloses	[336]
1030-1020	C–O–C bridge stretching (Cellulose, hemicelluloses)	[282]
904-890	β-glycosidic in cellulose and hemicellulose	[34]

This spectral difference may corroborate previous results that showed lower cellulose content in the AL sample, and lower hemicellulose content in AB sample, since these peaks are associated with O-H stretching vibrations in both cellulose and hemicellulose[18]. Relatively higher intensity peaks at 1237 cm^{-1} were recorded in AM, AT, and AD samples, possibly due to their high hemicellulose content. Furthermore, the peak at 1030 cm^{-1}, associated with the stretching vibrations of the C-O-C bridge in both cellulose and hemicellulose[282], showed

significant variation between samples, which in turn reinforces the presence of variation in the content of at least one of these two components within a season's samples.

III.7 Physicomecanical properties of fiber

III.7.1 Density measurement

The results of Table III. 5. indicate a clear variation in Alfa fiber density among the five studied sites during the four seasons. The lowest density was recorded in Maadid during winter (0.992 ± 0.004 g/cm^3) while the highest density was recorded in Laghouat during spring (1.378 ± 0.019 g/cm^3). A recurring seasonal pattern is observed in most of the sites: Low densities during winter and summer, then gradually increasing, reaching the highest values in spring then fall (Figure III.11). These seasonal changes in density can be linked to seasonal changes in the chemical composition of various Alfa fibers, which were analyzed in the previous section.

Table III. 5. Seasonal variations in the apparent densities of Alfa fibers from five growth sites.

	Density (g/cm^3)			
Seasons / Sites	**Winter**	**Spring**	**Summer**	**Autumn (Fall)**
Tiaret	1,155 ± 0.038	1.317±0.026	1. 132±0.042	1.218±0.058
Laghouat	1.170 ± 0.044	1.378±0.059	1.190±0.052	1.207±0.022
Djelfa	1,027 ± 0.053	1.268±0.024	1.139±0.037	1.213±0.039
Boussaada	1,037 ± 0.016	1.206±0.055	1.185±0.025	1.305±0.019
Maadid	0,992 ± 0.024	1.241±0.036	1.157±0.047	1.260±0.027

During the spring, the cellulose concentration reaches its peak. This results in the formation of a robust crystalline network that is interconnected via strong hydrogen bonds[275]. thereby enhancing fiber cohesion and reduces internal micro porosity, leading to increased density. These results clearly support the strong relationship between cellulose and plant fiber density, this is consistent with what Karimah, et al. [4] reported about the positive correlation of density with cellulose.

In summer, high temperatures and intense solar radiation stimulate lignification processes, leading to an increase in lignin content within the cell wall. This increase in lignin content

stimulates the formation of more bonds with cellulose chains. This causes the cellulose chains to diverge, leading to a less regular structure and less compact packing. resulting in a lower density. This supports Mukhopadhyay and Mukherjee's hypothesis [356] that the density of jute fibers in late stages of growth is lower than in early stages, when lignin content is lower, leading to weaker binding of lignin to cellulose chains.

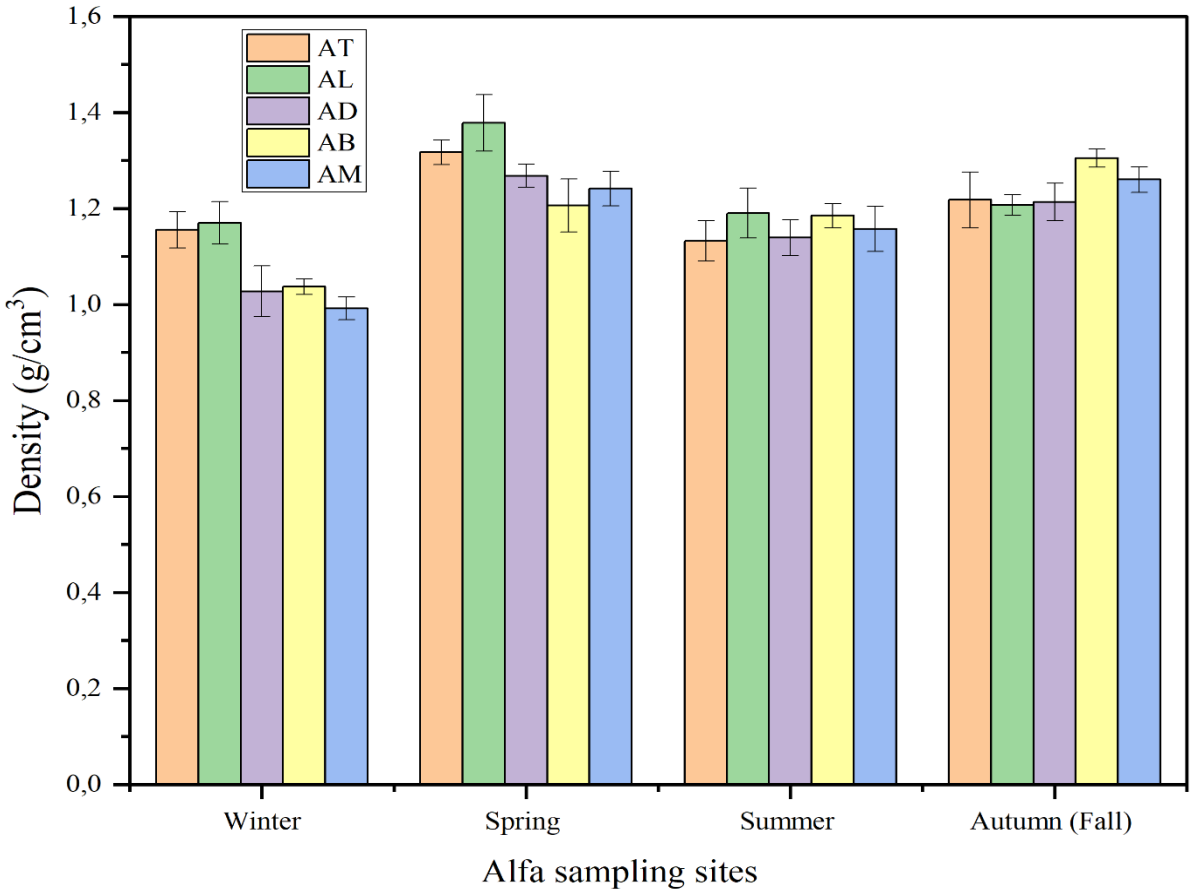


Fig III.11 Evolution of Alfa fiber density across seasons and growth sites.

It is also consistent with the findings of Jiang, et al. [357] that lignin affects the increase in mesopore diameter of cellulosic fiber network due to the interference of lignin with the hydrogen bonding between fibers. In addition, winter refers to a period of physiological dormancy, during which the plant's metabolic activity diminishes, resulting in less mature fibers with significantly higher water content, lower levels of cellulose, and a less crystalline cellular structure. Additionally, the increase in non-cellulose content (lignin and hemicellulose) during this season, which is negatively correlated with density, may explain the low density observed during this season [358].

III.7.2 Moisture content of fiber

Moisture content in plant fibers governs their dimensional stability, microbial susceptibility, and processing requirements, such as drying energy and treatment efficacy [359, 360].

As demonstrated in Table III. 6, the moisture content of Alfa fibers is evidently influenced by seasonal variations and growth sites, thereby underscoring the dynamic interplay between local environmental factors and the plant's physiological system. During the spring, Alfa fibers maintained low levels of moisture (approximately 4.03 % - 4.45 %), despite significant rainfall. This is likely due to the nature of the growing season, where most of the absorbed water is directed toward cell expansion and initial cell wall formation in young tissues, rather than being stored as free water in mature leaf tissues.

Table III. 6. Seasonal and geographical variation in the moisture content of studied Alfa fibers.

	Moisture content (%)			
Seasons / Sites	**Winter**	**Spring**	**Summer**	**Autumn (Fall)**
Tiaret	6.56±0.32	4.03±0.27	3.27±0.24	6.27±0.35
Laghouat	5.76±0.28	4.16±0.29	4.01±0.19	4.93±0.41
Djelfa	6.47±0.67	4.45±0.21	3.23±0.31	6.10±0.33
Boussaada	5.37±0.23	4.27±0.57	4.19±0.25	4.27±0.38
Maadid	6.49±0.29	4.29±0.31	6.05±0.41	6.51±0.43

This decrease in moisture content might also be explained by the low hemicellulose content recorded in most spring samples, since hemicellulose is the most influential component in plant fiber moisture uptake due to its free hydroxyl group that binds to water [361-363]. In contrast, leaves moisture content peaked during winter, with the highest seasonal values (6.56 ± 1.01 %) at Tiaret site and (6.47 ± 0.67 %) at Djelfa site. This increase is likely due to the high relative humidity and low temperatures recorded during this season in most of the sites (Table 1). These results are consistent with previous reports [21], [360] , which linked low temperatures and high atmospheric humidity with increased water holding capacity of plant tissues. while in summer, a significant decrease in moisture content was observed in most of the studied samples, such as in AD (3.23 ± 1.03 %) and AT (3.27 ± 0.24 %), likely due to water stress, high temperatures and

increased evaporation rates (Table II.1) [364]. It should be noted that only the AM sample recorded a relatively higher moisture content during this season (6.05 ± 0.41%), possibly due to local factors, as Maadid is located in a mountainous area, which may provide unique environmental conditions such as the proximity of the water table or partial shading that contributes to reducing evaporation rates [365].

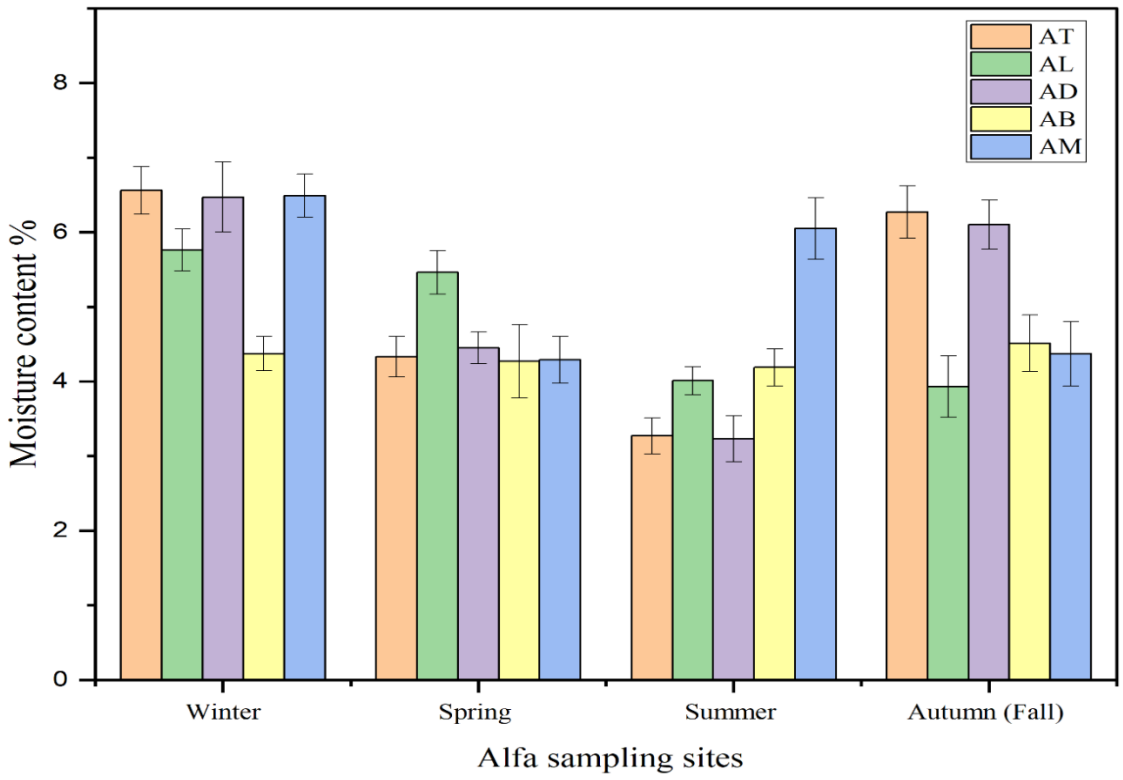


Fig III.12 Evolution of Alfa leaves moisture content across seasons and growth sites.

This seasonal variation in moisture content reflects the combined influence of several environmental factors in determining the water uptake and retention behavior of plant fibers, including: (i)The effect of relative humidity and precipitation in enhancing water uptake during wet seasons (winter and spring). (ii) Hemicellulose content as the main driver of moisture absorption due to the free hydroxyl group that binds to water [353]. (iii) the effect of high temperatures and water stress in reducing moisture during the summer. as well as soil texture and organic matter levels, and partial shading from surrounding vegetation Content, as clay soils lead to higher water retention compared to sandy soils with high permeability [365]. The impact of seasonal variations on moisture content in natural fibers is critical for optimizing their mechanical properties and ensuring their effectiveness in various applications.

III.7.3 Mechanical characterization

Mechanical analysis by tensile testing of various Alfa fibers throughout the seasons is an essential step in evaluating the strength, stiffness, and durability of fibers, allowing their suitability for various industrial applications to be determined.

The typical stress-strain curve of different Alfa fibers during seasons (Figure III.13) shows a two-phase mechanical behavior, a pattern also documented in similar studies on natural plant fibers such as Centaurea hyalolepis and Hyphaene thebaica L (Makri, et al. [10] ;Mansour, et al. [336]).

In the first stage, linear behavior is observed, characterized by an increase in stress with elongation, reflecting an elastic response of the cell wall under gradual loading. During this stage, the fibers follow elastic behavior consistent with Hooke's law, and the Young's modulus is determined from the slope of the linear portion of the curve. This behavior is attributed to the ability of cellulose chains to stretch in an orderly manner at low stress levels, indicating high initial structural integrity within the fiber structure.

In the second stage, the curve begins to deviate gradually from linearity, followed by a sharp drop in load upon reaching the failure point, indicating a brittle fracture nature, which is a characteristic behavior of plant fibers as documented by (Belouadah, et al. [366], Laifa, et al. [367], Kouidri, et al. [282],Bouchareb, et al. [34]).

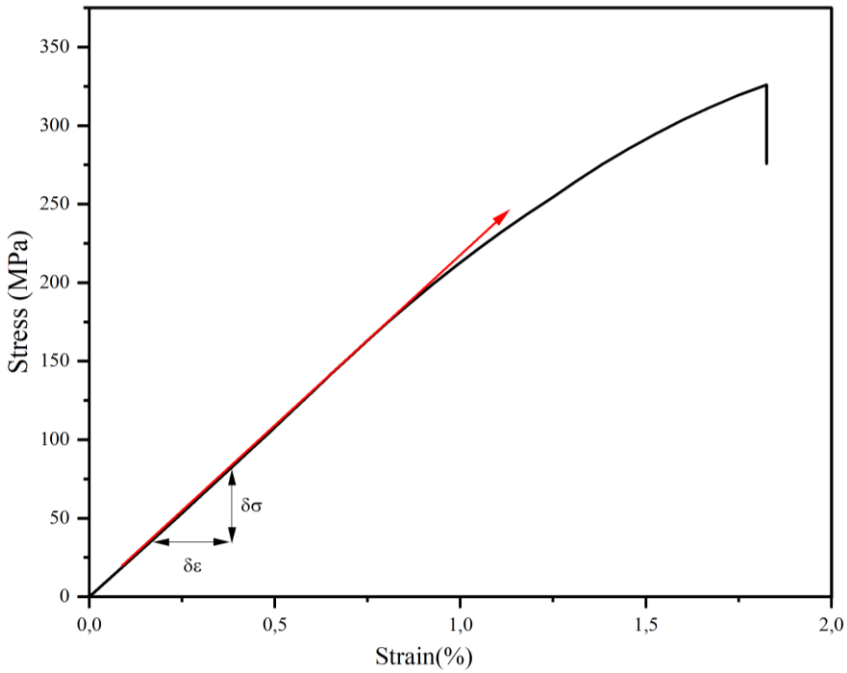


Fig III.13 Typical tensile Stress-Strain curve for Alfa fibers.

As illustrated in Figure III.14, the histograms clearly reveal variations in tensile strength, Young's modulus, and elongation at break among the different Alfa fiber samples. This suggests that seasonal variation and different growth sites have a significant impact on the mechanical properties of Alfa fibers.

