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Etude du comportement en rupture des composites

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Dedication

*To the memory of **my beloved father**, who left too soon but lives forever in my heart. Every line of this work bears the mark of his love, and every achievement echoes his silent sacrifices. May Allah open for him the gates of His vast paradise.*

*To **my mother**, a woman with a heart of gold, whose quiet but immense strength has watched over me, prayed for me, and encouraged me... This work is the fruit of her prayers, her tears, and her infinite love.*

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Belkacem Mokhtar

Abstract

This thesis falls within the field of materials mechanics and focuses on the fracture behaviour of polymer matrix composites reinforced with natural fibers. Two research directions are explored. The first involves studying the effect of natural ageing on the flexural mechanical properties of an Alfa/polyester composite. This composite is developed using two types of Alfa fibers (coarse and fine) at three different weight fractions (10%, 20%, and 30%). The second axis investigates the effect of artificial ageing through ultraviolet (UV) radiation on a flax/epoxy-based composite and its hybrid versions. The adopted methodology is based on three-point bending tests and optical microscopy observations for the analysis of fracture mechanisms. Additionally, a numerical simulation using the finite element method in Abaqus is implemented to validate the experimental results and deepen the analysis of mechanical behaviour. The results highlight the significant impact of ageing, whether natural or UV-induced, on the mechanical performance of biocomposites. These degradations directly affect flexural strength and fracture mechanisms, underlining the need to consider such phenomena for the sustainable development of natural fiber-based composite materials.

Keywords : Alfa/polyester, Flax/epoxy, Natural ageing, UV ageing, Three-point bending, Finite element method.

المخلص

تندرج هذه الأطروحة ضمن ميكانيك المواد وتركّز على سلوك الكسر للمواد المركّبة ذات المصفوفة البوليمرية المدعّمة بالألياف الطبيعية.

تمّ استكشاف محورين بحثيين:

المحور الأول يتناول دراسة تأثير التقادم الطبيعي على الخواص الميكانيكية الانحنائية لمركّب حلفاء/بوليستر. وقد جرى تطوير هذا المركّب باستخدام نوعين من ألياف الحلفاء (خشنة وناعمة) بثلاثة كسور وزنية مختلفة (10%، 20%، و30%).

أما المحور الثاني فيبحث تأثير التقادم الاصطناعي بفعل الأشعّة فوق البنفسجية على مركّب قائم على الكتّان/إيبوكسي ونسخه الهجينة.

تعتمد المنهجية المعتمدة على اختبارات الانحناء ثلاثي النقاط ومشاهدات المجهر البصري لتحليل آليات الكسر. بالإضافة إلى ذلك، تم تنفيذ محاكاة عددية باستخدام طريقة العناصر المحدّدة في برنامج اباكوس بهدف التحقق من صحة النتائج التجريبية وتعميق تحليل السلوك الميكانيكي.

تُبرز النتائج الأثر الكبير للتقادم سواء الطبيعي أو الناتج عن الأشعّة فوق البنفسجية على الأداء الميكانيكي للمواد الحيوية المركّبة، إذ تؤثر هذه التدهورات مباشرةً في مقاومة الانحناء وآليات الكسر، مما يؤكد ضرورة أخذ هذه الظواهر في الحسبان عند السعي إلى تطوير مستدام للمواد المركّبة المعتمدة على الألياف الطبيعية.

الكلمات المفتاحية: حلفاء/بوليستر، كتّان/إيبوكسي، التقادم الطبيعي، التقادم بالأشعّة فوق البنفسجية، الانحناء ثلاثي النقاط، طريقة العناصر المحدّدة، آليات الكسر.

Résumé

Cette thèse s'inscrit dans le domaine de la mécanique des matériaux et porte sur l'étude du comportement en rupture de composites à matrice polymère renforcée par des fibres naturelles. Deux axes de recherche sont abordés. Le premier concerne l'étude de l'effet du vieillissement naturel sur les propriétés mécaniques en flexion d'un composite Alfa/polyester. Ce composite est élaboré à partir de deux types de fibres d'Alfa (grosses et fines) à trois fractions massiques différentes (10 %, 20 %, et 30 %). Le second axe s'intéresse à l'effet du vieillissement artificiel par rayonnement ultraviolet (UV) sur un composite à base de lin/époxy et ses versions hybrides. La méthodologie adoptée repose sur des essais de flexion trois points ainsi que des observations au microscope optique pour l'analyse des mécanismes de rupture. Par ailleurs, une modélisation numérique par la méthode des éléments finis sous Abaqus est mise en œuvre afin de valider les résultats expérimentaux et approfondir l'analyse du comportement mécanique. Les résultats obtenus mettent en évidence l'impact significatif du vieillissement, qu'il soit naturel ou induit par UV, sur les performances mécaniques des biocomposites. Ces dégradations influencent directement la résistance en flexion et les mécanismes de rupture, soulignant la nécessité de prendre en compte ces phénomènes pour le développement durable des matériaux composites à base de fibres naturelles.

Mots clés : Alfa/polyester, Lin/époxy, Vieillissement naturel, Vieillissement UV, Flexion trois points, Méthode des éléments finis.

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- Etude comparative des propriétés mécaniques des aciers inoxydables AISI304 et AISI430 vis à vis la sensibilité à la fragilisation par l'hydrogène (FPH). Décembre 2023

List of tables

Table N°	Title	Page N°
01	Table I.1 : List of largest producer countries of plant fiber.....	15
02	Table I.2 : The chemical composition of plant fiber.....	16
03	Table I.3 : Some main characteristics of thermoplastic matrix.....	18
04	Table I.4 : Some main characteristics of thermoplastics matrix materials.....	18
05	Table III.1 : Mechanical properties of polyester/alfa composite reinforced with fine fibers.....	71
06	Table III.2: Mechanical properties of polyester/alfa composite strengthened with coarse fibers.....	72
07	Table IV.1 : Summary of the different configurations manufactured and tested.....	88
08	Table IV.2 : Flexural Performance of Unaged and UV-Aged Flax-Based Epoxy Composites : A Comparative Analysis.....	99

List of figures

Figure N°	Title	Page N°
01	Figure I.1 : Different types of Composite Material.....	06
02	Figure I.2 : Types of natural fiber.....	07
03	Figure I.3 : Global distribution of alfa grass shown in green color...	09
04	Figure I.4 : Alfa plant and its traditional use (A) alfa plant growing and (B) example of traditional handicraft items made from Alfa.....	09
05	Figure I.5 : Categories of the potential application of AF reinforced composite in packaging.....	12
06	Figure I.6 : Development of bio nanocomposite packaging films based on AF-CNC.....	13
07	Figure I.7 : Poly (vinyl alcohol) nanocomposite films based on AF- CNF with enhanced mechanical properties.....	13
08	Figure I.8: Unidirectional alfa fibres used as fillers in composite materials, with potential applications in the automotive and aerospace industries	14
09	Figure I.9 : Diagram of natural plant cell walls	16
10	Figure I.10 : Application of natural fiber reinforced polymer composite.....	24
11	Figure I.11 : Various categories of natural FRP hybrid composites..	26
12	Figure I.12: Fibre arrangements within natural fibre-reinforced polymer (FRP) hybrid composites, illustrating: (a) interply, (b) intraply, and (c) intrayarn configurations.....	27
13	Figure I.13 : Amount of fibre spreading in (a) two layers, (b) alternating layers, (c) bundle-bundle dispersion and (d) irregular	

	dispersion.....	28
14	Figure I.14 : (a) Tri-wheel auto-wheel hub, (b) ceiling fan blade and (c) wheeler side mirror casing made from twisted natural FRP hybrid composites.....	29
15	Figure I.15 : Evaluation of tensile characteristics of selected natural/synthetic FRP hybrid composite. Data for the plot are from various publications cited in this article.....	31
16	Figure I.16 : Evaluation of flexural responses of selected natural/synthetic FRP hybrid composites.....	33
17	Figure II.1: Constituents of the matrix (resin and hardener).....	40
18	Figure II.2: Alfa (Stipa tenacissima) in natural conditions.....	40
19	Figure II.3: oven for drying Alfa fibers.....	41
20	Figure II.4: blade crusher.....	42
21	Figure II.5: Sieve.....	43
22	Figure II.6: Sodium Hydroxide.....	43
23	Figure II.7: OPTIKA optical microscope, model: HDMI Easy – 4083. 13E.....	45
24	Figure II.8: Alfa fibers: (a) Crushed Alfa fibers, (b) Fine fibers, (c) Coarse fibers.....	46
25	Figure II.9: Workshop Workstation: CNC Milling Machine for Industrial Manufacturing.....	46
26	Figure II.10: Mold for three-point bending specimen.....	47
27	Figure II.11: Mold for toughness specimen.....	47
28	Figure II.12: polyester/alfa composite specimens for bending test...	48
29	Figure II.13: different configurations of the Alfa polyester	

	composite for the toughness test.....	48
30	Figure II.14 : cutting of rectangular and round profiles.....	50
31	Figure II.15 : Manufacturing of Test Supports : Milling Operation with Digital Control.....	50
32	Figure II.16: Bend testing machine.....	51
33	Figure II.17: Sequence of the experimental 3 points bending test until failure: (a) Initial state, (b) Final state.....	52
34	Figure II.18: Single edge notched bend (SENB) specimen.....	54
35	Figure II.19: Composite specimens SENB during three-point bending test.....	54
36	Figure II .20: Stress-strain polyester/Alfa composite with different fractions of reinforcement (0, 10, 20, 30 wt%) for both sizes of Alfa fibers (fine and coarse).....	50
37	Figure II. 21: Effect of three months atmospheric ageing on the mechanical behaviour of the different categories of the polyester/Alfa composite.....	57
38	Figure II. 22: Effect of three months atmospheric ageing on the bending strength of the polyester/Alfa composite with different fractions of reinforcement (0, 10, 20, 30 wt %) for both sizes of Alfa fibers (fine and coarse).....	58
39	Figure II. 23: Young's modulus variation of different Polyester/Alfa composites (Coarse and fine size).....	59
40	Figure II.24: Microvoids observed experimentally in the polyester/Alfa composite.....	60