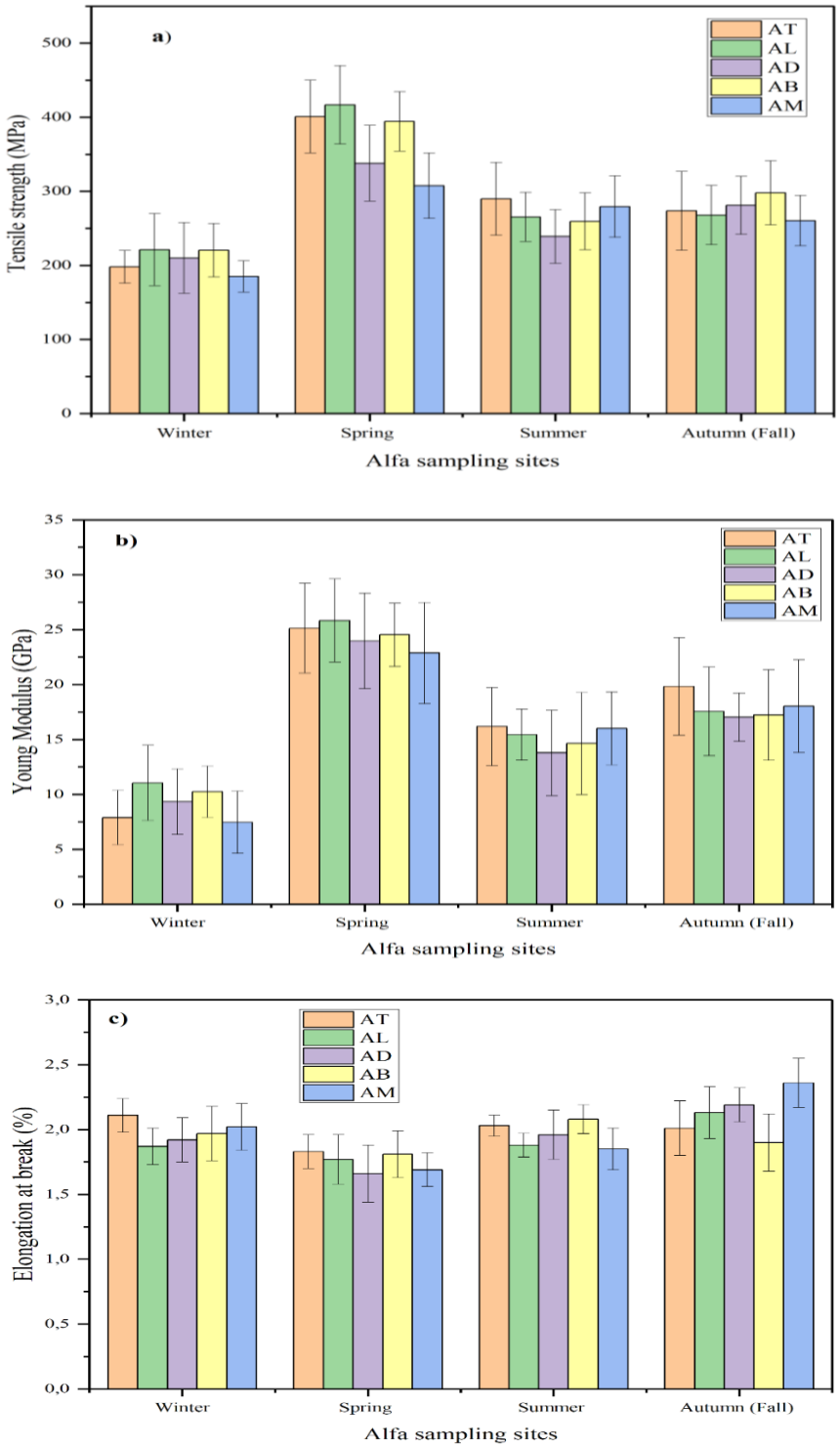


Fig III.14 Evolution of mechanical properties of Alfa fibers across seasons and growth sites: a) Tensile strength (MPa); b) Young Modulus (GPa); c) Elongation at break (%).

The results showed that fibers harvested in spring exhibited the highest values for both ultimate strength and Young's modulus (307.59±44.11MPa-416.78±53.01MPa), (22.87±4.61 GPa- 25.83±3.79 GPa), respectively, while elongation at break was moderate to relatively low(1.66±0.22 % - 1.93±0.13 %) compared to Alfa fibers harvested in the other seasons. This good mechanical performance can be attributed to several factors: (i) The high cellulose content recorded during this season, which is consistent with several studies confirming that a high cellulose content enhances tensile properties especially tensile strength and Young modulus, due to the arrangement of its linear molecular chains, which allow for efficient stress transfer through the regular fiber structure [82, 148, 368, 369]. This is also reflected in the high crystallinity index, which indicates a regular structure that contributes to improving the overall mechanical performance of the fibers [9, 369, 370] .(ii) Moderate values of hemicellulose and lignin. Several researchers have indicated that the mechanical properties of plant fibers can be improved by reducing non-cellulosic components through chemical treatments[18, 28, 34, 148, 353, 371, 372]. (iii) The relative high fiber density compared to other season, indicating structural regularity and low porosity, with narrow fiber cell lumens, as shown in the morphological images (MEB) of the cross section This structural pattern supports more uniform load transfer [373]. (iv)The low moisture content, which reduces water interference with hydrogen bonds, ensuring greater stability of bonds within the fiber and enhancing stress transfer efficiency under load (Garat, et al. [360],Liao, et al. [374]).

The moderate to low elongation at break values recorded during spring may be due to mechanical behavior reflecting stiffness resulting from high cellulose content and crystallinity index, which restricts the movement of polymer chains and reduces the ability of fibers to deform before breaking[353],[375],[376]. It is clear from the research that a high crystalline cellulose content is linked to low elongation at break and increased brittleness. On the other hand, the low hemicellulose content, which is one of the factors contributing to fiber flexibility, may contribute to the reduced elongation at break [377]. Furthermore, the decrease in moisture content during this season reduces the ability of fibers to stretch due to the reduced plasticizing effect of water[378].

The histograms of the experimental results of the tensile test in Figure III.14a clearly show that the tensile strength values of the fibers harvested during the autumn and summer seasons were similar, ranging from(260.33±34.09 MPa to 297.89±43.22 MPa) and(239.01±36.19 MPa to 289.88±49.23 MPa) respectively. This is due to the similarity in cellulose content between the two seasons, as the difference between the tensile strength of the samples from the two seasons

does not exceed 1% to 2%. This is a key factor in enhancing the fibers' ability to resist mechanical stress, as it is the most influential structural component in tensile resistance, as reported by Le Guen, et al. [379].

In contrast, summer samples showed relatively lower Young's modulus values (ranging from 13.79±3.89 GPa to 16.18±6.56 GPa) compared to the autumn samples (ranging from 17.03 ± 2.21 GPa to 19.83 ± 5.44 GPa), Figure III.14b. This can be attributed to the increase in lignin content, which shows a negative correlation with the Young's modulus, along with the decrease in hemicellulose content, which is known for its positive correlation with the Young's modulus[380]. The lower moisture content recorded in these samples may also contribute to reducing fiber flexibility[378]. The fibers selected in winter exhibit a noticeable deterioration in their mechanical properties, as evidenced by the significant decrease in both tensile strength and Young's modulus compared to the other seasons (Figure III.14a,b). This mechanical weakness can be attributed to the specific chemical composition of the fibers collected during this season, which show the lowest cellulose content, the primary component responsible for enhancing fiber strength Karimah, et al. [82].

In contrast, the lignin content increases relatively, and this aromatic polymer is negatively correlated with fiber strength and stiffness, as it promotes brittleness and reduces Young's modulus [80]. The recorded increase in hemicellulose content during spring, as an amorphous component, also contributes to a reduction in the crystallinity index of the fibers[381], weakening their internal cohesion and increasing their susceptibility to deformation [380]. Furthermore, the inhibitory effect of both high moisture content and dissolved extracts, which negatively affect the internal adhesion between cell wall components and weaken the hydrogen bonds responsible for enhancing mechanical stiffness [382]. A study by Xian, et al. [382] indicated that high moisture content leads to a decrease in the Young's modulus, as water acts as a natural plasticizer that weakens the inter-linkages within the cellulose structure, reducing fiber stiffness and increasing its flexibility. In light of these findings, the decline in the mechanical performance of winter fibers is attributed to the complex interaction between their low cellulose content on the one hand, and high levels of lignin, hemicellulose, moisture, and soluble extracts on the other, resulting in a less cohesive and regular fiber structure, and weakens their ability to effectively transfer loads. This is consistent with the review by Faruk, et al. [9] and Karimah, et al. [82], which reported that fibers with low cellulose and high lignin content exhibit limited mechanical tensile properties compared to their high-cellulose content counterpart

Table III. 7. Seasonal variation in mechanical properties of Alfa fibers from the studied growth sites.

Seasons / Sites	Winter			Spring			Summer			Autumn (Fall)		
	σ(MPa)	**E (GPa)**	**ε (%)**	**σ(MPa)**	**E (GPa)**	**ε (%)**	**σ(MPa)**	**E (GPa)**	**ε(%)**	**σ(MPa)**	**E (GPa)**	**ε (%)**
Tiaret	198.05±22.01	7.89±2.48	2.11±0.13	401.01±49.17	25.13±4.08	1.83±0.13	289.88±49.23	16.18±3.56	2.03±0.08	273.60±53.27	19.83±4.44	2.01±0.21
Laghouat	221.13±48.68	11.05±3.41	1.87±0.14	416.78±53.01	25.83±3.79	1.77±0.19	265.33±33.13	15.44±2.32	1.88±0.092	267.92±40.05	17.56±4.05	2.13±0.2
Djelfa	210.01±47.69	9.33±2.99	1.92±0.17	337.89±51.19	23.97±4,.33	1.66±0.22	239.01±36.19	13.79±3.89	1.96±0.19	281.14±39.15	17.03±2.21	2.19±0.13
Boussaada	220.47±36.16	10.24±2.35	1.97±0.21	394.29±40.09	24.54±2.88	1.81±0.18	259.51±38.49	14.65±4.65	2.08±0.11	297.89±43.22	17.23±4.12	1.9±0.22
Maadid	185.15±21.14	7.46±2.84	2.02±0.18	307.59±44.11	22.87±4.61	1.69±0.13	279.33±41.57	16.01±3.32	1.85±0.16	260.33±34.09	18.03±4.22	2.36±0.19

σ(MPa): Average Tensile strength, **E (GPa):** Average Young modulus**, ε (%):** Average Elongation at break.

III.8 Scanning electron microscopy

The effect of seasonal variations on the morphological characteristics of different of Alfa fibers can be observed by analyzing scanning electron microscope (SEM) images. In this study, a sample of fibers from the Laghouat region (AL) was selected because it showed significant properties compared to the other samples.

The examination of SEM micrographs (Figures III. 15,16) of Laghouat Alfa fibers (AL) reveals clear morphological differences across the seasons. These distinctions reflect the influence of seasonal variations on the structural composition of the fibers. The longitudinal sections display external surface characteristics, while the cross sections highlight internal structural features, both indicating a complex interaction between seasonal environmental conditions and the intrinsic structure of the fibers.

In spring, Alfa fibers (FigureIII.15a) showed a relatively smooth and homogeneous surface structure, covered with a medium layer of surface impurities, including waxes, oils, and extractables[153]. Individual micro-fiber or elementary fibers displayed a distinct degree of regularity in shape and size, with a closely spaced cylindrical structure and diameters ranging from 14 to 16 µm held together by hemicellulose and lignin[383] ,suggesting that the most of the micro-fibers had attained structural maturity. Additionally, the samples exhibited regular fiber orientation and cohesion between bundles, which may be attributed to the structural bonds provided by cell wall components, particularly hemicellulose and lignin[384]. The cross-sectional view of Alfa fibers (Figure III.15b) shows densely packed fiber cells with approximately regular circular shape, very thick cell walls with extremely narrow lumens (nearly closed lumens)[385], indicating an advanced degree of structural maturity, and a limited number of pores or voids. This suggestion aligns with the relevant findings of Richely, et al. [386], who recognized the thickness of cell walls as a reliable indicator for evaluating the maturity of plant fibers. These features are indicative of a complete cellular growth pattern and adequate moisture content, which contributes to the formation of a stable crystalline structure. This structural compactness and wall thickness is attributed to the high content of cellulose, in contrast to the low proportions of non-cellulosic components during this season[4]. This structural cohesion is consistent with the high-density values and low moisture content and is enhanced by the good thermal performance, as confirmed by the thermogravimetric analysis results.

Fall season corresponds to a renewed growth phase of Alfa fibers following a harsh summer stagnation, imparting a transitional nature to their structural and cellular organization. SEM micrographs (Figure III.15c) of Alfa fiber collected during this season showed morphological features indicating a resumption of growth activity, albeit with a lower degree of structural regularity than in the spring. A clear improvement in structural regularity and surface smoothness was observed, as evidenced by the formation of regular, aligned fiber bundles along the fiber.

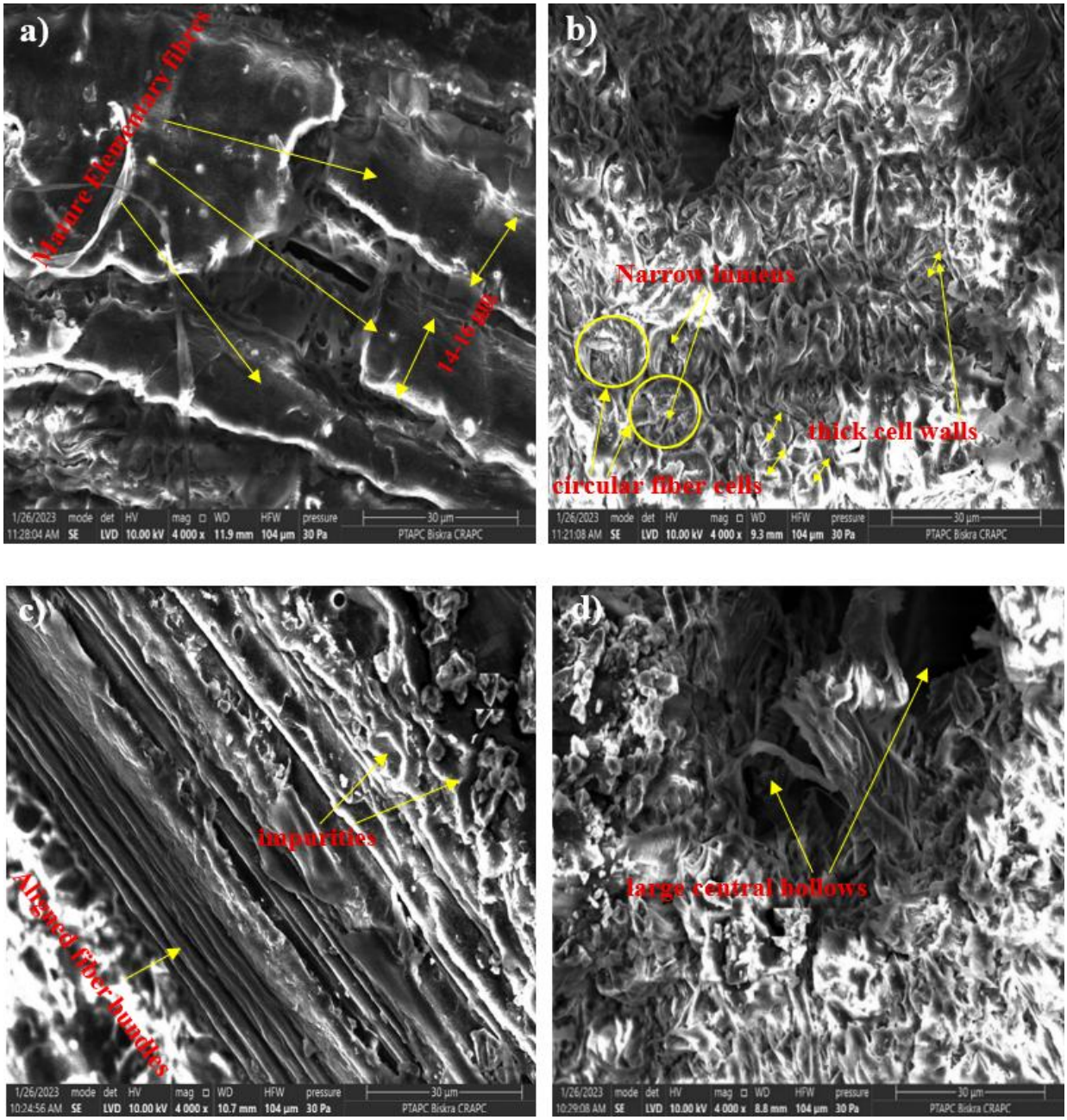


Fig III.15 SEM micrographs of longitudinal and cross-sectional views of Alfa fibers in growth phase: spring (a, b) and fall (c, d).

However, a significant amount of non-cellulosic substances and impurities still covered some fibrous surfaces, which could be due to the lingering effect of the high lignin content recorded during the summer [153].Taken together, these features indicate that the reactivation of cellular processes is still in its initial stages and has not yet reached full structural stability. In cross-section (Figure III.15d), the fibers showed a less regular structure than in the spring. Fiber cells were characterized by a quasi-circular shape, a relatively heterogeneous distribution, and cell walls ranging from moderately thick to thick. with relatively two large central hollows and limited interstitial spaces between fiber cells.

This structural pattern is likely related to a relative improvement in cellulose content compared to summer, offset by a decrease in lignin and an increase in hemicellulose content, which may support the hypothesis of a gradual restoration of biological activity in wall formation. In addition, the physical data show moderate density and relatively moderate moisture content, especially in the AL and AB samples, which may indicate a gradual functional

rebalancing after summer drought conditions. The TGA results showed a slight improvement in thermal stability, although not reaching the levels observed in the spring samples.

The longitudinal view of Alfa fiber (Figure III.16e) during winter showed morphological features reflecting a clear structural deterioration, as indicated by a rough surface with visible grooves, irregular fissures and thick layer of impurities. Elementary fibers appeared to have different diameters (from very small to medium), partially separated from each other and with a wavy appearance possibly due to environmental stress and reduced cellular activity. The micro-fibers surfaces were also covered with a clear layer, mostly composed of non-cellulosic substances like lignin, indicating defensive deposition to enhance cell wall rigidity and resistance to harsh winter conditions, especially under low cellulose content. These factors contributed to the loss of structural cohesion and the appearance of cracks and grooves extending along the fibers. In the cross-sectional view (Figure III.16f), fiber cells appeared to be of varying shapes, ranging from quasi-circular and oval to relatively irregular or slightly deformed shapes (relatively distorted). Furthermore, a significant number of wide lumens were observed, including two that appeared to be notably large, with clearly visible interstitial spaces between fiber cells. It is possible that these differences in the shape and size of the lumens can be attributed to variations in cell wall thickness[386]. The majority of walls appear to be thin, while a smaller proportion are of medium thickness. This heterogeneous structural organization reflects a weakness in structural stacking and a lack of internal cohesion.

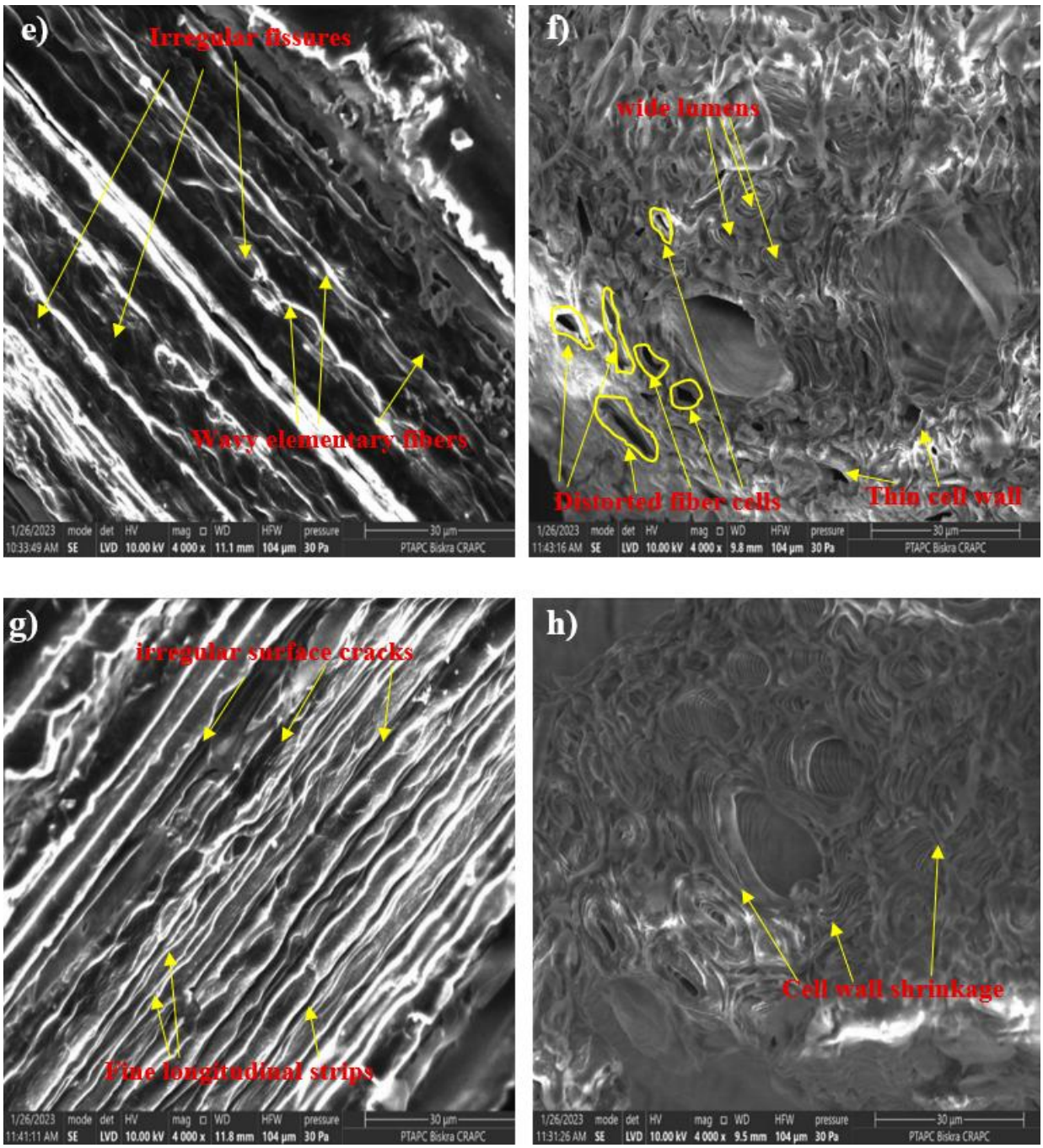


Fig III.16 SEM micrographs of longitudinal and cross-sectional views of Alfa fibers latency phase: winter (e, f) and summer (g, h).

This structural defect appears to be related to low cellulose content, high lignin and hemicellulose content, and high extract concentration. These factors may explain the fragility and irregularity of the cell walls. These observations are consistent with the recorded physical properties, which indicate a decrease in density and a relative increase in moisture content. The results of thermogravimetric analysis also show a decrease in thermal stability, reflecting a

decline in crystallinity and deterioration in molecular structure during this dormant phase of Alfa fiber cycle.

According the longitudinal view (Figure III.16g), summer Alfa fiber shows morphological features that probably indicate a significant decline in cellular activity compared to the active growth phase in spring. with fine longitudinal strips and irregular surface cracks observed, and the elementary fibers generally appear wavy. These features likely reflect a direct response to harsh climatic conditions, especially high temperatures and low moisture content. These features are thought to be the result of cell wall shrinkage caused by the contraction of internal lumens under water stress[386], which is consistent with previous studies on plant cell wall behavior under low water content. The contraction of plant fiber dimensions due to progressive dehydration has already been reported, depending on the variety, growing conditions, lignin content, and anatomical location within the stem[387, 388].

The cross-sectional configuration Alfa fibers during summer season (Figure III.16k) exhibited a distinct structural heterogeneity, characterized by a significant variation in the cell walls thickness. Some walls appeared thick and compact, while others appeared thinner, reflecting a heterogeneous tissue response to the decline in growth activity during this phase. The cell lumens also exhibited variations in shape and size, ranging from wide to medium width, narrow or shrunken, to semi-closed lumens. These variations may suggest a partial continuation of some features associated with spring growth, despite the gradual transition to summer dormancy.

This morphological diversity is attributed to the effects of environmental stresses associated with high temperatures and low humidity levels, which cause plant cells to strengthen their cell walls and reduce water loss at the expense of cell division and expansion. This is supported by the chemical results, which showed a decrease in cellulose and hemicellulose content, compared to a noticeable increase in lignin, which is known for its structural role in enhancing wall strength and stability [22].Taken together, these facts indicate that the fibers exploit resources accumulated during the period of active growth in spring to initiate structural and functional changes that enable them to adapt to the dry and harsh conditions of summer.

III.9 Conclusion

Through the chemical, physicomechanical, thermal, and morphological analyses conducted on Stipa tenacissima L fibers which collected from different growth regions across the four seasons, it was clearly demonstrated that environmental growth conditions play a decisive role in determining the structural, physical, and mechanical properties of these fibers. The results showed that climatic factors, such as temperature, water availability, and solar radiation intensity, directly influence the distribution of cell wall components (cellulose, hemicellulose, lignin, and pectin) as well as their degree of crystallinity and structural compactness.

The analyses also revealed that spring samples exhibited the best performance in chemical, thermal, and mechanical properties across all studied regions, confirming that this stage of the biological cycle represents the peak of the plant's physiological activity and the optimal conditions for forming high-quality fibers in terms of structure, stiffness, and mechanical efficiency.

Moreover, the results indicated that geographical variations significantly affect the chemical composition, crystallinity, and density of the fibers, which directly impact their mechanical and thermal behavior. Fibers collected from the Laghouat and Tiaret regions showed higher tensile strength and Young's modulus values, attributed to their elevated cellulose content, along with improved structural and thermal features.

Overall, the findings demonstrate that the chemical, physical, thermal, and mechanical properties of Alfa fibers are strongly interconnected with their microstructural organization, and that the interaction between seasonal and environmental factors governs the dynamics of cell wall formation and fiber maturation. Therefore, controlling growth conditions and selecting optimal harvesting sites can serve as an effective strategy to enhance the quality of Alfa fibers and tailor their characteristics for advanced industrial applications, particularly in the fields of composite materials and sustainable products.

Part II: Effect of chemical treatment and fiber architecture

III.10 Physical properties

In this work, the apparent density of the three types of Alfa fibers (A_{Un}, A_N, and A_P) was determined through five repeated measurements, and the mean values were then calculated. The results show that the densities of A_{Un}, A_N, and A_P fibers are 1.269 ± 0.014 g/cm³, 1.358 ± 0.029 g/cm³, and 1.460 ± 0.071 g/cm³, respectively. These values fall within the same range reported for other natural fibers such as jute, banana, kenaf, bamboo, and sisal [16, 389, 390].

It is clear that chemical treatment exerts a notable influence on the density of Alfa fibers, which agrees with previous findings in the literature [391, 392]. The increase in density observed in A_N and A_P fibers compared to untreated A_{Un} fibers may be attributed to several mechanisms:

(i) morphological modifications induced by treatment, which reduce the presence of micro-voids inside the fibers and consequently lead to higher density [393].

(ii) removal of low-density substances such as surface waxes and fatty components, which densifies the fiber cell wall [394].

(iii) partial filling of internal pores with grafted molecules generated during chemical treatment, which contributes to volume reduction and mass increase [395].

Regarding fineness, since fiber diameter and cross-sectional geometry are not constant along the length, this parameter was expressed as the linear density (mass per unit length). The equivalent diameter and linear density values of the tested fibers were calculated, and the results are summarized in Table III. 8.

From the obtained results, it was observed that the equivalent diameter and the linear density of the chemically treated Alfa fibers (A_N and A_P) were lower than those of the untreated fibers (A_{Un}). This reduction can be explained by the removal of surface impurities and waxy substances during the chemical treatment, which leads to a decrease in the fiber's overall mass per unit length [155, 396, 397]. Similar tendencies have also been reported in previous studies on other natural fibers, confirming that surface purification and structural modifications induced by treatment contribute significantly to the reduction in fiber fineness [336].

Table III. 8. Physical properties of untreated and treated Alfa fibers compared to some natural fibers.

Fibers	Density (g/cm^3)	Linear density (Tex)	Equivalent diameter (µm)	References
Untreated Alfa (A_{Un})	1.269 ± 0.014	59.015 ± 9.880	235.130 ± 27.340	Current work
Alkali treated Alfa (A_N)	1.358 ± 0.029	55.73 ± 12.394	210.120 ± 20.410	
Permanganate treated Alfa (A_P)	1.460 ± 0.071	52.363 ± 13.492	201.812 ± 22.120	
Untreated Doum Leaf sheath	0.47	94.27	689	[393]
Alkali Treated Doum Leaf sheath	0. 5 - 0.99	40.16 - 91.04	280.35 - 680.35	
Untreated Doum Leaf stalk	1.26	78.06	280.86	[397]
Alkali Treated Doum Leaf stalk	1.32	45.67	209.88	
Untreated Doum Folioles	1.15	35.70	198.45	
Alkali Treated Doum Folioles	1.41	7.14	80.31	
Untreated Sisal	1.14	24	205	

III.11 ATR-FTIR analysis

The ATR-FTIR technique was employed to investigate the chemical structure of the lignocellulosic Alfa fibers. Figure III.17 presents the spectra of untreated and chemically treated samples, highlighting the modifications induced by alkali and permanganate treatments. A broad absorption band in the region of 3610–3150 cm^{-1} corresponds to hydroxyl groups and the O–H stretching vibrations involved in hydrogen bonding within carbohydrates such as cellulose and hemicellulose [371, 398]. Two distinct peaks located at 2919 and 2840 cm^{-1} were attributed to the C–H stretching vibrations of aliphatic groups (CH and CH_2) in cellulose and hemicellulose. The gradual decrease in intensity of these peaks after chemical treatments reflects the partial removal of hemicellulose components.

The absorption peak at 1729 cm^{-1}, which corresponds to the C=O stretching vibration of non-cellulosic constituents such as hemicellulose, lignin, pectin, fats, and waxes [399, 400], disappeared completely from the spectra of both alkali- and permanganate-treated fibers. This disappearance confirms the effective elimination of these non-cellulosic compounds from the fiber surface. The peak at 1637 cm^{-1} was associated with H–O–H bending vibrations of absorbed water [401], while the small band at 1510 cm^{-1} corresponds to the aromatic C=C stretching in lignin's phenylpropane structure [402, 403]. Similarly, the band at 1418 cm^{-1} was assigned to C–H

vibrations of aromatic groups combined with C–O stretching in hemicellulose, lignin, and pectin [403].

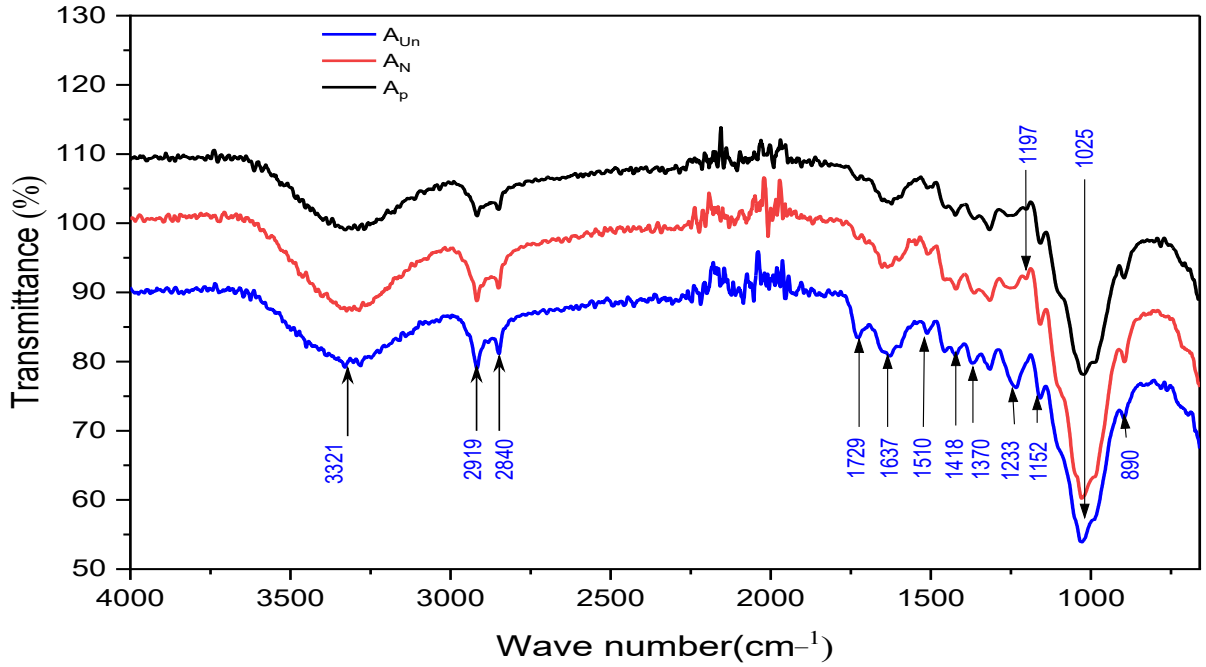


Fig III.17 ATR-FTIR spectra showing the chemical structure of untreated (A_{Un}), alkali-treated (A_N), and permanganate-treated (A_P) Alfa fibers.