41	Figure II.25: Load-displacement unaged polyester/Alfa composites curve with different fractions of reinforcement.....	61
42	Figure II.26: Load-displacement aged polyester/Alfa composites curve with different fractions of reinforcement (0, 10, 20, 30 wt %) for both sizes of Alfa fibers (fine and coarse).	62
43	Figure II.27: Crack propagation under mode I fracture.....	63
44	Figure II.28: Variation of fracture toughness (K_{IC}) of different Polyester/Alfa composites (Coarse and fine size).....	63
45	Figure III.1: Loading and boundary conditions applied to the model.....	68
46	Figure III.2: The finite element mesh model (sample, punch, and supports).....	69
47	Figure III.3 : Contour integration path around crack tip.....	73
48	Figure III.4: Loading and boundary conditions applied to the model.....	74
49	Figure III.5: 3D model with the direction of the fracture.....	75
50	Figure III.6: The finite element mesh model: the SENB specimen, the mesh around the crack emanating from the notch, and the mesh around the crack tip (from right to left).....	76
51	Figure III.7: Central area failure: experimental and numerical.....	77
52	Figure III.8: The experimental and numerical stress-strain curves of unaged Alfa/polyester composite with different fractions of	

	reinforcement (0, 10, 20, 30 wt%) for both sizes of alfa fibers: (a)	
	fine fibers and (b) coarse fibers.....	77
53	Figure III.9: The experimental and numerical stress-strain curves of aged Alfa/polyester composite with different fractions of reinforcement (0, 10, 20, 30 wt %) for both sizes of alfa fibers: (a) fine and (b) coarse.....	78
54	Figure III.10: von Mises stress distributions: Young polyester/Alfa composite with different fractions of reinforcement (0, 10, 20, 30 wt%) for both sizes of Alfa fibers (fine and coarse).....	79
55	Figure III.11: von Mises stress distributions: Aged polyester/Alfa composite with different fractions of reinforcement (0, 10, 20, 30 wt%) for both sizes of Alfa fibers (fine and coarse).....	79
56	Figure III.12: J-integral versus crack length for young composite alfa polyester with fine (a) and coarse fibers (b).....	80
57	Figure III.13: A three-dimensional representation of the fractured composite.....	81
58	Figure III.14: J-integral versus crack length for aged composite alfa polyester with fine (a) and coarse fibers (b).....	81
59	Figure IV.1 : Carbon Fiber Spools.....	85
60	Figure IV.2 : Unidirectional Glass Fiber Fabric.....	85
61	Figure IV.3 : FlaxPly® Natural Flax Fiber Fabric.....	86
62	Figure IV.4 : Kevlar® 49 Fabric Roll.....	86
63	Figure IV.5 : Araldite® Epoxy Resin and Aradur® Hardener.....	87
64	Figure IV.6 : Hand lay-up process.....	88
65	Figure IV.7 : hybrids composites specimens for bending test.....	89

66	Figure IV.8 : UV-C fluorescent lamps (GE S875, 28W).....	89
67	Figure IV.9 : Bend testing machine.....	90
68	Figure IV.10. Experimental three-point bending test	90
69	Figure IV.11 : stress-strain curve of Flax epoxy hybride composite.	92
70	Figure IV.12 : stress-strain curve of Flax Kevlar epoxy hybride composite.....	93
71	Figure IV.13 : stress-strain curve of Flax Carbon epoxy hybride composite.....	94
72	Figure IV.14 : stress-strain curve of Flax Glass epoxy hybride composite.....	95
73	Figure IV.15 : Young's modulus variation of different hybrid composites.....	97
74	Figure IV.16 : Bending strenght variation of different hybrid composites.....	98
75	Figure IV.17 : Bending strain variation of different hybrid composites.....	99

Nomenclature

A

AF	Alfa fiber
AFPC	Alfa fiber polymer composites
R-AF	Raw Alfa Fibres
AT-AF	Alkali-Treated Alfa Fibres
AF-CMF	Alfa Fibre–Cellulose Microfibres
AF-CNC	Alfa Fibre–Cellulose Nanocrystals
AF-CNF	Alfa Fibre–Cellulose Nanofibres
ASTM	American Society for Testing and Materials

C

CNF	Cellulose nanofibres
CNC	Cellulose nanocrystals

D

DCB	Double Cantilever Beam
-----	------------------------

F

FVF	Fibre volume fraction
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N

NFRPCs	Natural fiber-reinforced polymer composites
NaOH	Sodium hydroxide

P

PTFE	polytetrafluoroethylene
PHBV	polyhydroxybutyrate-co-valerate
PP	polypropylene
PVC	polyvinylchloride
PW	Plain Weave

R

RTM	Resin Transfer Moulding
S	
SENB	Single-Edge Notch Bending
U	
UV	Ultraviolet

General introduction

General Introduction

In recent decades, the field of composite materials has witnessed remarkable scientific and industrial progress. Owing to their advantageous mechanical, thermal, and physicochemical properties, these materials have become essential in sectors such as aerospace, automotive, construction, and biomedical engineering. Their ability to combine lightness, strength, and durability makes them ideal for addressing growing demands for performance, energy efficiency, and environmental sustainability.

However, in response to mounting environmental concerns such as pollution, depletion of non-renewable resources, and waste management researchers and industries are increasingly shifting toward a new generation of materials: biocomposites. These are developed using a polymer matrix reinforced with natural fibres of plant or animal origin. Beyond being biodegradable or recyclable, biocomposites hold significant potential for the valorisation of local and renewable resources.

Among the available plant fibres, alfa (*Stipa tenacissima*) a Mediterranean plant abundant in Algeria stands out due to its attractive mechanical properties, low weight, local availability, and low cost. The use of alfa fibres as reinforcement in a polyester matrix opens up promising avenues for the development of environmentally friendly materials. However, the reinforcing efficiency of these fibres depends on several parameters, notably their length and content within the matrix.

These two factors directly influence fibre/matrix adhesion, stress distribution, and thus the overall mechanical performance of the composite. Fibres that are too short may fail to provide adequate load transfer, while excessively long or overly abundant fibres may cause agglomeration, poor dispersion, or reduced material homogeneity. Therefore, determining the optimal fibre length and content is crucial to achieving a balance between stiffness, strength, and toughness.

This leads to the central research question :

What is the effect of alfa fibre length and content on the mechanical performance of a polyester-based biocomposite, and how can these parameters be optimised to enhance the material's overall behaviour ?

One major challenge to the industrial application of these biocomposites lies in their durability, particularly under varying environmental conditions such as humidity, UV radiation, temperature fluctuations, and oxygen exposure. As a lignocellulosic material, alfa fibre is particularly sensitive to environmental ageing. Prolonged exposure to humidity can

cause fibre swelling and degradation of the fibre/matrix interface, while UV radiation may degrade the polymer chains in the matrix. These effects progressively diminish the material's mechanical properties, limiting its service life and potential applications.

This raises another key research question :

How do the mechanical and structural properties of a polyester/alfa fibre biocomposite evolve under accelerated atmospheric ageing, and what strategies can be employed to improve its resistance to these conditions ?

The general objective is to evaluate the durability of an epoxy/alfa fibre biocomposite under simulated atmospheric ageing, and to identify the parameters influencing its long-term performance.

In parallel with the study on alfa fibre-based biocomposites, another composite material was investigated, this time using flax fibres as reinforcement in an epoxy matrix. Flax fibres are widely utilised in natural fibre composites due to their excellent mechanical performance, low density, renewability, and compatibility with various polymers.

Nevertheless, flax-based biocomposites have certain limitations, particularly in terms of impact resistance, dimensional stability, and long-term durability in harsh environments. To address these issues, a widely adopted approach is hybridisation, which consists in combining flax fibres with synthetic reinforcements such as Kevlar, carbon, or glass fibres within the same epoxy matrix. This strategy aims to develop hybrid composites whose mechanical performance is enhanced through synergy between the various fibre types.

This raises another research question :

Which synthetic fibre Kevlar, carbon, or glass most effectively improves the mechanical behaviour of a flax fibre/epoxy resin composite ?

Composites, whether natural, synthetic, or hybrid, are increasingly employed in outdoor applications, where they are exposed to accelerated ageing factors such as humidity, temperature fluctuations, and UV radiation. Among these, UV radiation is one of the most critical factors contributing to the degradation of polymer matrix composites. It can significantly affect mechanical properties, interfacial adhesion, and the internal morphology of the materials.

This leads to a final research question :

What is the effect of artificial UV ageing on the mechanical performance of hybrid composites based on flax and synthetic fibres (Kevlar, carbon, glass), and how does this ageing process influence their durability and structural integrity?

This study, therefore, aims to investigate the effects of atmospheric and UV ageing on the mechanical behaviour of natural fibre-based composites, with a view to enhancing their performance and broadening their potential applications.

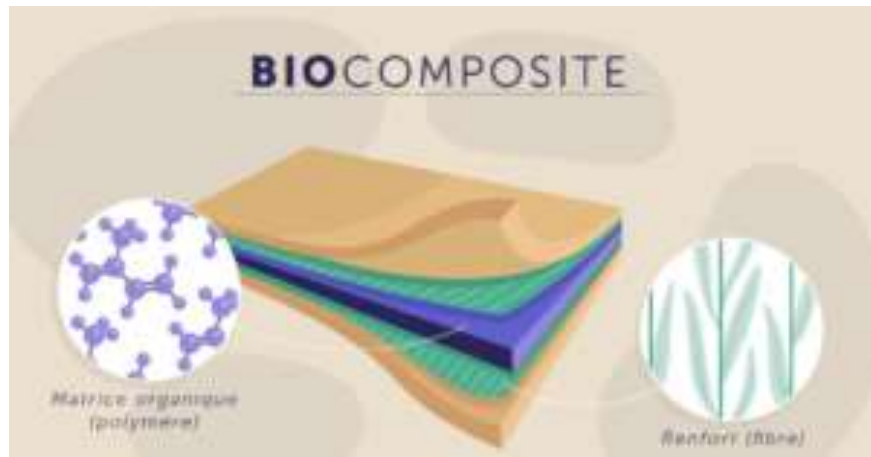
This thesis is structured into four chapters:

Chapter I presents a comprehensive review of biocomposites, focusing on natural fiber-reinforced polymer composites, especially those using alfa fiber. It covers types and treatments of natural fibers, manufacturing methods, and factors influencing mechanical performance. The chapter also explores hybrid composites, their advantages, and environmental impacts. Various applications and challenges are discussed, offering insights into the development of sustainable materials.

Chapter II outlines the materials and experimental methods used to develop and test polyester/alfa fiber composites. It describes the preparation and treatment of alfa fibers, their morphological analysis, and the composite fabrication process. Details on mold manufacturing, natural aging, and sample machining are provided. Finally, mechanical tests—including flexural and toughness tests—are conducted, followed by discussion of the results.

Chapter III focuses on the numerical validation of the experimental results from the three-point bending tests of the polyester/alfa fibre composite, as well as on the fracture behaviour. This approach combines experimental data with advanced numerical modelling using Abaqus 2022, enabling an in-depth analysis and rigorous validation of the material's performance.

Chapter VI is devoted to studying the effect of accelerated ultraviolet (UV) aging on the mechanical behaviour of flax/epoxy composites and their hybrid versions reinforced with carbon, Kevlar, and glass fibres. Artificial aging was performed over 720 hours in a custom-designed UV chamber equipped with germicidal UV-C fluorescent lamps (254 nm, 28W). Three-point bending tests were conducted to evaluate the mechanical degradation of the materials before and after exposure. The measured parameters included stress–strain behaviour, flexural modulus, ultimate strength, and surface hardness. Results showed significant degradation of mechanical properties across all composite types, with varying degrees depending on the reinforcement type. Additionally, a numerical model was developed in Abaqus to simulate the bending behaviour, showing good correlation with experimental data. This study highlights the substantial impact of UV aging on the structural integrity and durability of flax-based hybrid composites.



Chapter I

A state of the art review on biocomposites

Chapter I provides a state-of-the-art review on biocomposites, focusing on their composition, properties, and environmental advantages over traditional materials. It explores various natural fibres and biodegradable matrices, manufacturing techniques, and current application areas. This chapter offers a comprehensive theoretical foundation for understanding recent developments and challenges in the field of biocomposites.

Chapter I : A state of the art review on biocomposites

I.1. Introduction

Heightened environmental awareness and social concerns, together with tightening environmental regulations and the unsustainable reliance on petroleum, are driving the development of innovative, eco-friendly components. Natural fibers are among the range of environmentally friendly materials. In addition, natural fiber-reinforced polymer composites (NFRPCs) offer several advantages over synthetic fiber systems, including lower weight and cost, enhanced mechanical performance, superior molded-part surface finish, renewability and ready availability [1], processing flexibility, excellent biodegradability, and minimal health risks [1,2]. A substantial share of contemporary academic and industrial research is directed toward developing eco-friendly, biodegradable composites that incorporate diverse natural fibers as reinforcements, with prospective applications spanning a wide range of social and industrial sectors. Natural fiber-reinforced composites exhibit mechanical and physical properties that range from moderate to excellent [3]. However, natural fibers exhibit several limitations. Their chemical composition comprising cellulose, hemicelluloses, lignin, pectin, and waxy constituents renders them hydrophilic and prone to moisture uptake, which in turn weakens interfacial bonding between the fibers and the polymer matrix. Furthermore, the dissimilar chemical structures of natural fibers and polymer matrices often reflecting a polarity mismatch pose a persistent challenge to achieving effective interfacial bonding. These issues are the primary causes of inefficient stress transfer at the fiber–matrix interface of the resulting composites. The incorporation of nanomaterials offers a promising route to address these challenges. It is standard practice to incorporate additional entities such as fibers, fillers, particles, and whiskers into the reinforcements to enhance multiple attributes, notably the mechanical and physical properties of the target composites [4]. In general, the reinforcing effectiveness of fillers in composites is inversely related to their size and directly related to their surface-to-volume ratio [5]. Nanoparticles have emerged as exceptional fillers, capable of significantly improving and tailoring the mechanical and physical properties of polymer composites [6,7]. The high specific surface area of these nanofillers facilitates strong interfacial bonding with the polymer matrix, thereby further enhancing the overall composite properties [8,9]. Natural fiber-reinforced composites (NFRCs) incorporating nanoparticles are particularly advantageous due to their environmental friendliness, reduced water absorption, and improved mechanical performance. As a result, they are increasingly used in various sectors, including construction (partition boards, ceiling paneling), transportation

(automobiles, railway coaches), aerospace, packaging, consumer products, and more [10]. Moreover, the incorporation of specific nanomaterials into natural fiber–polymer composites can impart advanced functionalities including antibacterial and anti-odor effects, UV protection, and enhanced hydrophobicity [11–13]. Currently, a broad range of nanotechnology-based textiles is utilized across multiple sectors including sports equipment, the beauty industry, space technology, and apparel [1]. Layth Mohammed et al. [1] investigated natural fiber reinforcements in both thermosetting and thermoplastic composites to assess how various chemical treatments affect their mechanical and thermal properties, water absorption, flame retardancy, and tribological behavior. The authors concluded that chemical treatment of natural fibers enhances interfacial adhesion between the fiber surface and the polymer matrix, thereby improving the physicommechanical and thermochemical properties of the NFRPCs. Roopesh Kumar Sinha et al. [3] investigated nano-SiO₂ as a filler to enhance the dynamic response of epoxy composites reinforced with natural fibers. They characterized and compared the mechanical and dynamic properties of composites with and without the filler. The findings indicate that nano-SiO₂ is a suitable filler material owing to its outstanding properties, low cost, low density, and ready availability. Vijayakumar Pujar et al. [14] investigated the effects of primary reinforcements glass, carbon, and Kevlar—together with secondary reinforcements, including SiC, Si₃N₄, Al₂O₃, MoS₂, WS₂, TiO₂, SiO₂, ZrO₂, MbO₂, ZnS, CaCO₃, CaO, MgCO₃, Ta/NbC, MgO, TiC, polytetrafluoroethylene (PTFE), graphite, and hexagonal boron nitride, on the mechanical and tribological (wear and friction) properties of polymer-based composites. In addition to variations in particle size and volume fraction of the secondary reinforcements, the influence of the **morphology** of the primary reinforcements is likewise emphasized. Love Kerni et al. [15] surveyed the various types of natural fibers that can serve as reinforcements in polymer composites, employing a range of production and processing techniques. Subsequently, the composites’ mechanical, tribological, and related properties are analyzed and reported, together with a range of prospective applications. The contemporary challenges and issues concerning the mechanical and tribological properties of plant-based natural fiber-reinforced thermoset composites have been addressed by Vijay Chaudhary et al. [16]. They found that interfacial adhesion is the primary factor governing mechanical and tribological performance. The wear and frictional behavior of plant fiber–reinforced thermoset composites can be tailored by judicious selection of fillers and by optimizing reinforcement orientation. A.V. Kiruthika et al. [17] provided a comprehensive review of polymer composites reinforced with various bast fibers (banana, flax, hemp, jute, kenaf, and ramie), including the effects of chemical treatments on their

physicomechanical properties and applications. Several review articles have examined NFRPCs from different perspectives ; however, none has addressed the influence of inorganic nanoparticles on their overall performance. To address this gap, the present work reviews the properties of natural fibers and their applications, including mechanical, flame-retardant, thermal, tribological, water-absorption, and biodegradable characteristics. This review provides an overview of material selection for the synthesis of advanced composite materials. It discusses the advantages of bio-based fibers as reinforcements, the diversity of natural fibers, their resulting properties, and their hierarchical structures. Furthermore, the paper aims to provide a brief overview of general methods for improving the interface between these fibers and polymer matrices filled with inorganic particles. Additionally, this article summarizes textile technologies used to develop various fiber architectures such as woven roving and strands and the corresponding reinforcing capabilities. It also addresses processing methods for natural fiber–reinforced composites. Finally, the review correlates processing routes, structure, and the resulting composite properties to provide a comprehensive understanding of the topic.