The peak at 1370 cm^{-1}, assigned to C–H stretching in lignin, exhibited reduced intensity after treatments, indicating the removal of significant amounts of lignin. Furthermore, the absorption at 1233 cm^{-1}, attributed to –COO stretching of acetyl groups, was prominent in untreated fibers but almost disappeared following chemical modification, suggesting the degradation of hemicellulose [400, 401]. In contrast, the band at 1197 cm^{-1}, corresponding to the C–O antisymmetric bridge stretching of cellulose, remained unchanged, demonstrating that NaOH and permanganate treatments did not compromise the cellulose structure [404].

Additionally, the strong band at 1025 cm^{-1} was assigned to C–O vibrations of hydroxyl groups and C–O–C antisymmetric stretching in cellulose and hemicellulose [393]. Furthermore, the absorption band at 890 cm-1, assigned to the β-glycosidic linkages between monosaccharide units of cellulose and hemicellulose, was clearly identified in the spectra [393]. The ATR-FTIR analysis therefore confirms that chemical treatments removed a considerable portion of hemicellulose and other non-cellulosic compounds from the Alfa fibers.

Overall, the ATR-FTIR analysis confirms that chemical treatments significantly reduced the content of non-cellulosic components such as hemicellulose, lignin, and waxes, while preserving

the crystalline cellulose structure. This structural modification not only enhances the purity of the fibers but also improves their interfacial compatibility with polymeric matrices.

In addition, the treatments reduced the hydrophilic tendency of Alfa fibers. This reduction can be attributed mainly to two factors: (i) the decrease in available hydroxyl groups, which limits hydrogen bonding capacity within the cellulose network, and (ii) the removal of carboxyl groups combined with the incorporation of Mn=O functionalities during permanganate treatment. These structural changes reduce water affinity and thereby further promote the compatibility of Alfa fibers with hydrophobic polymer matrices.

Table III.9. Main characteristic peaks of ATR-FTIR spectra as reported in the literature.

Wave numbers (cm^{-1})	Vibrations modes	Sources	References
3610 –3150	O–H stretching	Cellulose, hemicelluloses	[371, 398]
2919 –2840	C–H stretching	Cellulose, hemicelluloses	[400]
1729	C = O stretching	Hemicelluloses, lignin and extractives	[399, 400]
1637	H–O–H	bending band of absorbed water	[401]
1510	C=C aromatic ring	Lignin	[402, 403]
1370	C–H stretching	Lignin compounds	[402]
1233	-COO stretching	Hemicelluloses	[400, 401]
1197	C–O bridge stretching	Cellulose	[404]
1025	C–O–C bridge stretching	Cellulose, hemicelluloses	[393]
890	β-glycosidic linkage	Cellulose, hemicellulose	[393]

III.12 X-ray diffraction analysis

XRD is a non-destructive technique used to determine the crystalline structure of materials. Figure III.18 shows XRD patterns of three types of Alfa fibers. Each diagram represents two main peaks. For A_{Un}, A_{N}, and A_{p}, the first peak represents the overlapped peak [400] of hemicellulose, corresponding to the amorphous plane I_{am} demonstrated at 2θ = 16.279°, 16.402° and 16.155° respectively. The second intense peak corresponds to the crystallographic plan of cellulose I_{002}, which is represented by the crystalline peaks observed at 2θ = 22.120°, 22.366° and 22.243° respectively. The crystallinity index, CI of A_N was higher at 61.255% followed by A_p at 60.909 % then A_{Un} at 57.767%. It seems that the fibers treated with alkaline or permanganate were better performed than raw fibers. The increased index (CI) is due to the removal of hemicellulose and

non-cellulosic components, better packing of cellulose chains, and rearranging of crystalline regions [405]. This result supports what the ATR- FTIR spectra and SEM analysis showed in previous results.

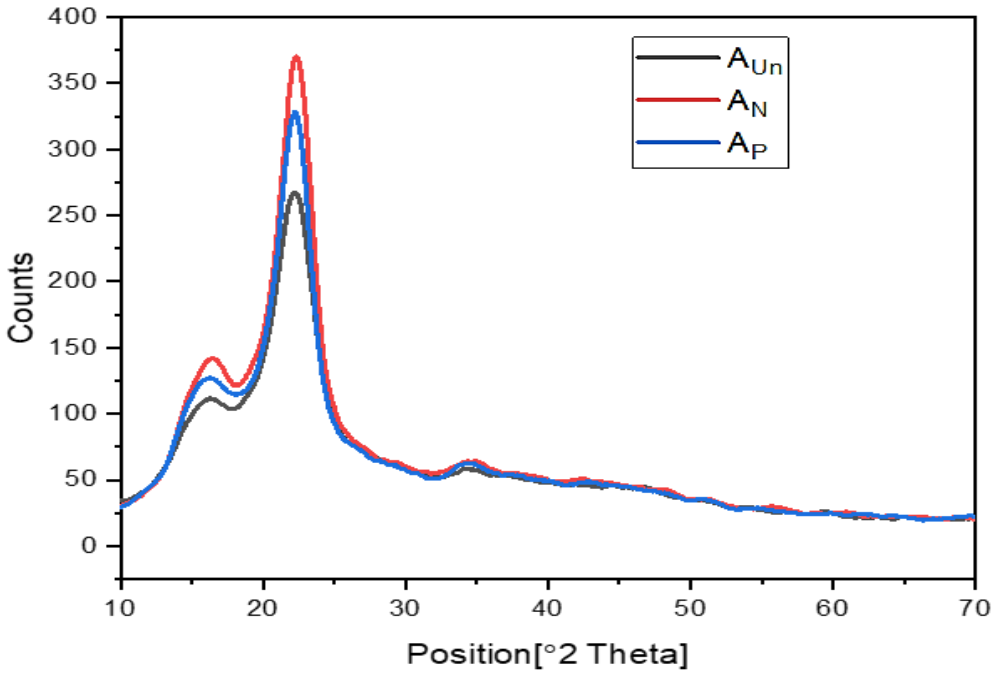


Fig III.18 X-Ray diffraction patterns of A_{Un}, A_N and A_p.

III.13 Tensile test

An average of 30 samples were selected from raw, alkali, and permanganate fibers for the tensile test. The results of tensile properties (tensile strength, Young's modulus, and elongation at break) are presented in Table 4 and compared with those mentioned in some published works.

Figure III.19 shows the typical stress-strain of A_{Un}, A_N, and A_p. fibers. All tested fibers have exhibited brittle behavior, accompanied by a sudden load decrease until failure. All tested fibers displayed two phases of behavior. The first phase showed linear deformation of nearly 0,99 %, 1.36 %, and 1.42 % of strain for A_{Un}, A_N, and A_p fibers, respectively. The Young's modulus for each fiber was calculated using the slope of the curve in this linear region. In the second phase, nonlinear deformation continued until the maximum stress was reached.

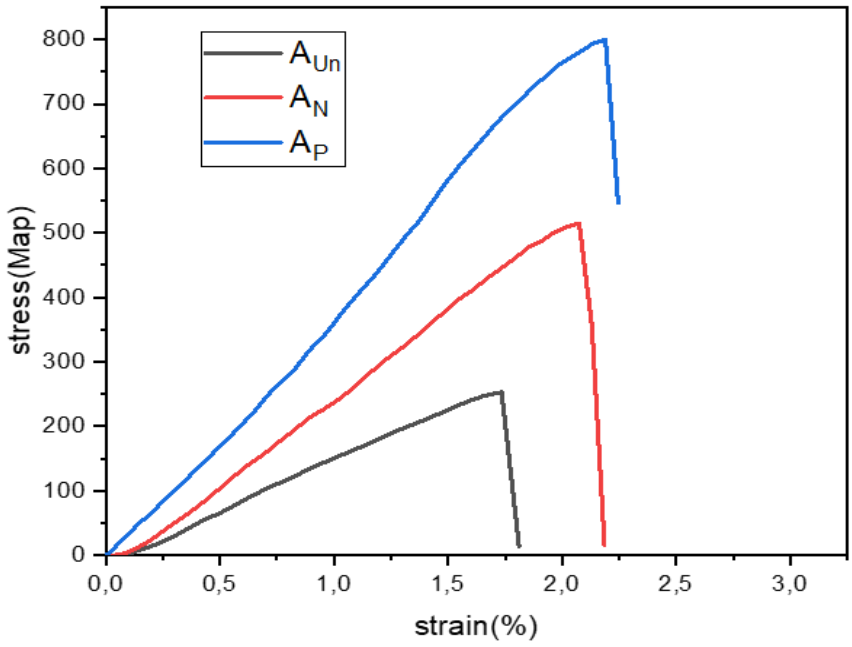


Fig III.19 Typical Stress–strain curves of A_{Un}, A_N and A_p.

It can be seen that the treated A_N exhibits higher tensile strength and elongation at break compared to the raw fibers, while Ap fiber records the highest values. This could be attributed to ; (i) the removal of hemicellulose and lignin from the interfibrillar regions which leads to improving the cellulose chain packing along the direction of tensile deformation [406]; and (ii) the increasing of cellulose content after the chemical treatment of fibers [148]; and (iii) increased CI of the cellulose. Madhu, et al. [407] have found that the treated Agave Americana fiber has a higher percentage of cellulose when compared to the untreated one. Many studies confirm the positive correlation between the amount of cellulose and the tensile strength of the fiber [408, 409]. In other words, the high cellulose content leads to enhanced tensile strength and Young's modulus of fibers. Conversely, the hemicellulose content will reduce the strength of the fibers.

Both treatments have significant effect on the fiber performance. Alkali treatment can enhance the cellulose content [410] , which subsequently increases the tensile strength of the resulting composite material . Research indicates that using a NaOH solution for fiber alkalization is more effective than potassium hydroxide (KOH), maleic anhydride ($C_4H_2O_3$), or silane (SiH_4) in improving the compaction between the fiber and the matrix [150] .In other hand, our results show that the use of permanganate treatment can give the fiber more reliable mechanical properties. This is due to: (i) the modification of the fiber surface, which creates an oxidative layer that enhances chemical interaction with the polymer matrix [411]. (ii) reducing water absorption (via hemicellulose reduction)[412], and (iii) increasing hydrophobicity by

disrupting hydrogen bonds and generating Mn^{3+} ions[413]. This is consistent with the study by Judawisastra and Refiadi [152].

Table III. 10. Comparison of Mechanical properties of raw and treated Alfa fiber with other fibers.

Fibers	Tensile strength (MPa)	Young's modulus (GPa)	Elongation at break (%)	References
Untreated Alfa (A_{Un})	297.82 ± 55.76	16.78 ±5.34	1.44 ± 0.28	Current work
Alkali treated Alfa (A_N)	504.04 ± 95,.49	24.77± 3.33	1.83 ± 0.27	
Permanganate treated Alfa (A_P)	612.19 ± 34.86	27.68 ± 3.719	1.90 ± 0.35	
Untreated Sansevieria cyl (SCF)	656.72 ± 46	5.96 ± 5.50	11.03 ± 1.40	[414]
Alkali treated (ASCF)	721.82 ± 55	5.45 ± 3.60	13.24 ± 1.20	
Permanganate treated (PSCF)	811.73 ± 24	4.16 ± 2.80	19.51 ± 1.30	
Untreated Strelitzia reginae (SRF)	148.16-420.36	6.02-13.68	2.45-3.43	[389]
Alkali treated Strelitzia reginae (SRF)	269.45-533.33	60 - 80	1.2 - 1.6	
Banana	529 -759	8 - 20	1 - 3.5	[390]
Bamboo	440 - 600	35 - 46	1.4	
Sisal	227-700	9 – 20	3 – 14	
Hemp	550-900	70	0.8-3	[415]
Kenaf	223-1191	11-60	1.6-4.3	
Bagasse	170-350	27-40	6.3-7.9	

III.14 Micromechanical test

The interfacial shear strength results for various Alfa fibers and polyester droplets are shown in Table III. 11. These results are compared with existing literature. The permanganate-treated Alfa fibers exhibited the highest interfacial shear stress at 11,474 ±0.531 MPa, followed by alkali-treated fibers at 7.794 ± 1.757 MPa, and untreated fibers at 2,895±0.710 MPa. The enhanced interfacial shear stress in the treated fibers can be attributed to: (i) increased friction due to greater surface roughness, and (ii) improved mechanical interlocking and a larger contact area between the matrix and fiber. This highlights that chemical treatments significantly enhance the adhesion properties at the fiber/matrix interface. This aligns with the findings of Sreenivasan, et al. [149] and Moussaoui, et al. [391] which indicate that combined treatments may demonstrate even greater effectiveness [416].

Table III. 11. Interfacial Shear Strength of Alfa fibers and Polyester Droplets: Comparison with literature.

Fiber /matrix	IFSS (MPa)	References
Alfa (A_{Un})/ UP*	2.895 ± 0.910	Current Work
AT** Alfa (A_N)/ UP	7.794 ± 0. 570	
P Alfa (A_P)/ UP	11.474 ± 0.531	
Abaca / UP	0.89	[83]
AT abaca / UP	5.415	
GPS Silane treated abaca / UP	5.887	
Kenaf / UP	5.729	
AT kenaf / UP	7.221	
GPS Silane treated kenaf / UP	7.780	
Oil palm / UP	4.057	
AT oil palm / UP	5.311	
GPS Silane treated oil palm / UP	5.485	
Inula Viscosa/ Epoxy	2.93	[57]
AT inula Viscosa/ Epoxy	3.36	
P*** inula Viscosa/ Epoxy	4.50	
Hemp/polylactide	5.55	[84]
AT Hemp/ polylactide	11.41	
AT and silane treated Hemp/polylactide	9.87	
Hemp/ UP	9.9	
AT Hemp/ UP	11.7	
AT and silane treated Hemp/ UP	20.3	

(*) Resin: Unsaturated polyester, (**) Alkali treated: AT, (***) Permanganate treated: P

III.15 Scanning Electron Microscopy

Scanning Electron Microscopy (SEM) can be used to determine the surface morphology of three studied Alfa fibers. Figure III.20 shows the SEM micrographs of the longitudinal surface of these fibers. From Figure III.20 (a ,b) it can be seen that the A_{Un} fiber surface consists of several small conical protrusions spines (trichomes) composed of silica [151], with a length of (50 -15) µm homogeneously distributed on the external surface of the fiber. The surface of the spines is uneven and appears to be covered with an amount of non-cellulosic compounds (such as fatty and waxy substances). The noticeable presence of these granules on the surface may negatively affect the nature of interfacial adhesion between fibers and matrix [153]. However, the alkali treatment can significantly improve the surface of Alfa fiber. As indicated by Figure III.20 (c, d), the surface of Alfa fiber became clean after alkali treatment and considerable quantities of spines disappeared from the surface of the treated fiber. Moreover, the alkali treatment effectively participates to fragment the fiber bundle into smaller microfibrils Figure III.20d, thereby enhancing the effective surface area. These results are in agreement with those obtained by other researchers in this area[155, 336, 371]. The treatment of Alfa with permanganate seems to have more impact on the

surface morphology of Alfa fibers, Figure III.20(e, f) shows clearly that almost all spines disappeared from the surface and only a circular trace remains in the form of holes or pits.