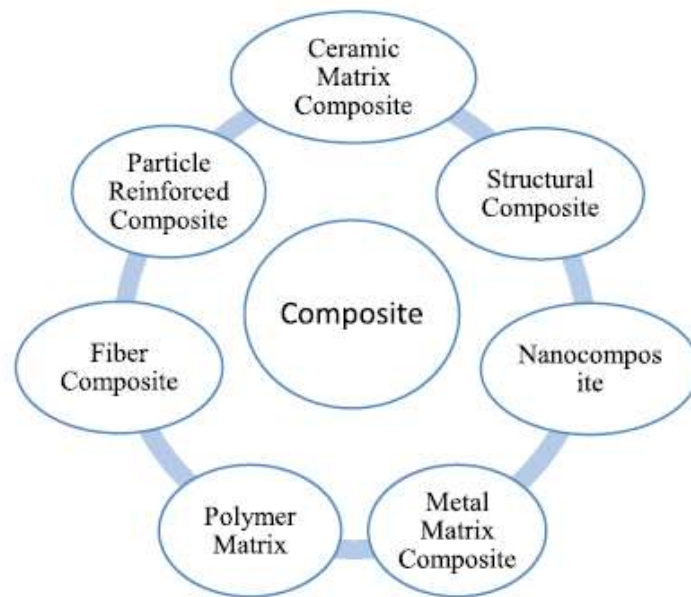


Figure I.1 : Different types of Composite Material.

I.2. Composites

Composite materials are formed by combining two or more constituents to attain distinct structural characteristics. Within such materials, the individual constituents retain, to some extent, their inherent properties [18]. The concept of composites is not new; it has been practiced for centuries throughout human history [19]. Synthetic reinforcements typically include carbon, thermoplastics, alumina, boron, silicon carbide, steel, and silicon nitride.

Additionally, materials such as coconut shells, eggshells, palm kernel shells, periwinkle, *Thaumatococcus daniellii*, bagasse, banana peel, and yam peel are increasingly investigated as natural fillers in composites, with the aim of enabling successful applications across multiple sectors. Typically, a composite comprises two primary constituents: the matrix and the filler or reinforcement (Figure I.1). The matrix serves as an adhesive for the reinforcements, binding the filler materials and transferring the applied load to the reinforcing elements. It also stabilizes the reinforcement architecture and protects the composite from environmental damage. In contrast, the fillers or reinforcements are the load-bearing constituents of a composite, providing the strength and rigidity required to sustain structural loads. They are typically supplied in fibrous forms such as natural fibers or in specific morphologies, including chopped glass and carbon [19, 20]. Additionally, nanomaterials are employed as secondary reinforcement or filler phases to enhance the composite's properties. These elements thereby constitute a fiber-reinforcing phase that upgrades the composite by improving its mechanical and physical properties. Composites can be classified into various categories based on the nature of the matrices and fillers/reinforcements. Composites can be categorized into four types based on the type of reinforcements: fiber-reinforced, particulate-reinforced, structural composites, and nanocomposites. Regarding the matrix, composites are classified into three types : metal matrix, ceramic matrix, and polymer matrix composites [20, 21].

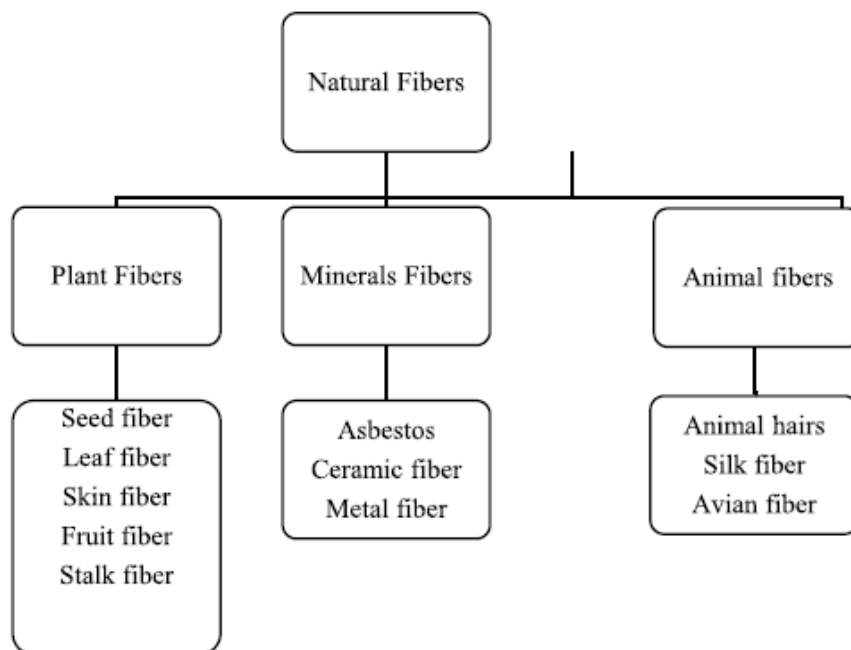


Figure I.2 : Types of natural fiber.

I.3 Natural Fiber

Natural fibers derived from plant or animal sources are neither synthetic nor man-made. Fibers such as oil palm, sisal, flax, and jute sourced from both renewable and nonrenewable resources have attracted considerable attention over the past few decades for use in composite materials [1]. The principal classification of natural fibers is as follows (Figure I.2):

I.3.1 Animal Fiber

Animal fiber is a natural, protein-based fiber derived from animals, including wool, mohair, silk, alpaca, and angora. In general, animal-based fibers such as silk and wool are employed as reinforcements in greens composites. Attributes such as surface toughness, high aspect ratio, flexibility, and reduced hydrophilicity give animal fibers a practical advantage over other natural fibers.

I.3.2 Mineral fiber

Mineral fibers are fibers or slightly altered fibers derived from minerals. They can be classified into the following categories: asbestos, serpentine, and anthophyllite amphiboles. Asbestos, which consists of six naturally occurring mineral fibers, is the most well-known group. However, these fibers are associated with health risks such as lung fibrosis, pleural disease, lung cancer, and mesothelioma [22]. Due to these health concerns, asbestos and other mineral-based fibers are rarely used.

I.3.3 Plant fiber

Plant fibers among the most extensively investigated natural fibers are widely used as reinforcements. They are commonly classified into six primary categories: seed fibers, bast fibers, leaf fibers, fruit fibers, stalk/straw fibers, and others (Figure I.2). Seed fibers originate from plant seeds (e.g., cotton, kapok), whereas bast (skin) fibers are derived from the outer cell layers of stems and generally exhibit higher tensile strength than most other natural fiber types ; typical sources include flax, jute, hemp, ramie, and kenaf. Leaf fibers are extracted from leaves (e.g., abaca, sisal, pineapple). Fruit fibers are obtained from the fruit coconut coir being a canonical example. Stalk (straw) fibers come from plant stalks, including wheat, corn, and barley straws, as well as various bamboo and grass species; tree wood is also considered a stalk fiber [23]. From an application standpoint, plant fibers offer a credible alternative to synthetic fibers [24]. Their types vary with the anatomical origin within the plant (Table I.1). Owing to their biodegradability and eco-friendly nature, the use of plant/natural fibers has increased substantially, leading to a growing preference for these materials over synthetic

counterparts. They are further sought for their high strength, stiffness, and renewable character [24].

I.4 Alfa fiber

Alfa is a perennial grass primarily found in the semi-arid regions of North Africa and the southern Iberian Peninsula, including Spain and Portugal (Figure I.3) [25]. It is commonly known as Halfa in North African countries and Esparto in southern Spain [26, 27]. The species is currently widespread over a broad geographical range, especially in Morocco and Algeria, covering approximately 2.2 million hectares in Morocco and 4 millions hectares in Algeria [28]. Characteristic of Mediterranean arid and semi-arid environments, Alfa plays a crucial role in combating desertification. It grows in clumps, reaching heights of approximately 1–1.2 m, and forms broad tufts with an average density of around 4,000 plants per hectare in typical stands (Figure I.4).