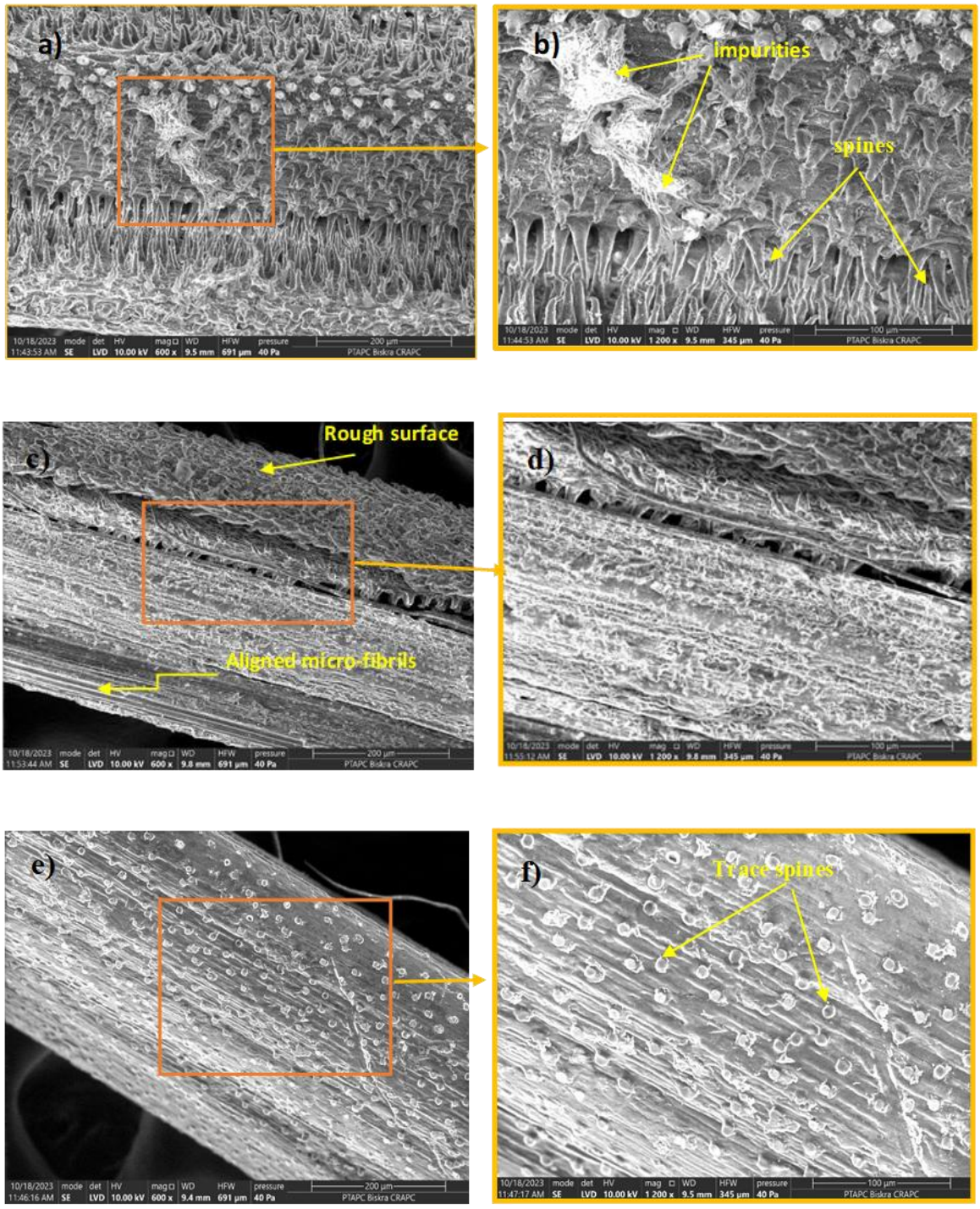


Fig III.20 SEM micrographs of the longitudinal surface of: (a, b) A_{Un}, (c, d) $A_{N,}$ and (e, f) A_p.

Figure III.21 represents the SEM micrographs of the cross-sectional area of A_{Un}, $A_{N,}$ and A_P fibers. The presence of lumens can be seen in Figure III.21(a, b). Similar observations have been reported by Senthamaraikannan and Kathiresan[400] . The disappearance of almost all of the

lumens in both cross-section micrographs of A_N and A_P Figure III.21(c, d, e, f) following the chemical treatment. This may be attributed to the filling up of the lumens [417].

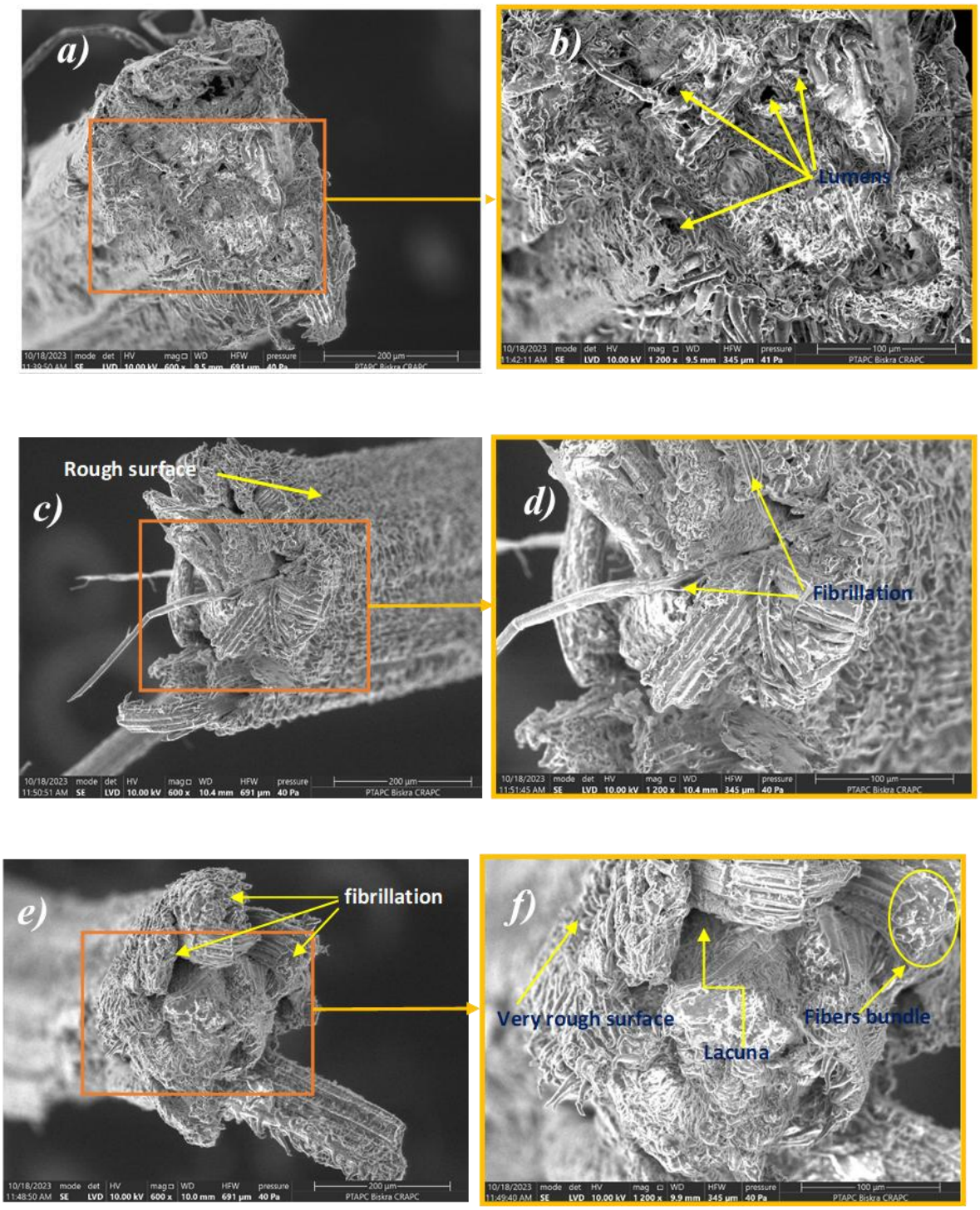


Fig III.21 SEM micrographs of the cross-section of: (a, b) A_{Un}, (c, d) $A_{N,}$ and (e, f) A_p.

The cross-section views of treated fibers, Figure III.21(c, d, e, f), confirmed the reduction of spines and removing the binding materials such as pectin, lignin, and hemicellulose. As a consequence, the surface of treated Alfa fibers is very rough compared to the untreated fibers, and the exposed elementary fibers are visible (fiber fragmentation). One impact of these morphological modifications is an increased effective surface area available for contact with any matrix [179, 392]

III.16 Mechanical and physical Characterization of intra-ply hybrid fabric composites

III.16.1 Density and void content

The density and void content of various composite materials is summarized in Table III.12. According to Sharma, et al. [418], a void content of more than 5 % indicates poor composite quality, while a void content of less than 3% reflects good quality. The lower void content signifies improved bonding between the matrix and reinforcement, which enhances the composite properties. In this work, Comp-A exhibiting the highest percentage (5.854 %), followed by Comp-D (3.861%). These elevated levels of void content are likely due to the use of untreated Alfa and jute fibers in these intra-ply hybrid composites. Conversely, the lower void content observed in Comp-C (1.174 %) and Comp-B (2.723 %) can be attributed to the effect of chemical treatments on Alfa fibers. On the other hand, the density seems to be better for the composites with treated fiber than the composites with the untreated ones.

Table III.12. Densities and void content of different material composites.

Composite	Density ($g \cdot cm^{-3}$)		Void (%)
	Experimental	Theoretical	
Comp-A	1.180 ± 0.0015	1.253 ± 0.0003	5.854 ± 0.108
Comp-B	1.205 ± 0.0019	1.238 ± 0.0018	2.723 ± 0.030
Comp-C	1.272± 0,0006	1.257 ± 0.0018	1.174 ± 0.101
Comp-D	1.173 ± 0.0024	1.220 ± 0.0021	3.861 ± 0.035

III.16.2 Tensile properties

This section discusses the results of static tensile tests conducted on four composites based on polyester reinforced with hybrid intra-ply woven fabric A_{Un} /J, A_N /J, A_P /J, and J/J respectively. Figure III.22 shows that these materials composites exhibited a brittle behavior. Each curve

showed linear progression up to 1 % - 1.5 % deformation, followed by controlled behavior until brittle failure of the composites. Furthermore, and as illustrated in Figure III.22, the composite materials reinforced by intra-ply fabric J/J (Comp-D) registered the highest tensile strength (σ = 40.78 ± 1.37 MPa) and a strain ε = 2.389 ± 0.001 % values, while the two composite materials reinforced by intra-ply woven fabric A_N /J and A_P /J (Comp-B, Comp-C) respectively appear to have pretty near values σ = 23.47± 4.62 MPa, σ = 23.08± 3.43 MPa and the same deformation Finally, the composite material reinforced by intra-ply woven fabric A_{Un} /J (Comp-A) records the most basic values (breaking strength σ = 19.86 ± 2.79 MPa and ε = 1.665 ± 0.001 %) compared to other materials composite studies.

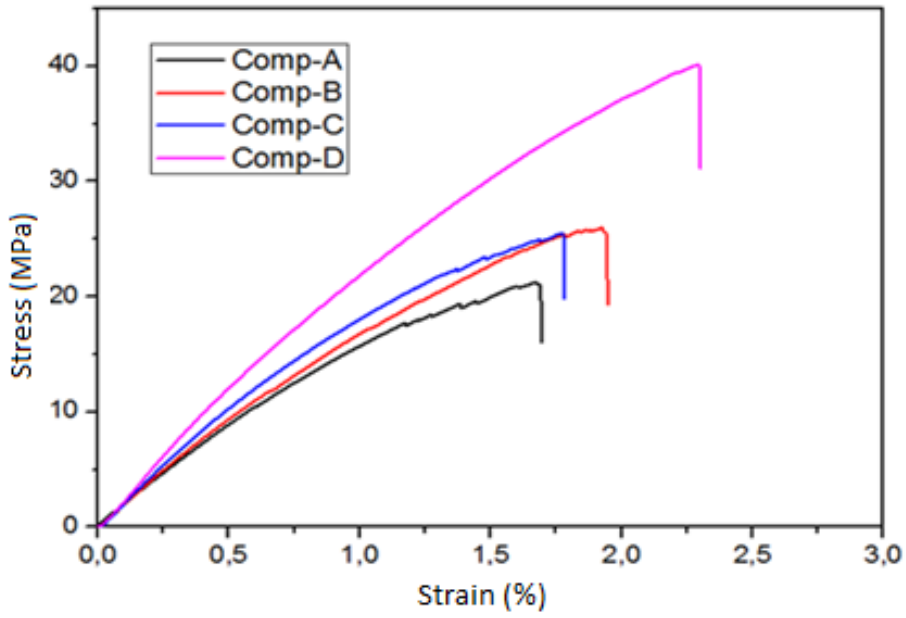


Fig III.22 Typical Stress–strain curves of different material composites.

Figure III.23 (a,b) depict the histograms of the tensile strength and modulus of various hybrid materials. The composite material reinforced by intra-ply woven fabric J/J (Comp-D) exhibits the highest stiffness (E = 2345.10 ± 180 MPa), followed by both materials composite reinforced by intra-ply woven fabric A_P /J and A_N /J (Comp-C and Comp-B), with a modulus of E = 2229 ± 120 MPa and E = 2176 ± 110 MPa, respectively. It is noted that the composite materials reinforced by intra-ply woven fabric A_{Un} /J (Comp-A) exhibited a lower rigidity (E = 1536,80± 322,409 MPa) than the composites previously discussed.

The tensile strength and Young's modulus values of the Comp-B and Comp-C demonstrated an improvement of 17 % and 30 % respectively in comparison to the material Comp-A. This improvement appears to be the result of Alfa fiber chemical treatments, as indicated by the

previous results of both equivalent diameters and linear density, ATR-FTIR, and SEM analysis of Alfa fiber, the chemical treatment of this fiber has resulted in the removal of impurities and non-cellulosic components, which has led to a rougher surface. This latter facilitates both bonding reactions of the fiber with the polyester matrix, and the mechanical interlocking when the matrix

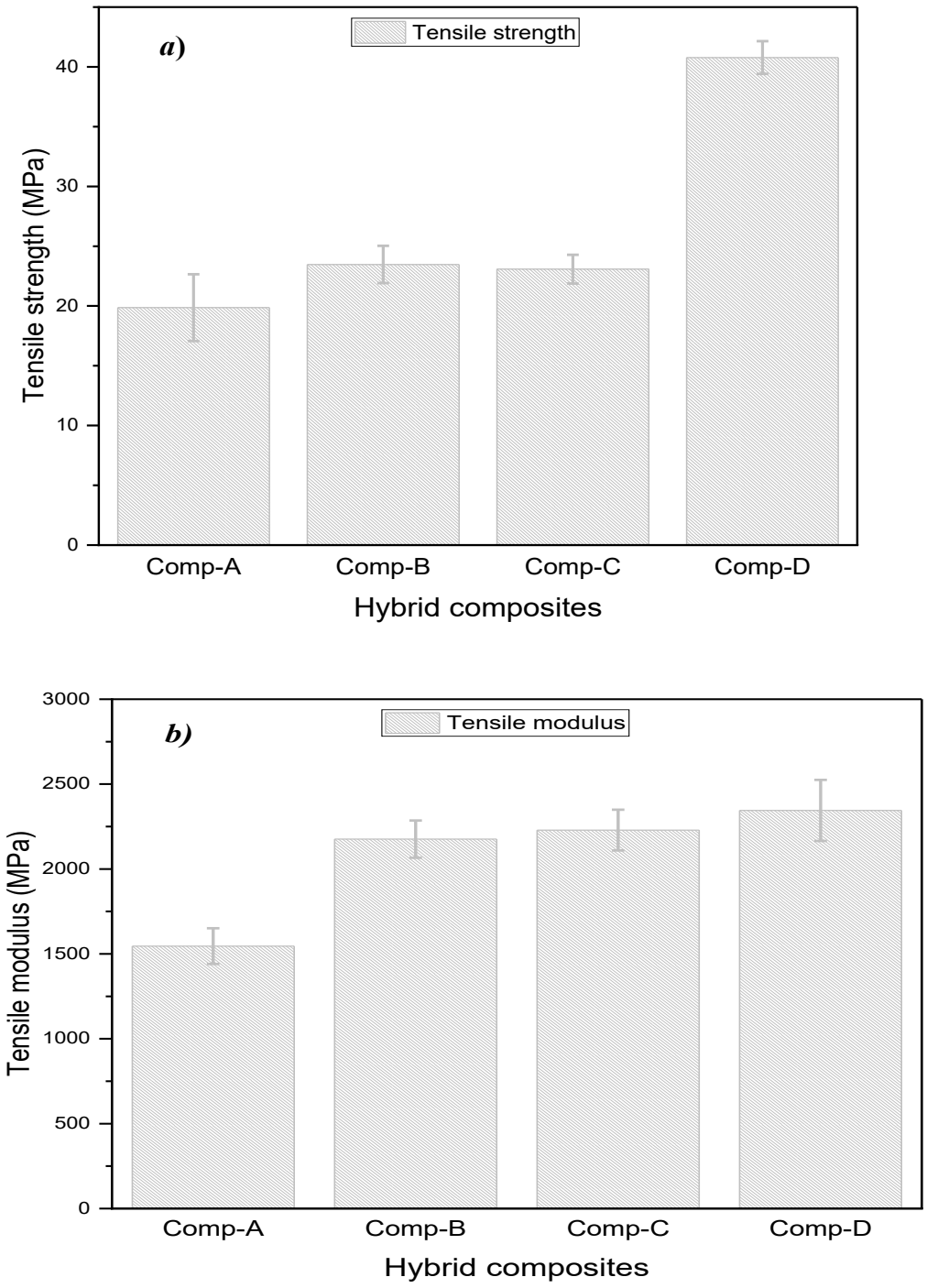


Fig III.23 Evolution of tensile properties of different hybrid composites: a) Tensile strength, b) Tensile modulus.

penetrates the crevices, peaks, and valleys or other irregularities of the substrate, thereby better interfacial interaction between the fiber and matrix is achieved [419]. On the other hand, the nature of woven fabrics (i.e., weaving or hybridization). Alavudeen, et al. [33] have indicated that weaving natural fibers in different directions makes the composites stronger, and can be compared to those reinforced with synthetic ones. In contrast, Bandaru, et al. [420] have reported that intra-ply hybrid composites with an alternating weave of Basalt and Kevlar yarns exhibit better static properties than inter-ply hybrid composites, which comprise alternating layers of Kevlar and Basalt fabrics. A comparison of the results presented in Table III.13 indicates that the tensile strength of composite reinforced with permanganate-treated Alfa is higher than that of polyester/ Untreated Jute-Glass (17.9 MPa) [421] , and is close to that of polyester/ Untreated Banana-Jute (27.36 MPa) [422]. However, the tensile strength of our materials is less than that of polyester/permanganate-treated Jute-Banana (35.2 MPa) [423] and polyester/Untreated Jute-Ramie (37.20 MPa) [424].