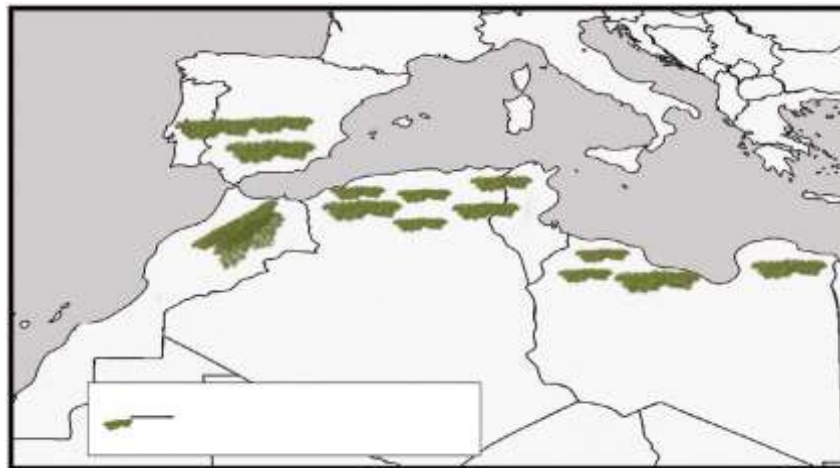


Figure I.3 : Global distribution of alfa grass shown in green color.



Figure I.4 : Alfa plant and its traditional use (A) alfa plant growing and (B) example of traditional handcraft items made from Alfa.

Alfa fibers based polymer composites

Polymer composites are formed by combining multiple constituents, with at least one polymer serving as the matrix [30,32,33]. This integration yields enhanced mechanical and thermal properties compared with a single, monolithic material [31]. Manufacturing generally proceeds in two principal stages : (i) preparation of the matrix and filler, followed by (ii) addition of the reinforcement to the matrix [30,31]. Alfa fiber (AF) and its derivatives have been employed as reinforcements in numerous studies, contributing to improved mechanical and thermal performance of polymer matrices. This section provides an overview of the processing and properties of Alfa fiber polymer composites (AFPC).

Properties of alfa fibers reinforced polymer composites

In recent years, natural fiber based polymer composites have gained growing importance across a wide range of applications [30]. The selection of natural fibers for composite fabrication is guided by properties such as recyclability, abundance, low cost, biodegradability, and high specific strength. Consequently, numerous studies have examined diverse fiber sources including alfa, rice husk, luffa, hemp, sisal, kenaf, coir, flax, pinecone, and bagasse [42–46]. In the semi-arid regions of North Africa, alfa fiber is the most commonly used reinforcement for polymer composites, primarily due to its abundant availability in the area [42]. The advantages of alfa fibers as reinforcing agents have been assessed in conjunction with polymer matrices to produce composites exhibiting notable mechanical, thermal, and chemical properties [45]. For this purpose, alfa-derived fillers may be incorporated as R-AF, AT-AF, AF-CMF, AF-CNC, or AF-CNF. The properties of polymer composites based on natural alfa fibers have been extensively investigated in the literature [39]. Beyond their economic and environmental benefits, these fibers constitute renewable feedstocks for new green products with appreciable physical and chemical characteristics. Moreover, incorporating R-AF into polymer matrices has demonstrated significant improvements in both tensile and flexural strength [41]. This enhancement also depends on the hydrophilic nature of the polymer matrix, which can improve tensile and flexural strength [31]. Conversely, the thermal degradation of alfa fibers is a critical property to assess: between 50 °C and 400 °C, the fibers undergo chemical and physical changes (hydrolysis, dehydration, and depolymerization) [36]. However, the use of native alfa fibers as reinforcements can be limited by their thermal stability, high moisture uptake, and weak interfacial bonding. These issues can be addressed through chemical and physical treatments, notably alkali treatment and silane coupling agents. Chemical routes include alkali delignification, bleaching to extract

cellulosic fibers, and hydrolysis to produce nanocellulose. Consequently, several studies have investigated alkali treatment to modify fiber surfaces and enhance the properties of alfa fiber/polymer composites [32, 42].

Elfehri *et al.* investigated alkali treatments of alfa fibers and their effects on the mechanical and thermal properties of biocomposites [29]. Their findings show a significant increase in crystallization rate upon alfa-fiber incorporation and an improvement in thermal stability following alkali treatment [29]. In addition, bleaching is employed as an alternative surface-modification route to enhance both the appearance and performance of composites. Multiple studies report that bleached-alfa-fiber composites exhibit improved mechanical properties relative to untreated counterparts primarily due to stronger fiber-polymer interfacial adhesion while also displaying enhanced visual appearance [38 ; 30,39]. Another route to enhance the utility of alfa fibers in composites is acidic hydrolysis using phosphoric acid, sulfuric acid, or a citric/hydrochloric acid mixture to produce nanocellulose. Such nanocellulose has been widely used to reinforce polymers, yielding nanocomposites with optimized filler contents and attractive thermal and mechanical properties [49]. Alfa-derived nanocellulose is frequently employed to improve the mechanical and barrier performance of biodegradable polymers, which generally underperform relative to synthetics. Several studies have reported edible nanocomposite films by incorporating CNF or CNC as nanoreinforcements into polymer films [50]. Specifically, El Achaby *et al.* observed a significant enhancement in tensile properties of nanocomposite films upon adding alfa-derived nanocellulose [49]. In addition, incorporating nanocellulose into polymer matrices improves thermal stability and interfacial adhesion, as demonstrated by Kassab *et al.* in poly (vinyl alcohol) matrices [36].

Application of alfa fibers composites

For many years, environmental pollution has been exacerbated by the increasing use of petroleum-based polymers [51–53]. Accordingly, the potential of biodegradable, natural fibers such as alfa fiber (AF) has been explored across diverse applications over the past decades [51, 42, 54]. Biopolymers represent an emerging trend in polymer science still under development aimed at replacing synthetic polymers for environmentally friendly products [51]. Given the excellent properties of R-AF and its derivatives, a wide range of applications has been reported for alfa fiber polymer composites (AFPC) [36]. The main application areas span multiple industrial sectors, including packaging, automotive, aerospace engineering, and construction engineering.

Alfa fiber composites in the packaging materials manufacturing

The packaging industry is among the leading sectors in which natural fibers hold substantial promise [33]. It accounts for a major share of global plastic consumption, thereby contributing to pervasive environmental pollution [51]. For environmental reasons, the use of biodegradable polymers has gained increasing support from both industry and researchers. However, their relatively high cost remains a significant barrier [57]. A logical progression is the development of biodegradable polymers reinforced with natural fibres, such as alfa (AF) [56]. To provide the packaging sector with eco-friendly, sustainable, and cost-effective solutions, researchers have developed various alfa fibre polymer composites (AFPCs) that show promising potential for packaging applications.

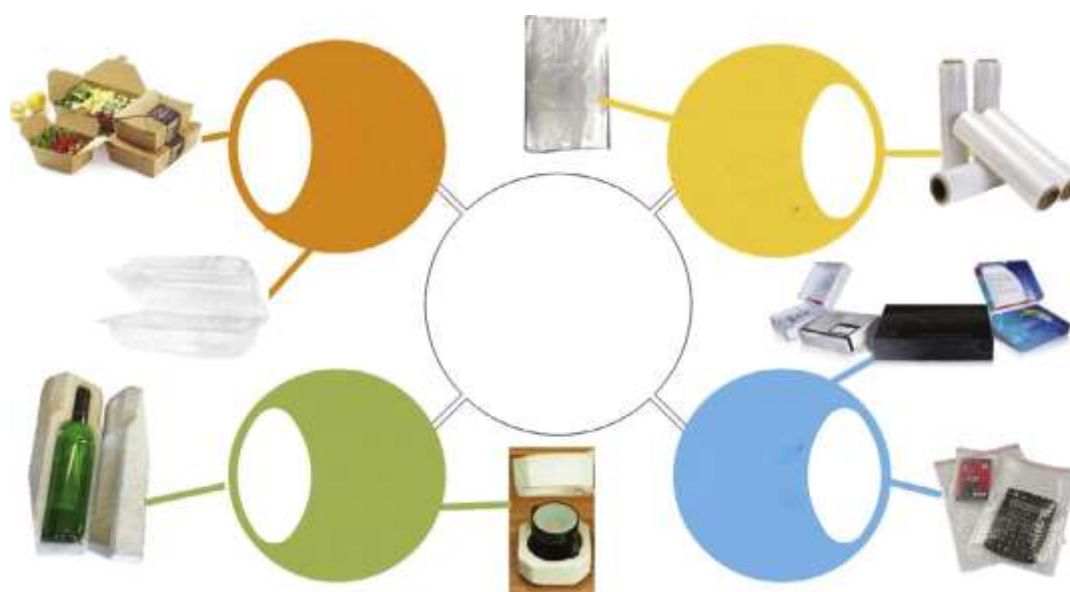


Figure I.5 : Categories of the potential application of AF reinforced composite in packaging.

Figure I.5 illustrates the main categories of packaging applications for AFPC materials. The use of raw alfa fibers (AF) or their derivatives in packaging primarily targets (i) improved mechanical properties elongation, stiffness, and strength (ii) weight reduction, and (iii) cost lowering by decreasing the polymer content in AFPCs [51,58]. For instance, Hammiche et al. investigated AF in poly(hydroxybutyrate-co-valerate) (PHBV) [42] : alkali-treated AF increased the tensile strength by 4–16% (depending on AF content) and stiffness by 7–25% in PHBV-AF composites, outperforming untreated fibers [42]. Similar trends were reported for biocomposites with a sunflower-oil-based matrix and AF filler, where the best tensile and thermal properties occurred at 7.5 wt. % AT-AF [53]. Moreover, in AF-CNC nanocomposites, alginate, κ -carrageenan, and chitosan films containing 8 wt. % AF-derived CNC (Figure I.6) achieved improvements of 94% and 117%, 145% and 104%, and 85% and 60% in tensile strength and Young's modulus, respectively [49].

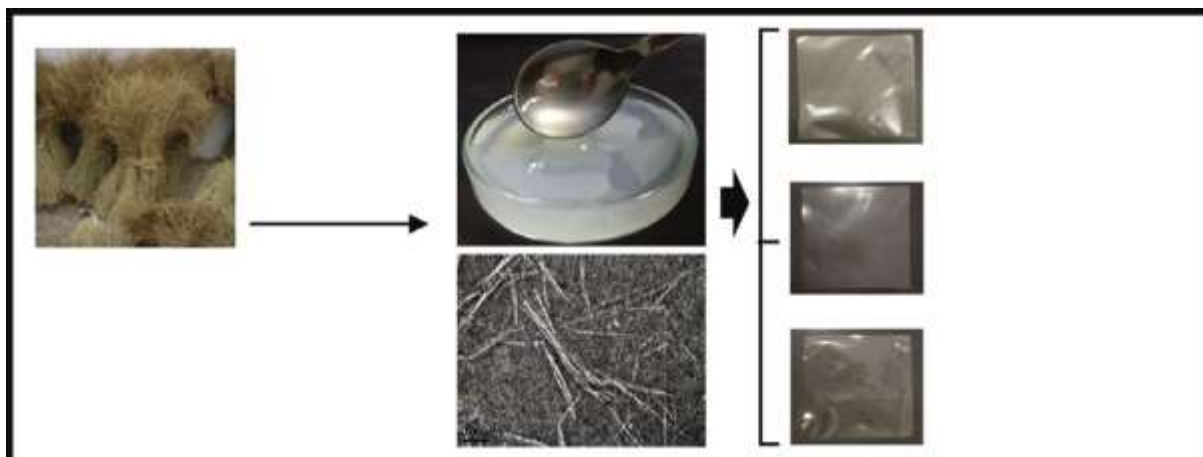


Figure I.6 : Development of bio nanocomposite packaging films based on AF-CNC [49].

In another study, AF-CNF–based composite films were developed: poly (vinyl alcohol) nanocomposite films containing 15 wt. % AF-CNF (Figure I.7) exhibited increases of 74% in tensile strength and 90% in tensile modulus, respectively [36].

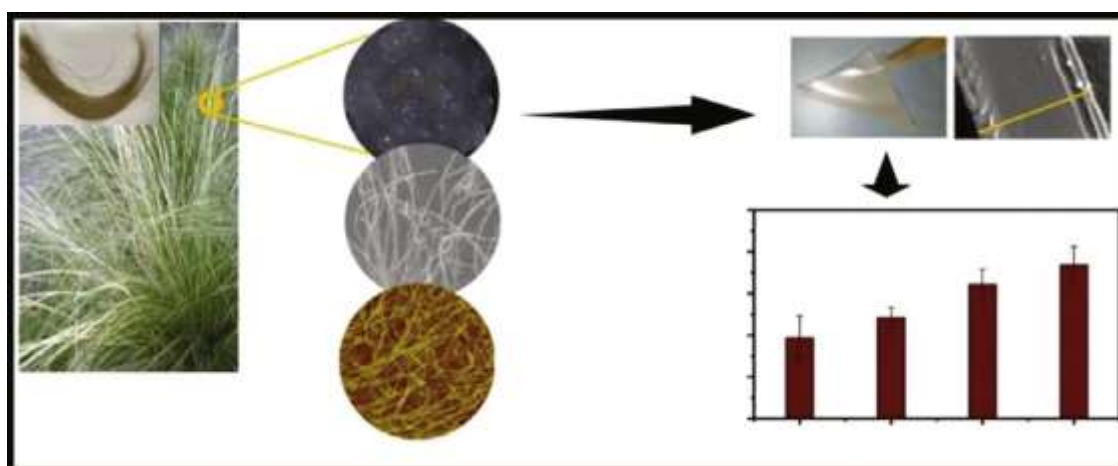


Figure I.7 : Poly (vinyl alcohol) nanocomposite films based on AF-CNF with enhanced mechanical properties [36].

Other AF-based composite materials with enhanced mechanical properties and potential for packaging applications include AF-reinforced polycaprolactone [37], AF-based Mater-Bi-type bioplastics [29], AT-AF-reinforced poly(β -hydroxybutyrate) -co-poly(β -hydroxyvalerate) (PHB-co-PHV) [59], and cellulosic AF-based starch and polylactic acid (PLA) systems [51].

Alfa fiber composites in the automotive and aerospace engineering

The automotive and aeronautical industries are the primary drivers of greens composites, as eco-friendly materials help address sector-specific pollution challenges ([60] ; S.B. [61]). Numerous automotive and aerospace parts are now produced from natural-fiber-based composites (S.B. [61]). In this context, AFPCs are applied to vehicle and aircraft components such as door panels, roofs, headliners, dashboards, and interior trims (S.B. [40]). Compared

with traditional glass or mineral fiber reinforcements, natural fibers offer the advantage of lower density, reducing overall vehicle weight and thus lowering fuel consumption [34].

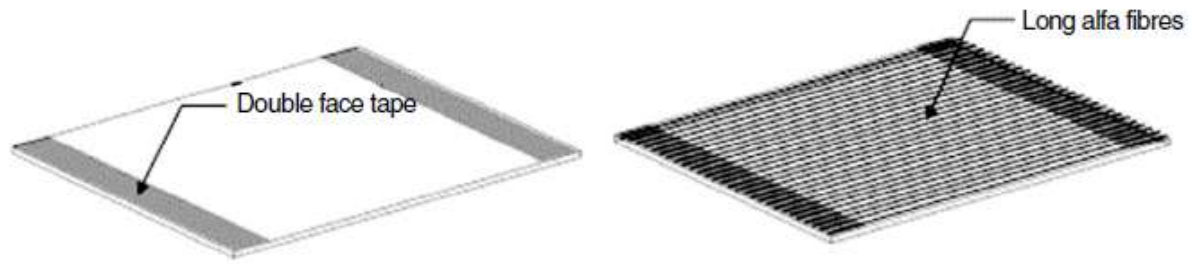


Figure I.8: Unidirectional alfa fibres used as fillers in composite materials, with potential applications in the automotive and aerospace industries [40].

In this context, Brahim et al. prepared a polyester composite reinforced with unidirectional alfa fibers (AF) (Figure I.8) and demonstrated that AF can yield stiff and strong materials suitable for aerodynamic and automotive components (e.g., wings, deflectors) (B.S. [62] ; S.B. [40]). Furthermore, polypropylene (PP)-based composites containing short AF were identified as attractive candidates for automotive technical parts, as AF effectively reinforces PP under both quasi-static and dynamic loading (strain rates up to $\approx 5 \text{ s}^{-1}$) and across various loading directions [61]. Similarly, employing bleached AF pulp board as a filler in an unsaturated polyester resin showed strong potential for automotive interior applications due to good impregnation between the matrix and the AF-based filler, leading to robust mechanical performance [25]. Additional AFPC systems investigated for potential automotive and aerospace use include AF-reinforced PP [63], AT-AF-based PP [55], AF-CMF-based polyurethane [35], and AF-CMF-based PP.

Alfa fiber composites in the construction applications

Natural fiber based structural composites for construction applications are emerging materials poised to play a pivotal role in next-generation building technologies [28,39]. Within this class, alfa-fiber (AF) based composites show substantial potential as alternative materials, particularly as wood substitutes in the construction sector. Accordingly, several AF-based composite systems have been developed with promising building-sector applicability. For instance, Sair et al. produced an AF-based polyurethane composite targeted at acoustic insulation and thermal applications ; incorporating 20 wt.% AT-AF increased tensile strength and Young's modulus by 74.12% and 48.14%, respectively, evidencing strong mechanical performance [39]. AF has also proven an effective reinforcement for high-density polyethylene (HDPE) composites, where tensile strength rose proportionally with fiber

content across the 20–50 wt.% range [44]. In a separate study, composite earth bricks containing 1 wt. % AT-AF were identified as potential building materials, exhibiting improved thermal conductivity alongside excellent mechanical properties [64]. Additional AF-based systems with prospective use in construction/structural engineering include AF-reinforced PVC [48], AF-reinforced plaster [34], AF-based poly(β -hydroxybutyrate) [59], long unidirectional AF-reinforced mortar, and AF-CMF-based polyurethane [65].

I.5 Composition of natural fiber

Natural fibers are composed primarily of cellulose (α -cellulose), hemicelluloses, lignin, pectin, and waxes [66]. Figure I.9 provides a schematic of the natural plant cell wall [67]. Structurally, fiber cells resemble hollow tubes whose walls comprise a primary cell wall and a secondary wall itself differentiated into three sub-layers (S1, S2, S3) surrounding a central lumen (the inner cavity of the cell). The walls are chiefly constituted of cellulose, hemicelluloses, and lignin. The properties of the fiber's regular strands (microfibrillar assemblies) vary with botanical source, plant age, and isolation/extraction method [68]. The chemical composition of selected plant fibers is summarized in Table I.2

Table 1.1: List of largest producer countries of plant fiber [69].