III.16.3 Flexural properties

The objective of this section is to identify the mechanical properties of different composites. Figure III.24 depicts the typical evolution of stress as a function of strain for specimens of the four composites subjected to static bending. From this Figure, it is clear that the best performance among the studied composites is exhibited by composite materials reinforced by intra-ply woven fabric A_N/J Comp-B ($\sigma = 66.63 \pm 9$ MPa), followed by composite material reinforced by intra-ply woven fabric J/J Comp-D ($\sigma = 61.19 \pm 6$ MPa), then, the material composite reinforced by intra-ply woven fabric A_P /J Comp-C ($\sigma = 52.00 \pm 8$ MPa). Finally, the material intra-ply woven fabric A_{unt} /J Comp-A ($\sigma = 46.30 \pm 6$ MPa).

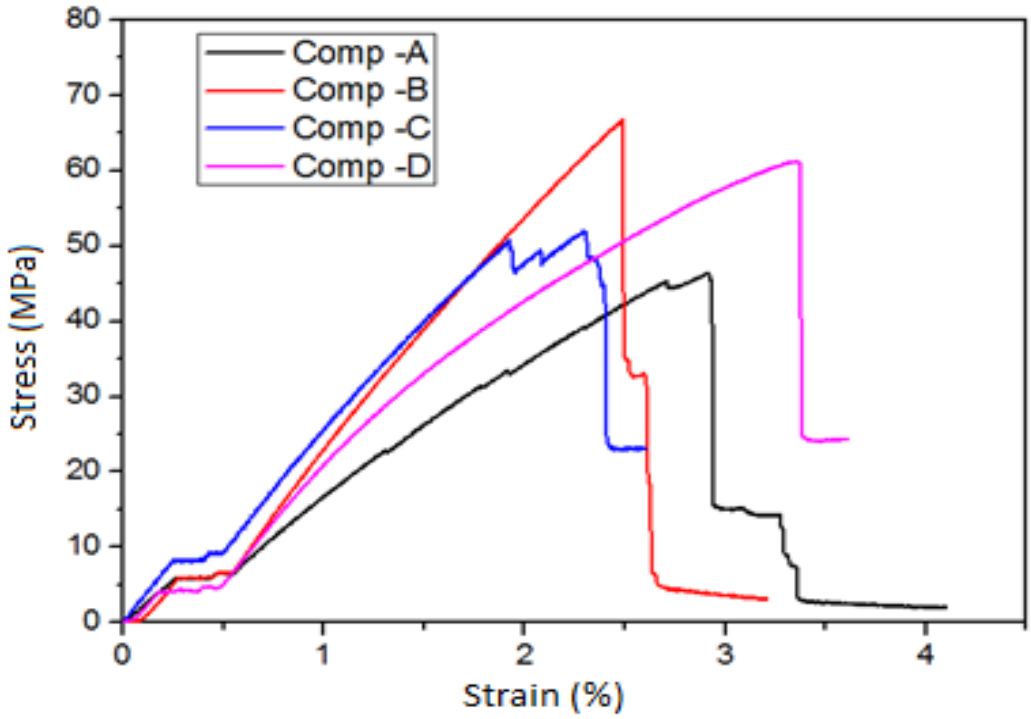


Fig III.24 Evolution of the stress as a function of the deflection.

Figure III.25 a and b present histograms of flexural strength and stiffness modulus values for various hybrid materials tested. Comp-B exhibits the highest stiffness (E = 3.472 ± 0.12 GPa), followed by Comp-C (E = 2.99 ± 0.17 GPa) and Comp-D with a modulus of E = 2.63 ± 0.12 GPa. Finally, the Comp-A material exhibited a stiffness modulus of E = 1.715 ± 0.08 GPa (Figure III.25 b). Notably, this value is lower than those observed for the other tested composites.

The evolution of these materials in flexion and tensile is not gone as inspected. Generally, for this type of materials, tensile and flexural strength and modulus should follow similar trends, as indicated by the reviewer. In other hand, some researchers such as Kumar, et al. [425],Cavalcanti, et al. [426] and Senthamaraikannan [427] indicate that in hybrid composites these properties didn't follow similar trends. For instance, the study of Kumar, et al. [425] ,on woven composites, shows that some composites had strong tensile properties, however others composite excelled in flexural strength. Similarly, Cavalcanti, et al. [426] research revealed that untreated jute and Curaua composites demonstrated high tensile strength, whereas these composites performed better in flexural strength. In another study [427], treated interwoven flax/sisal showed the highest tensile strength, while treated inter woven jute/sisal excelled in flexural strength. There may be a correlation between the weaving pattern and the tensile and flexural properties of the tested materials.

Table III.13. shows that the best flexural strength performances of composites among all the investigated composites and previous studies are evidenced by composites reinforced with alkali-treated Alfa fiber Comp-B. Furthermore, the flexural strength of composites reinforced with permanganate-treated Alfa Comp-C is higher than polyester /Untreated Jute -glass (39.6 MPa), and polyester / Untreated Banana - Jute (41.37 MPa). And comparable to polyester / untreated Jute- Banana (54.00 MPa). It's important to signal that both tensile and flexural moduli obtained in this work are the best values compared to those in Table III.13. These results reflect the good rigidity and resistance to extension of the studied intra-ply woven hybrid composites.

Three-point bending tests have demonstrated that alkali and permanganate treatments of Alfa fiber and intra-ply woven hybridization of Alfa fibers with Jute have also a significant impact on the flexural properties of composite materials.

Table III. 13. Comparison of mechanical properties of Alfa intra-ply woven fabric composites (*) with other previous studies.

Intra-ply woven fabrics (**)	Tensile strength (MPa)	Tensile modulus (GPa)	Flexural strength (MPa)	Flexural modulus (GPa)	References
Untreated (A_{unt} / J)	19.86 ± 2.79	1.54 ± 0.23	46.30 ± 8	1.72 ± 0.08	Current work
Alkali treated (A_N/J)	23.47 ± 4.62	2.18 ± 0.11	66.63 ± 9	3.47 ± 0.12	
Permanganate treated (A_P /J)	23.08 ± 3.43	2,23 ± 0.12	52.00 ±8	2.99 ± 0.17	
Untreated (J/J)	40.87 ± 1.37	2.35 ± 0.18	61.19 ±6	2.63 ± 0.12	
Untreated B / J	27.36 ± 5.90	1.50 ± 0.10	41.37 ± 11.71	1.66 ± 0.51	[422]
Untreated J / B	33.40 ± 2.41	1.75 ± 0.05	54.00 ± 5.8	2.60 ± 0.21	
Alkali treated J/ B	35.00 ± 3.05	1.83 ± 0.08	58.00 ± 2.3	2.85 ± 0.10	[423]
Permanganate treated J/ B	35.20 ± 2.38	1.48 ± 0.05	58.00 ± 3.6	2.81 ± 0.15	
Untreated J / G	17.90	0.93	39.60	1.72	[421]
Untreated J / R	37.20	3.80	-	-	[424]

(*) Resin: Polyester & (**) A: Alfa – J: Jute – B: Banana – G: Glass – R: Ramie.

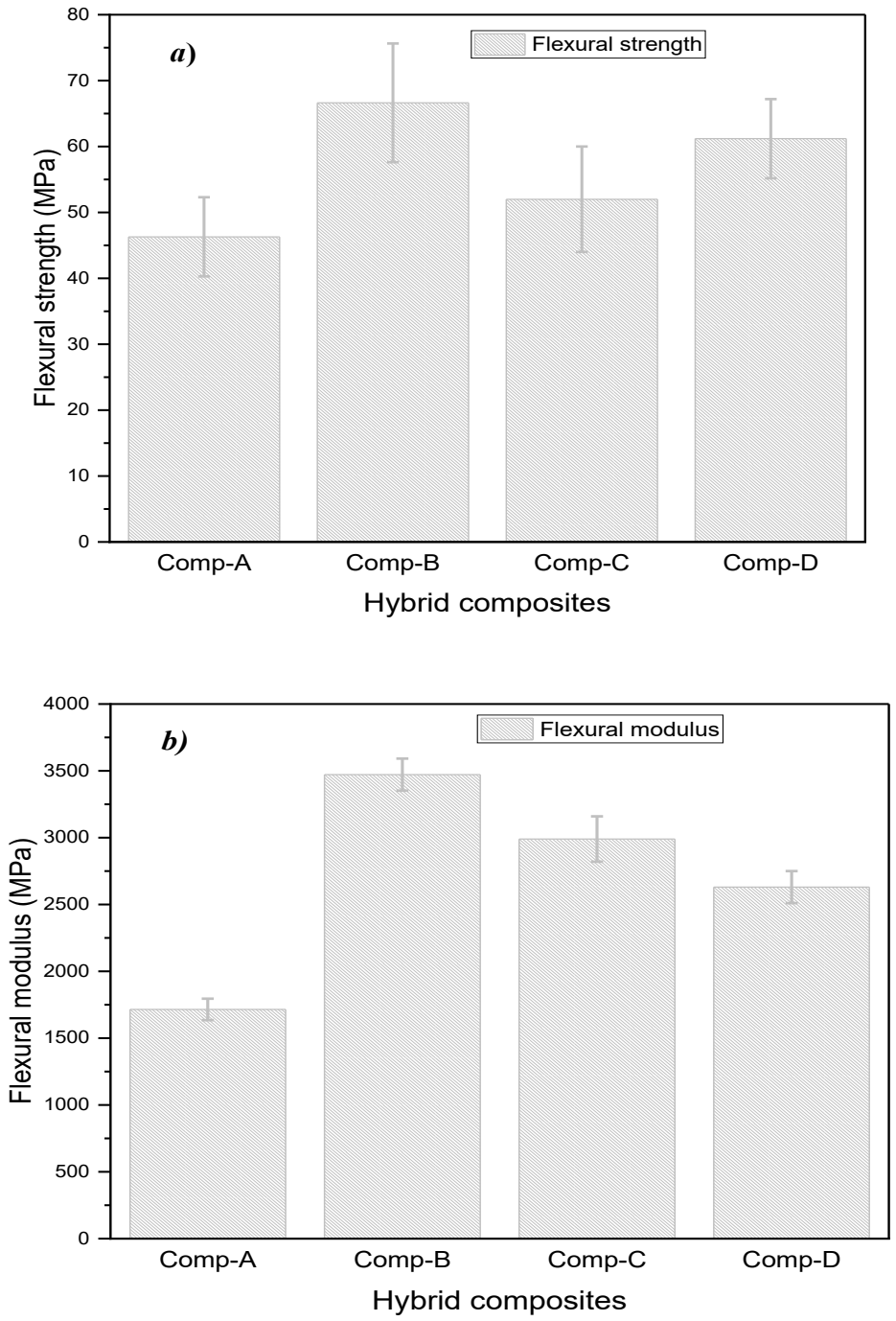


Figure III.25 Evolution of flexural properties of different hybrid composites: a) Flexural strength, b) Flexural modulus.

Generally, the performance of composite mainly depends on the fiber nature , its volume fractions, matrix, and manufacturing process [428]. During the fabrication process, the final intra-layer hybrid fabric composites are not free from imperfections which can manifest as voids, gaps and/or overlaps, and twisted tows. Even with advanced automated manufacturing technologies

such as automated fiber placement, these defects are consistently detected and can significantly impact their mechanical properties [429]. Experimental or numerical investigations indicated that gaps defects are the most common [430, 431]. Sun, et al. [429]demonstrates that the presence of gaps within composite materials can lead to the formation of resin-rich areas or induce fiber waviness. These phenomena critically influence the mechanical properties of the composites, compromising their structural integrity and performance. 4 % gap size relative to the strand width can lead to a 22 % reduction in main stiffness compared to fiber fabric without gaps. On the other hand, the presence of gaps in the laminate may diminish its mechanical strength due to a reduced fiber content compared to a laminate of the same design that is free of gaps. It was shown that the Optimally sized gaps in the pattern can enhance through-thickness permeability with minimal impact on compressive properties, though larger gaps reduce mechanical performance [432]**.** Suemasu, et al. [433] demonstrated through their analytical solution that the impact of gaps on stress becomes less significant as the gap size decreases. This is the case of our intra-layer hybrid fabric, Figure III.26, which shows clearly the special care taken during the weaving process to minimize the gap effects.

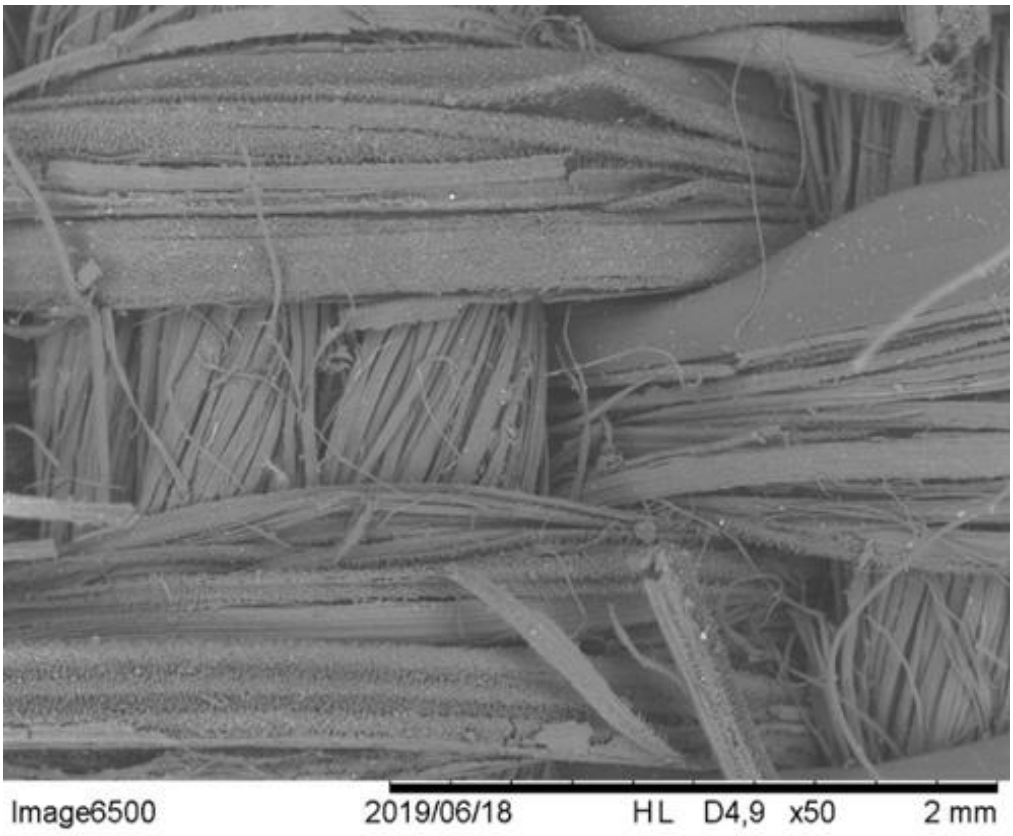


Fig III.26 Micrograph of inter woven fabric showing inter-yarns.