Plant fibers	Type	Worldwide production amount (103 ton)	Countries
Flax	Bast	830	Canada, France Belgium
Hemp	Bast	214	China, France, Philippines
kenaf	Bast	970	India, Bangladesh, USA
Ramie	Bast	100	China, Brazil, Philippines, India
Jute	Bast	2300	India, China, Bangladesh
Abaca	Leaf	70	Philippines, Ecuador, Costa Rica
Pineapple	Leaf	74	Philippines, Thailand, Indonesia
Sisal	Leaf	378	Tanzania, Brazil

Coir	Fruit	100	India, Sri Lanka
Palm	Fruit	40	Malaysia, Indonesia
Cotton	Seed	25000	China, India, USA
Bamboo	Grass	30000	India, China, Indonesia
Bagasse	Grass	75000	Brazil, India, China

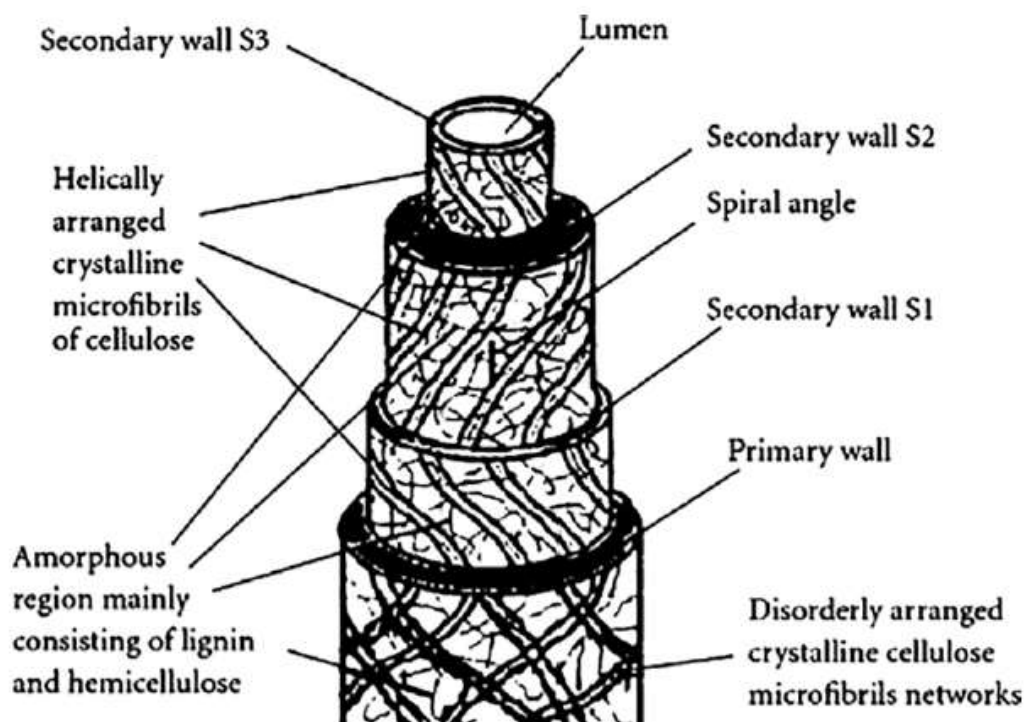


Figure I.9 : Diagram of natural plant cell walls [70].

Table I.2 : The chemical composition of plant fiber [66].

Plant fibres	Cellulose (wt %)	Hemicellulose (wt%)	Lignin (wt %)	Pectic (wt%)	Wax
Flax	71	18.6-20.6	2.2	2.3	1.7
Hemp	70.2-74.4	17.9-22.4	3.7-5.7	0.9	0.8
kenaf	31-39	21.5	15-19	3-5	-
Ramie	68.6-76.2	13.1-16.7	0.6-0.7	1.9	0.3
Jute	61-71.5	13.6-20.4	12-13	0.2	0.5
Abaca	56-63	20-25	7-9	1	3
Pineapple	70-82	-	5-12	1.1	-

Sisal	67-78	10-14.2	8-11	10	2
Coir	36-43	0.15-0.25	41-45	3-4	-
Palm	65	29	-	-	-
Cotton	82.7	5.7	-	0-1	0.6
Bamboo	26-43	30	21-31	-	-
Bagasse	55.2	16.8	25.3	-	-

I.6 Chemical treatment of natural fiber

The hydrophilic nature of natural fibres is inherently incompatible with the hydrophobic character of most polymer matrices, as the polarity of cellulosic fibres directly influences the fibre–matrix interface [71]. Pre-treatments enable chemical modification and/or cleaning of the fiber surface [72], thereby improving composite strength, mitigating aging effects, and enhancing interfacial adhesion [73]. An appropriately engineered interface not only increases durability but also imparts structural stability to the composite [74,75]. In practice, the overall performance of a composite can be tuned by tailoring the chemistry of the fiber–matrix interphase, since specific interfacial properties govern load transfer from matrix to fiber, limiting matrix-borne stress and improving mechanical properties. To this end, fiber surfaces are commonly modified via chemical treatments to strengthen interfacial bonding and promote mechanical interlocking with the matrix. Among these, alkaline (alkali) treatment is one of the most widely applied for natural fibers. It is used with both thermoplastics and thermosets [76] to improve wetting, increase surface roughness, and expose additional cellulose on the fiber surface thereby increasing the number of potential reaction sites [77]. Although natural fibers offer numerous advantages, their hydrophilicity promotes moisture uptake, which in turn degrades fiber–matrix adhesion during composite formation [78]. A typical protocol employs 5–6 wt. % NaOH for a limited duration to enhance interfacial adhesion strength; this treatment increases surface roughness, removes lignin, hemicellulose, waxes, and oils, and enriches the surface in accessible cellulose [79]. Numerous studies have reported significant alkali-treatment effects on the tensile strength of different composite systems [38,81]. As an illustrative case, Hameem et al. [82] investigated the effect of water absorption on the tensile and flexural properties of polyester composites reinforced with both treated and untreated Napier grass fibres. All specimens exhibited property degradation after immersion ; however, composites with untreated fibers absorbed more water and,

consequently, showed greater reductions in tensile and flexural strength than those reinforced with treated fibers.

I.7 Natural Fiber Reinforced Polymer Composites (NFRPCs)

Natural fiber reinforced polymer composites (NFRPCs) comprise a polymer matrix embedded with high-strength natural fibers [83]. The matrix serves as a binder, consolidating the fibers and transferring load among them, while also shielding the reinforcement from environmental and mechanical damage. The chemical makeup of matrices and thus their reactivity toward fiber surface functionalities varies across systems. Polymers are generally classified as thermoplastics or thermosets. Among these, thermoset polymers by virtue of their crosslinked architecture offer substantial design flexibility in tailoring target characteristics, along with high strength and modulus [80,81]. In practice, thermoplastics such as polyethylene [84], polypropylene (PP), and poly (vinyl chloride) (PVC) are widely paired with biofibers, whereas phenolic, polyester, and epoxy resins are representative thermosets used in NFRPCs [83] (Tables I.3 and I.4).

Table I.3 : Some main characteristics of thermosets matrix [85].

Thermosets	Density (g/cm³)	Tensile modulus (GPa)	Tensile strength (Mpa)		Compression strength (Mpa)
Polyester	1.0-1.5	2.0-4.5	40-90		90-250
Epoxy	1.1-1.6	3.0-6.0	28-100		100-200
Vinyl ester	1.2-1.4	3.1-3.8	69-86		86
Phenolic	1.29	2.8-4.8	35-62		210-360

Table I.4 : Some main characteristics of thermoplastics matrix materials [86].

Thermoplastics	Density (g/cm³)	Tensile modulus (GPa)	Tensile strength (Mpa)	Melting temperature (°C)
Polypropylene (PP)	0.90-0.91	1.1-1.6	20-40	175
Polyethylene (PE)	0.91-0.95	0.3-0.5	25-45	115
Polyvinyl chloride (PVC)	1.38	3.0	53	212
Polystyrene (PS)	1.04-1.05	2.5-3.5	35-60	240

High density PP	0.94–0.97	0.5–1.1	30–40	137
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Across diverse studies of NFRPCs, it is consistently observed that their properties depend on fiber type and origin as well as moisture conditions. Beyond these, additional determinants microfibrillar angle [87], defects [88], cell dimensions [89], physical attributes [81], chemical composition, and the fiber–matrix interaction [90] critically govern composite effectiveness. Nevertheless, As previously discussed, NFRPCs have inherent drawbacks : the chemical dissimilarity between hydrophilic natural fibres and hydrophobic polymer matrices impedes effective interfacial stress transfer, resulting in inefficient load sharing at the fibre-matrix interface. Achieving a high-quality interface therefore necessitates chemical modification of natural fibers. During such treatments, reagent functional groups can interact with fiber constituents and alter fiber structure [91]. For example, the abundance of hydroxyl groups renders natural fibers hydrophilic, promoting weak interfacial bonding with hydrophobic matrices and, consequently, inferior mechanical and physical performance [92]. Even so, composite characteristics remain tunable through fiber hydrophilicity [93], fiber loading [94,95] which can increase tensile properties at appropriate contents [92] and processing parameters, which influence both bulk properties and surface characteristics [83]. Moreover, the chemical constituents of the fibers decisively shape composite behavior [81]. Reflecting these complexities, a substantial body of work has assessed the suitability, competitiveness, and capabilities of natural fibers in polymer matrices, emphasizing surface modification and processing strategies to enhance fiber–polymer compatibility [96,97]. Complementary studies have examined application-specific natural fibers and provided comparative analyses of their mechanical performance [87].