III.16.4 Scanning electron microscopy of hybrid composites

The scanning electron micrographs-cross-sections of different studied composites are illustrated in Figure III.27(a-f). From these photographs, the various composite materials seem to be void-free, this can be attributed to the composite manufacturing resulting from vacuum infusion molding. In contrast to other techniques that could potentially induce the formation of voids within the composites. Kadem, et al. [434] have concluded that the use of molding techniques to create composites reinforced with treated and untreated Alfa fibers reveals the presence of some defects in manufactured composites. These defects are manifested in the form of voids, which are caused by air bubbles. In addition, the resin penetration is observed in all plant fiber cell walls of the different composite cross-sections with varying degrees of penetration [435]. A better resin penetration is well observed in this composite reinforced with treated fibers, cases of Comp-B and Comp-C Figure III.27 (c-f), than in composite reinforced with untreated fibers (Comp-A) Figure III.27 (a, b). This can be interpreted by the gradual elimination of non-cellulose compounds (such as waxes, gummy and adhering non-fibrous materials), which leads to fibrillation. In other words, following chemical treatment, thinner and lighter fibers were obtained, i.e. breaking down of fiber bundles into smaller fibers is observed, thereby increasing the effective surface area available[393]. Furthermore, alkali and permanganate treatments contributed to the improvement of the fiber surface quality. By elimination of unwanted materials adhering on the raw fiber surface, the fiber surface became rough and more fibrils appeared, which increased significantly the compatibility between the treated fiber and the matrix through mechanical interlocking.

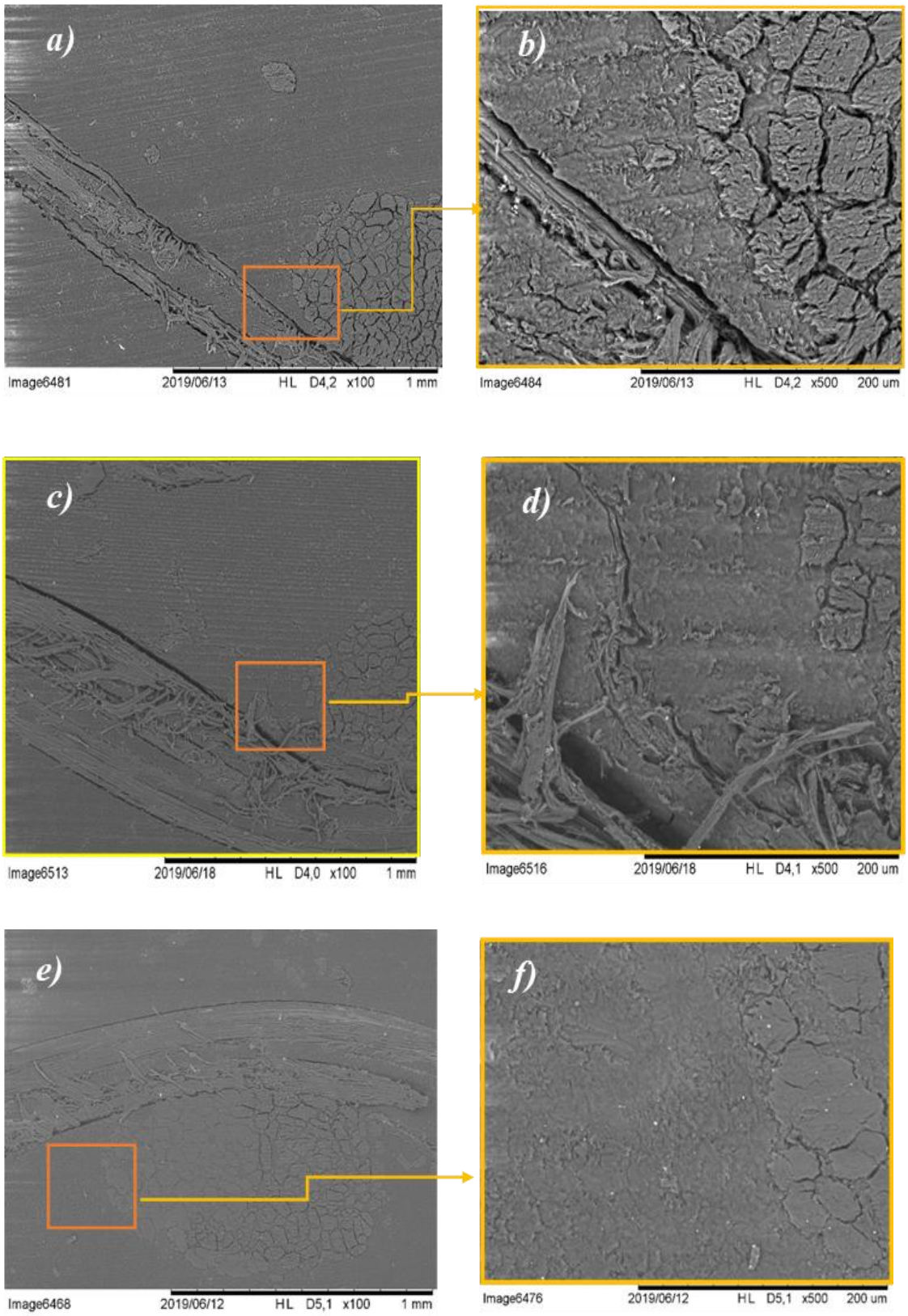


Fig III.27 SEM micrographs on the cross-section of different intra-layer fabrics reinforced polyester composite: A_{un} /J (a, b), A_N /J (c, d), and A_P /J (e, f).

III.16.5 Statistical analysis

The interfacial shear strength variability of various Alfa fibers (A_{Un}, A_N, A_p) with a polyester matrix, as well as the tensile strengths of the material composites, were statistically analyzed using the two-parameter Weibull distribution method ,according to previous studies [436]. This analysis compared Weibull distributions for two cases: a) the interfacial shear strength of treated and untreated Alfa fibers to evaluate the impact of chemical treatments on fiber surface quality, and b) the tensile strengths of composite materials reinforced with treated and untreated intra-ply hybrid fabrics to assess whether combining treated and untreated Alfa fibers with jute fibers gives composite materials with improved mechanical properties.

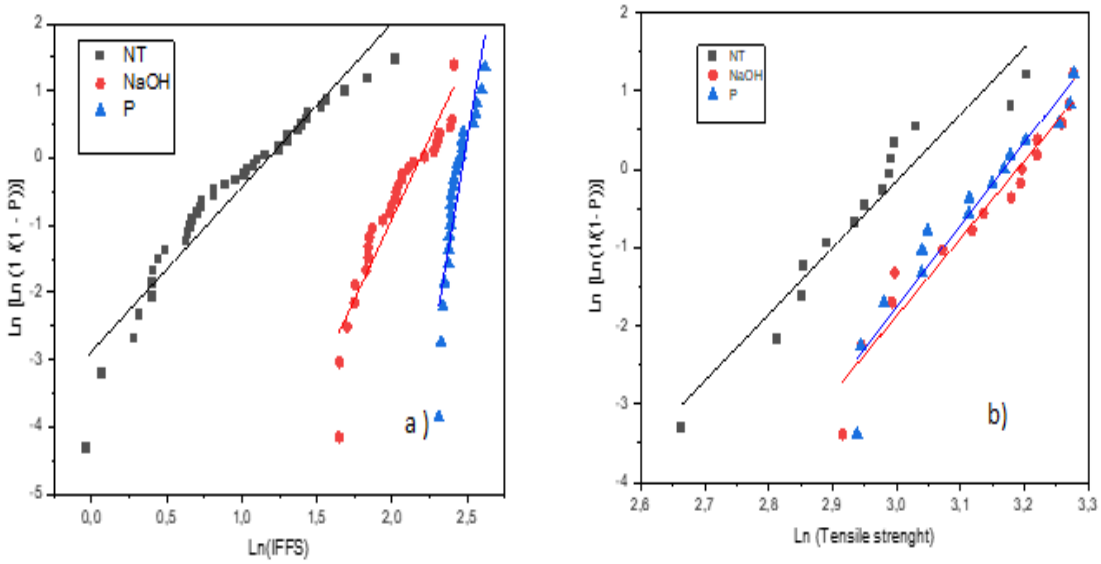


Fig III.28 Weibull probability plots for: a) Interfacial shear strength of various Alfa fibers, b) Tensile strength of the material composites.

The experimental data plots (Figure III.28a) show that shear strength of treated fibers aligns more closely with the Weibull distribution. Permanganate-treated Alfa fibers exhibit a higher Weibull modulus (m) of 6.28, followed by alkali-treated fibers at 4.77, compared to 2.43 for untreated fibers (TableIII.14). A higher m value indicates a less variability in shear strengths, reflecting fewer defects and improvements of the fibers surface quality, due to permanganate and alkali treatments. While the low m value indicates a high scattering in interfacial shear strength,

this may be attributed to the fiber heterogeneity and the presence of surface impurities and waxes[436]. Weibull probability plots reveal a significant correlation between the effects of surface chemical treatment and the interface shear strength of Alfa fiber/resin.

The Weibull probability plots for tensile strength of composites reinforced with treated Alfa and jute fibers using intra-ply hybrid fabrics (Comp-B, Comp-C) show a better fit with the experimental data (Figure III.28b). Comp-C and Comp-B exhibit higher Weibull modulus of 10.90 and 10.16, respectively, compared to 9.07 for untreated fibers (TableIII.15). A higher m value indicates less variability in tensile strengths of material composites, this may be due to improved mechanical interlocking due to intra-ply hybridization alongside the chemical treatment of the fibers [248].

Table III.14. Experimental and Analytical results of interfacial shear strength of various Alfa fibers (IFSS).

Fibers	Experimental IFSS (MPa)	Analytical results	
		Weibull modulus m_0	Scale parameter $IFSS_0$ (MPa)
Alfa (A_{Un})	2.89	2.43	3.26
Alkali treated Alfa (A_N)	7.79	4.77	8.96
Permanganate treated Alfa (A_P)	11.47	6.28	11.50

Table III.15. Experimental and Analytical results of tensile strength of composites.

Composites	Experimental Strength σ (MPa)	Analytical results	
		Weibull modulus m_0	Scale parameter σ_0 (MPa)
Comp-A	19.86	9.07	20.32
Comp-B	23.47	10.16	24.26
Comp-C	23.08	10.90	23.74

III.17 Mechanical and physical Characterization of unidirectional composite

III.17.1 Density and void content

The void content determined for the unidirectional composite reinforced with Alfa fibers (Un-Comp) was found to be 5.51 % (Table III. 16). According to the criteria established by Sharma, et al. [418], a void fraction exceeding 5% indicates moderate to poor composite quality, mainly resulting from incomplete fiber impregnation and the possible entrapment of air during the fabrication process. The relatively high void content observed in this composite suggests insufficient wetting of the Alfa fiber surface by the unsaturated polyester matrix, which can be attributed to the hydrophilic nature of untreated natural fibers. In addition, the presence of surface impurities such as waxes, lignin, and pectic substances covering the Alfa fibers further limits their interfacial compatibility with the hydrophobic polymer matrix, thereby reducing resin penetration and promoting void formation during composite processing.

Table III. 16. Densities and void content of unidirectional composite.

Composite	Density ($g \cdot cm^{-3}$)		Void (%)
	Experimental	Theoretical	
Un-Comp	1.182 ± 0.0017	1.251± 0.0013	5.51± 0.110

III.17.2 Tensile properties

This section presents the results of the static tensile test conducted on a unidirectional composite consisting of an unsaturated polyester matrix reinforced with unidirectional Alfa plies. As illustrated in Figure III.29, the stress-strain curve exhibits an almost linear evolution up to the point of fracture, reflecting a brittle elastic behavior characteristic of Un-Comp with limited plastic deformation capacity.

The curve shows a linear increase in stress within a strain range of 0.019 % to 0.303 %, corresponding to the purely elastic region, where the applied load is efficiently transferred from the matrix to the well-aligned Alfa fibers. This region defines the elastic modulus (Young's modulus) of this composite. The variation of elastic modulus depends strongly on fiber orientation and the degree of interfacial adhesion. Beyond this stage, the composite continues to carry the load progressively until reaching the maximum tensile strength, after which a sharp and sudden drop in stress occurs.

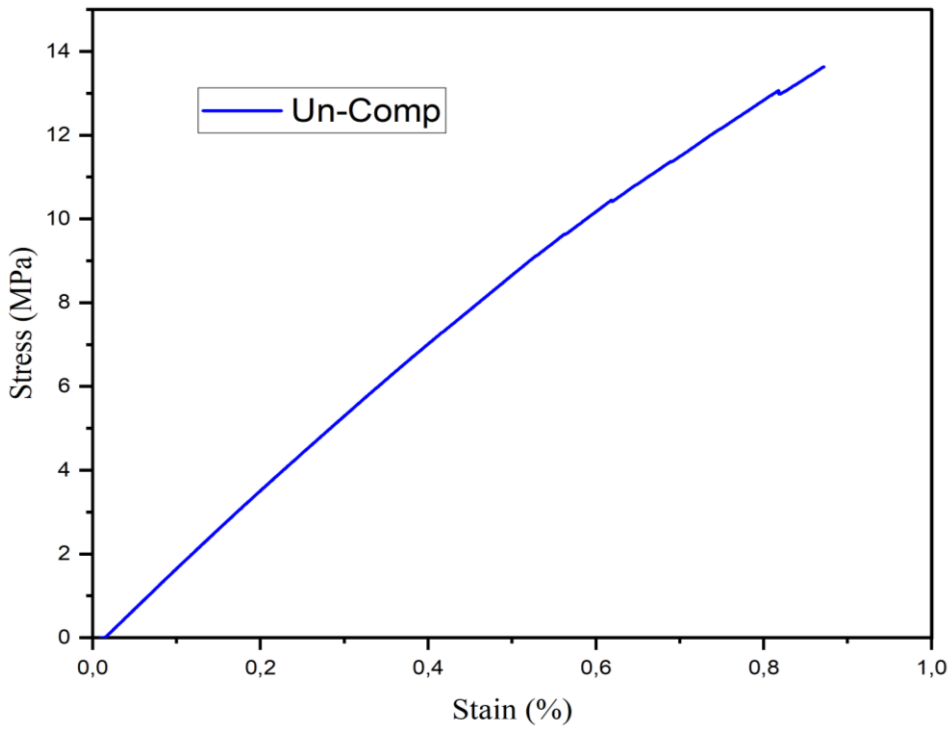


Fig III.29 Typical Stress–strain curves of Un-Comp.

A slight nonlinearity can also be observed prior to fracture, possibly due to micro-debonding at the fiber-matrix interface and the initiation of micro-cracks within the matrix. These early damages act as stress concentrators, weakening the interfacial bonding and leading to an abrupt failure once the applied load exceeds the interfacial strength. This behavior indicates the onset of fiber-matrix debonding, immediately followed by the complete brittle failure of the unidirectional composite.

Unidirectional composite exhibited a maximum tensile strength of σ =13.633 ± 1.07 MPa, a fracture strain of ε = 0.871 ± 0.005 %, and a Young's modulus of E = 1501.03 ± 101.10 MPa, confirming the material's linear elastic behavior and limited ductility. This behavior is attributed to the high stiffness of Alfa fibers combined with the inherent brittleness of the unsaturated polyester matrix.

Overall, the stress-strain response demonstrates that the unidirectional architecture enhances the load-bearing capacity along the fiber direction, while the brittle failure mode reflects the anisotropic mechanical response of the composite. The good alignment of the Alfa fibers promotes efficient stress transfer during the elastic phase, yet once interfacial adhesion limits are exceeded, failure occurs abruptly, typical of fiber-reinforced composites under tensile loading. Tensile

properties of unidirectional Alfa ply composite are summarized in Table III. 17, and compared with studies concerning unidirectional composite materials.

Table III. 17. Comparison of tensile properties of unidirectional Alfa ply composites with some previous studies.

Unidirectional composites	Tensile strength (MPa)	Tensile modulus (GPa)	Elongation at break (%)	Weight fraction (%)	References
Unidirectional Alfa / UP	13.63± 1.07	1.50 ± 0.10	0.87 ± 0.50	16.75	Current work
kenaf / PP	70	6.5	~1.9	40	[215]
Lin / PLA	339 ± 23	19.60 ± 1.3	-	49.3 ± 0.2	
Lin / PP	320.70 ± 18	22.50 ± 1.5	-	-	[437]
Lin /Epoxy	328 ± 2.38	1.48 ± 0.05	-	-	

UP: unsatured Polyester, PP: polypropylene, PLA: polylactic acid.

III.17.3 Flexural properties

This section reports the results of the static three-point bending test performed on unidirectional composites reinforced with Alfa fibers and an unsaturated polyester matrix. As demonstrated in Figure III.30, the flexural stress-strain curve displays linear behavior within the elastic region, followed by a sudden failure, which is characteristic of brittle natural fiber-reinforced composites. The elastic region extends from approximately 0.56 % to 1.22 % strain, during which the applied bending load is effectively transferred from the matrix to the aligned Alfa fibers. Within this range, the composite deforms elastically, and the slope of the curve defines the flexural modulus, reflecting the stiffness of the fiber-matrix system under bending. The linearity indicates efficient stress transfer and a strong interfacial adhesion in this initial phase.

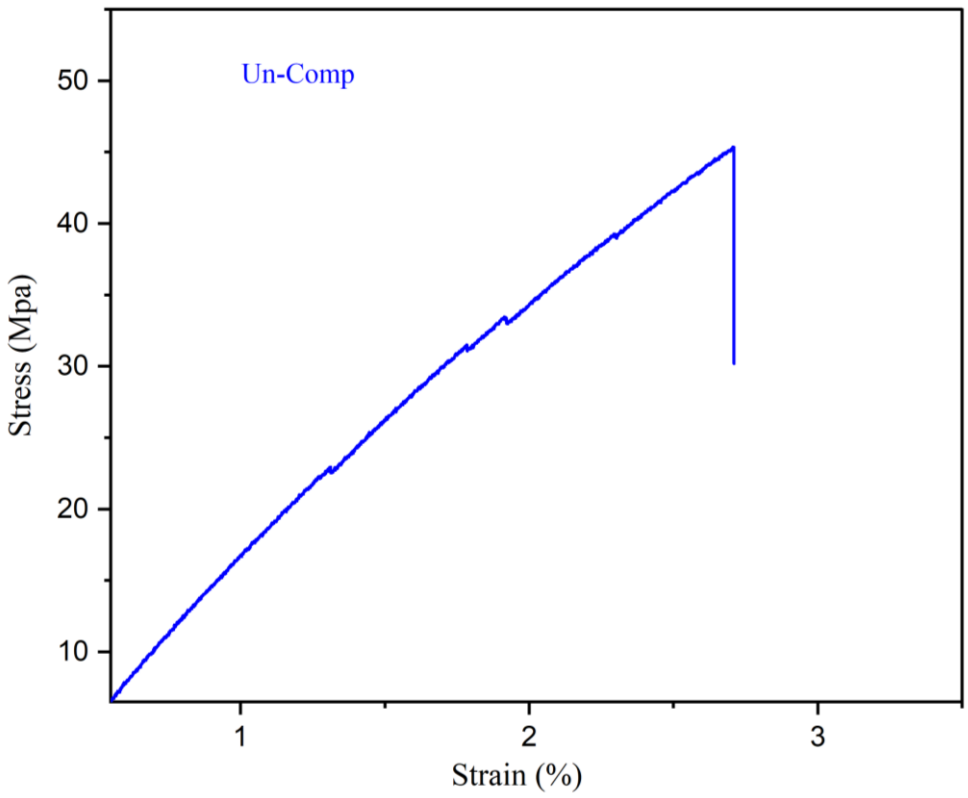


Fig. III.30. Evolution of stress as a function of deflection for Un-Comp.

Beyond the elastic region, the curve continues to rise gradually until reaching the maximum flexural stress of $\sigma = 45.37 \pm 4.73$ MPa at a fracture strain of $\varepsilon = 2.70 \pm 0.008$ %. The corresponding flexural modulus was measured as E = 1400.77 ± 121.12 MPa. A minor deviation from linearity is observed before fracture, likely due to the onset of micro-cracks within the matrix and partial fiber-matrix debonding, which serve as stress concentrators and accelerate failure. The sudden fall in stress when the peak is reached is indicative of the brittle nature of the composite under bending. Here, the load-bearing capacity is dominated by the fiber reinforcement along the bending direction. The high stiffness of Alfa fibers ensures that most of the applied bending stress is carried by the fibers, while the unsaturated polyester matrix contributes limited plasticity. This results in sudden fracture once interfacial or matrix limits are exceeded. It is evident that the unidirectional arrangement enhances bending performance along the fiber orientation, although the failure mode remains brittle. These results highlight the anisotropic mechanical response of the composite and underscore the importance of fiber alignment and interfacial adhesion in maximizing flexural performance. Flexural properties are summarized in Table III.18 and compared with literature data for natural fiber-reinforced unidirectional composite materials.

Table III. 18. Comparison of flexural properties of unidirectional Alfa ply composites with some previous studies.

Unidirectional composites	Flexural strength (MPa)	Flexural modulus (GPa)	Flexural strain at break (%)	Weight fraction (%)	References
Unidirectional Alfa / UP	45.37 ± 4.73	1.40 ± 0.12	2.70 ± 0.88	16.75	Current work
kenaf / PP	110	8.5	-	40	[215]
Lin / PLA	363	26	-	49.3 ±0.2	[437]

UP: unsatured Polyester, PP: polypropylene, PLA: polylactic acid.

III.18 Comparison of mechanical properties of unidirectional and bidirectional composites

The comparative table of mechanical properties of unidirectional and bidirectional composites Table III. 19. highlights that the fiber orientation within the polymer matrix plays a crucial role in determining the mechanical properties of Alfa fiber-reinforced composites. The unidirectional composite (Unidirectional Alfa/UP) exhibited relatively lower tensile strength (13.63 ± 1.07 MPa) and Young's modulus (1.50 ± 0.10 GPa), which can be attributed to the limited load distribution along a single direction and its high sensitivity to interfacial defects. In contrast, the bidirectional composite (Bidirectional Alfa/Jute/UP) showed a significant improvement in tensile strength (19.86 ± 2.79 MPa) and Young's modulus (1.54 ± 0.23 GPa) due to a more balanced stress distribution along two perpendicular directions, reducing stress concentration and delaying the onset of failure.

Table III. 19. Comparison of the mechanical properties of unidirectional and bidirectional composites.

composites	Tensile strength (MPa)	Tensile modulus (GPa)	Flexural strength (MPa)	Flexural modulus (GPa)	References
Unidirectional Alfa / UP	13.63± 1.07	1.50 ± 0.10	45.37 ± 4.73	1.40 ± 0.12	Current work
Bidirectional (A_{unt} / J)/ UP	19.86 ± 2.79	1.54 ± 0.23	46.30 ± 8	1.72 ± 0.08	

Regarding flexural properties, the bidirectional composite also recorded a higher flexural modulus (1.72 ± 0.08 GPa) compared to the unidirectional composite (1.40 ± 0.12 GPa), with similar flexural strength values, indicating the effectiveness of the bidirectional structure in resisting bending deformation. This behavior underscores the critical role of fiber interlacing and textile architecture in enhancing load transfer and overall composite stiffness. Accordingly, these

results confirm that adopting bidirectional architectures is more suitable for applications requiring balanced mechanical properties and higher structural stability compared to unidirectional composites.

III.19 Scanning electron microscopy of unidirectional composite

Figure III.31 presents a cross-sectional view of the fracture surface of unsaturated polyester composite reinforced with unidirectional Alfa fiber plies (Un-Comp). The image clearly reveals weak interfacial adhesion between the fibers and the matrix. This is evidenced by distinct separation zones at the fiber-matrix interface, delamination and partial debonding from the polymer matrix during loading. This pattern of separation is indicative of a weak interfacial bond, a behavior that is commonly observed in untreated natural fibers.

The micrograph also clearly shows the presence of non-cellulosic components, such as waxes, lignin and pectic substances, which are visible on the surface of the Alfa fibers. These elements are present in the form of irregular deposits and heterogeneous accumulations. Collectively, these elements form an insulating layer that hinders the development of effective interfacial bonding with the polymer matrix. This results in reduced mechanical adhesion and inefficient stress transfer across the interface. As a result, fracture in this type of composite generally initiates at the fiber-matrix interface due to the limited adhesion, before propagating into the matrix or fibers depending on the applied stress level.

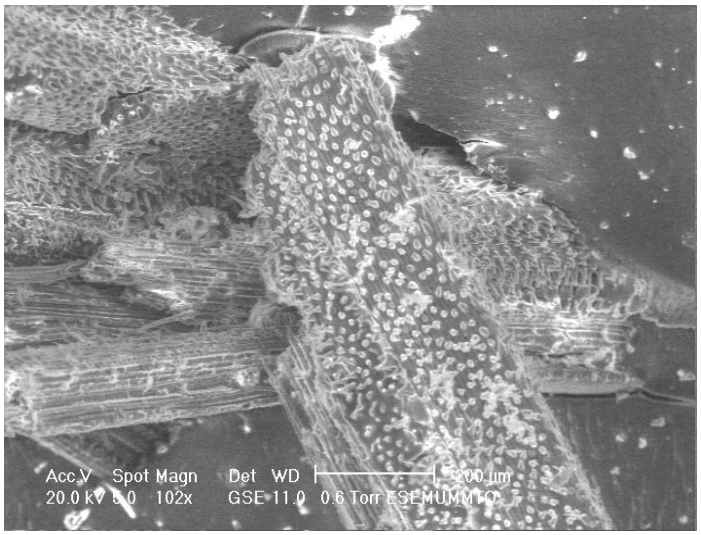


Fig. III.31. SEM micrographs on the surface fracture of unidirectional reinforced polyester composite.

These observations confirm that poor interfacial adhesion between the Alfa fibers and the polyester matrix is a key factor in the brittle mechanical behavior observed in the tensile response of the unidirectional composite. At the same time, they emphasize the significance of surface treatments, whether chemical or physical (such as alkaline or permanganate treatment), in order to enhance surface roughness and promote stronger fiber-matrix interfacial bonding in Alfa / UP composites.

III.20 Conclusions

The findings of this study confirm the crucial role of chemical surface treatments and intra-ply hybridization strategies in significantly enhancing the performance of Stipa tenacissima L. (Alfa) fibers and their reinforced composites. The observed improvements stem from the combined effects of chemical purification, structural reorganization, and improved fiber–matrix interactions, which collectively contribute to superior mechanical behavior and structural stability of the resulting materials.

Indeed, the alkali and permanganate treatments proved highly effective in removing surface impurities, waxes, and amorphous constituents such as hemicellulose and lignin, as verified by ATR-FTIR spectroscopy. This purification process led to an increase in fiber crystallinity, density, and stiffness, while also promoting a rougher surface topography conducive to stronger interfacial bonding with the polymer matrix. Consequently, the treated Alfa fibers exhibited improved tensile performance and enhanced compatibility with the polyester resin, as demonstrated by both interfacial shear strength (IFSS) tests and the statistical reliability confirmed through Weibull analysis.

Moreover, the integration of Alfa fibers with jute fibers in intra-ply hybrid woven fabrics resulted in a synergistic reinforcement effect, combining the flexibility and ductility of jute with the stiffness and light weight of Alfa fibers. This hybrid configuration produced composite materials with enhanced mechanical performance, achieving strength and stiffness values comparable to those of some commercially available synthetic fiber-reinforced composites. Morphological observations via SEM supported these findings, revealing improved resin impregnation, reduced interfacial voids, and enhanced wetting properties in the treated hybrid composites. It was also confirmed that unidirectional Alfa fiber-reinforced composites exhibit lower mechanical and physical properties compared to their bidirectional counterparts.

Overall, the results underscore the considerable potential of Alfa fibers as viable, sustainable reinforcements for polymer composites. Through appropriate surface treatments and hybridization approaches, these natural fibers can achieve mechanical and structural performances that meet the requirements of industrial applications, particularly in automotive, construction, and packaging sectors. This work therefore contributes not only to the valorization of an abundant local resource but also to the advancement of eco-efficient materials, supporting the global transition toward sustainable and circular material technologies

General conclusion

General conclusion

The comprehensive set of analyses carried out on Stipa tenacissima L. Fibers collected from five growth regions across seasons demonstrated that environmental and geographical conditions play a decisive role in determining the chemical, thermal, physical and mechanical properties of Alfa fibers. The main results can be summarized as follows:

- Spring fibers exhibited the highest cellulose content (≈ 49-51%), then fall (≈ 41-43%) whereas winter fibers showed the lowest values (≈ 39-40%).
- Hemicellulose content reached its maximum in winter (≈ 22-23%) compared to spring (≈19-21%), reflecting reduced physiological activity during colder conditions.
- Lignin content showed a clear increase in summer (≈ 26-27%), consistent with thermal stress and enhanced lignification.
- The highest extractives content was recorded in winter, ranging between 9-11% across all study regions, reflecting the slowdown of physiological activity and the accumulation of secondary metabolites.
- Ash content is high in winter and summer, especially in Djelfa (≈3%). Elevated ash levels reflect a greater presence of inorganic residues, which can hinder fiber-matrix adhesion and consequently reduce the mechanical performance of the composites.
- Best thermal properties of Alfa fibers were recorded in spring across most regions, with AL and AT samples showing the highest thermal stability. In addition, AT samples exhibited the maximum T_{max} and E_0.
- XRD results confirmed that spring fibers also achieved the highest crystallinity index (61-65 %), while winter fibers exhibited the lowest values (49-50 %).
- A strong positive correlation was observed between cellulose content and crystallinity, both of which directly influence fiber tensile performance.
- Mechanically, spring fibers recorded the best overall performance across all regions:

  Tensile strength of: 307- 417 MPa, Young's modulus: 22- 25 GPa, Elongation at break: 1.6-1.8 %.

- In contrast, winter fibers exhibited a 39-46% reduction in tensile properties, in line with their lower crystallinity and cellulose content. These results clearly establish a causal link between chemical composition, particularly cellulose and crystallinity and the mechanical behavior of Alfa fibers. SEM micrographs reinforced these findings.

- Spring samples displayed clean, smooth surfaces with clearly visible elementary fibers, indicative of high structural quality and well-consolidated cell walls. However, winter samples showed rough surfaces with deposits and impurities, reflecting reduced biological activity.

In continuation of these findings, the second part of the study demonstrated the pivotal role of chemical treatments and intra-ply hybridization strategies in enhancing the performance of Stipa tenacissima L. (Alfa) fibers and their composites. The main out Comes can be summarized as follows:

- Chemical treatments have a significant impact on the density of alfa fibers, as evidenced by the improved density values obtained post-treatment, thereby enhancing their strength and durability.
- ATR-FTIR analysis confirms the removal of wax, impurities, and non-cellulosic elements (hemicellulose and lignin) following alkali and permanganate treatments.
- As expected, the effectiveness of the chemical treatment on the Alfa fiber results in the improvement of the mechanical performance of this fiber as well as the enhancement of its crystalline structure.
- Chemical treatments have a considerable impact on the fiber/matrix interface, as evidenced by (i) improved interfacial compatibility of treated Alfa fiber-reinforced composite, (ii) better resin penetration into the fibers, and (iii) wettability of fibers. The enhanced interface led to an improvement in the mechanical properties of the composite materials.
- The combination of treated and untreated Alfa fibers with jute fibers through intra-ply hybrid fabrics gives composite materials with improved mechanical properties, which can approximate the mechanical properties of commercially available fabric-reinforced materials composite.
- Weibull probability plots for interfacial shear strength of treated Alfa fibers-resin and the tensile strength of material composites reinforced with treated Alfa and jute fibers using intra-ply hybrid fabrics showing a better fit with the experimental data.
- Unidirectional Alfa fiber–reinforced composites demonstrate inferior mechanical and physical properties compared to their bidirectional counterparts.

In summary, this study demonstrates that the performance of Stipa tenacissima L. fibers is strongly linked to their microstructural organization and to the environmental and seasonal factors that influence the distribution of cell-wall components, the degree of crystallinity, and structural density, all of which determine their thermal resistance and mechanical behavior. Accordingly,

improving growth conditions, selecting suitable harvesting sites such as Laghouat and Tiaret, and prioritizing spring harvesting represent key strategies for enhancing fiber quality and tailoring their properties for advanced industrial applications. Additionally, chemical treatment and hybridization of Alfa fibers offer a promising pathway to developing high-performance bio-composites, bridging the gap between natural and synthetic reinforcement systems while minimizing environmental impact.

Perspectives

Based on the findings obtained in this work, several promising research directions can be identified to further enhance the understanding of the structural and functional properties of Stipa tenacissima L. fibers and to expand their potential industrial applications. The first direction involves the development of multiscale numerical models that establish a link between the microstructural characteristics of the fibers such as crystallinity, lumen size, and fibrillar orientation and the mechanical and thermal behavior of the resulting composites. Finite Element Modeling (FEM) could play a crucial role in predicting composite performance as a function of harvesting season, growth location, or fiber surface treatment.

Another promising avenue involves the future production of continuous Alfa fiber yarns, enabling their integration into various textile architectures and thus opening opportunities in technical textiles and bio-engineered materials. In parallel, the fabrication of real composite prototypes and their evaluation under actual service conditions represents an essential step toward strengthening the reliability and transferability of laboratory-scale findings.

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