I.8 Processing and manufacturing of natural FRP composites

Processing techniques have a significant impact on the final properties of fibre-reinforced polymer (FRP) composites and must therefore be carefully selected to ensure optimal performance. Manufacturing outcomes are governed by process parameters most notably temperature, pressure, and fiber characteristics (e.g., aspect ratio and volume fraction). In particular, optimizing the fiber aspect ratio and volume fraction is critical to effective load transfer and overall composite efficiency. Production of FRP composites generally entails thermo-mechanical operations; however, natural-fiber FRPs are constrained by lower permissible processing temperatures, which can limit the choice of matrices and the processing window.

1.8.1 Hand lay-up

The hand lay-up technique is among the most widely used composite fabrication methods due to its simplicity, low cost, and modest skill requirements. It entails draping the reinforcement and impregnating it with resin, and is applicable to both large and small components without inherent size limitations. Notwithstanding these advantages, hand lay-up can yield poor surface finish and high void content ; however, process enhancements (e.g., improved deaeration, controlled curing) can mitigate such defects. The method is compatible with woven and non-woven mats as well as rovings, and is frequently combined with other processes such as compression molding and autoclaving [99].

1.8.2 Resin transfer moulding

Resin Transfer Molding (RTM) has become widely adopted for producing components with excellent surface finish and low void content. As a closed-mold process, it limits the emission of volatile species during fabrication. In RTM, the resin is injected into a two-part sealed mold without direct exposure to air after the reinforcements have been pre-placed; impregnation is driven by applied pressure or vacuum assistance. The method is extensively used to fabricate high-quality parts for aerospace and automotive applications. Notably, the high surface quality can be achieved at moderate pressures and comparatively low mold temperatures ($\approx 60\text{--}90\text{ }^{\circ}\text{C}$), thereby reducing tooling costs.

1.8.3 Pultrusion

Pultrusion is an advanced continuous manufacturing process for producing unidirectional fiber composites. In this method, fibers are drawn from rovings, passed through a resin bath for impregnation, and then pulled through a heated die. The die promotes complete wet-out, regulates resin content, and shapes and cures the laminate; the cured profile is subsequently cut to length. Typical matrices are thermosetting resins such as epoxy, polyester, vinyl ester, and phenolic. Pultrusion is attractive and cost-effective owing to its short cycle times. Contact curing and controlled resin content yield parts with good wettability and high surface quality, while the process readily achieves high fiber volume fractions, thereby enhancing mechanical performance. The principal drawback is the high cost of heated molds. Pultrusion is widely used to manufacture structural components such as beams and girders.

1.8.4 Filament winding

Filament winding is a process primarily used for producing hollow, circular, or oval-sectioned components, such as pipes and tanks. In this method, fibre tows are passed through a resin bath before being wound onto a mandrel in various orientations, with the fibre feeding mechanism and the rotation speed of the mandrel controlling the winding process. Factors such as the winding angle and drag pressure play a crucial role in determining the mechanical properties of the resulting composite structures. Recent advancements in filament winding include the integration of robots, enabling smart manufacturing and enhancing the precision and efficiency of the process.

1.8.5 Compression moulding

Compression moulding is a conventional technique employed to fabricate both thermoset and thermoplastic composites. Consolidation and/or full cure are achieved by applying pressure and temperature, which are adjusted according to the specifications of the target part. The method is used predominantly for producing large flat or curved components. A common challenge, particularly for large parts, is maintaining uniform thickness. Compared with alternative fabrication routes, compression molding is widely regarded as one of the most economical processes for manufacturing high-quality composite components.

1.9 Factors affecting mechanical performance of natural FRP composites

The overall mechanical performance of fibre-reinforced polymer (FRP) composites, including natural-fibre FRPs, is determined by the properties of both the fibre and the matrix. While the matrix primarily serves a supportive role protecting the fibers and maintaining their alignment the fibers carry the majority of the applied load. Although the matrix contributes less to the global stiffness and strength, it exerts a pronounced influence on compressive and in-plane shear strengths, which depend strongly on matrix type [100,101]. Several additional factors that affect the mechanical properties of natural-fiber FRP composites are outlined in the following sections.

1.9.1 Fibre surface modification

Surface modification of fibres is commonly used to improve fibre–matrix adhesion in FRP composites. For instance, Pickering et al. [102] demonstrated that treating hemp fibres with 10 wt. % NaOH, combined with an optimized processing temperature, enhanced the mechanical properties of hemp fibre-reinforced polypropylene (PP). The performance gains were attributed to alkali treatment–induced increases in fiber crystallinity relative to untreated

fibers. Consequently, fiber surface modification is pivotal for strengthening interfacial bonding, thereby elevating the overall composite performance.

1.9.2 Fibre types and manufacturing parameters

The fiber type exerts a decisive influence on the mechanical performance of natural FRP composites. In particular, fiber structure and morphology govern load-bearing capacity by dictating how fibers interact with the matrix, thereby affecting composite strength, stiffness, and overall stress response. In the study by Pickering et al. [102], at relatively low fiber loadings (30–40 wt. %), longer fibers produced a superior reinforcement effect, underscoring the importance of fiber length especially at lower fiber contents. By contrast, a higher loading (50 wt. %) reduced mechanical properties, a decline attributed to fiber damage arising from greater frictional contact during processing, which compromises fiber integrity and, consequently, composite performance. As is well established, fiber architecture random, non-woven, woven, short, or unidirectional long strongly shapes final properties. Moreover, an optimal fiber volume fraction typically exists beyond which gains in mechanical properties plateau or decline; further increases can even deteriorate overall performance. Moisture effects are similarly critical. In the context of natural FRP composites, the study in [99] on hemp fiber–reinforced unsaturated polyester reported an optimal volume fraction of ~26 vol.% ; tensile and flexural properties decreased both below and above this value. Additionally, moisture uptake increased with fiber volume fraction and was exacerbated at elevated temperatures, primarily due to fiber swelling at the fiber–matrix interface.

1.9.3 Fibre orientation/dimension

In FRP manufacturing, fiber orientation and dimensions are critical determinants of mechanical performance. Unidirectionally reinforced laminates consistently outperform short-fiber systems in tensile properties, reflecting the importance of maintaining load-bearing continuity along the fiber axis. Fiber length strongly influences overall response, with numerous studies indicating that maximum mechanical properties are obtained when fiber orientation is aligned with the loading direction [47].

1.10 Advantages and drawbacks of natural FRP composites

Advantages of natural FRP composites include:

- Abundance,
- Higher specific strength and modulus compared to conventional glass fibres

-
- Light weight,
 - Cost-effectiveness
 - Reliability
 - Biodegradability
 - Non-toxicity
 - Less energy required for manufacturing
 - CO₂ absorption during their growth [73,103]

Some drawbacks of natural FRP composites :

- Hydrophilicity due to their structural morphologies and chemical compositions (cellulose, hemicellulose, lignin, pectin and waxy substances) Property variation (growing conditions and climate influencing the overall properties of fibres)
- Sensitivity to climatic conditions
- Low thermal stability
- Poor microbial resistance [103-105]

Several strategies have been developed to mitigate the aforementioned drawbacks, particularly via surface modification. These include chemical, plasma, thermal, biological, and enzymatic treatments. In parallel, a range of additives has been explored to improve flammability and fire resistance as well as resistance to microbial attack in FRP composites. The current state of knowledge on flammability and fire performance in natural-fiber FRPs has been comprehensively reviewed elsewhere [106,107]. Notably, certain additives have been reported to enhance moisture repellency while simultaneously improving fiber–matrix compatibility, thereby contributing to better overall composite performance [108,109].

I.11 Application of Natural fiber-reinforced polymer composite

The numerous advantages of NFRPCs make them highly desirable for a wide range of applications. Natural fibres such as jute, hemp, kenaf, oil palm, and bamboo, when used in polymer composites, have found applications in various sectors, including automotive (interior and exterior panels, gas tanks, and bumpers), structural components, packaging, and the construction/development industry (e.g., foundational boards and building sections) [98] (Figure I.10). European car manufacturers, including major German automakers such as BMW, Audi Group, Ford, Opel, Volkswagen, Daimler Chrysler, and Mercedes, have made significant efforts to enhance the use of NFPCs, particularly cellulose fibre composites, in the automotive industry. These efforts are particularly focused on car interiors [110] and exterior auto body components [96]. Automotive implementations illustrate this trend. For example,

the Mercedes-Benz A-Class incorporates composites of coconut fibres, rubber, and latex in its seats, while the E-Class features flax–sisal fibre–mat–reinforced epoxy door panels [92]. Audi uses flax/sisal mat–reinforced polyurethane for door trim panels, and Ford integrates kenaf fibres in the Mondeo model [111]. It has also been reported that Volkswagen employs a flax-fibre polypropylene composite in the door panels of the 2000 Chevrolet Impala [112]. Additionally, Toyota, Proton, Volvo, and other manufacturers have incorporated cellulose fibres in various vehicle components.

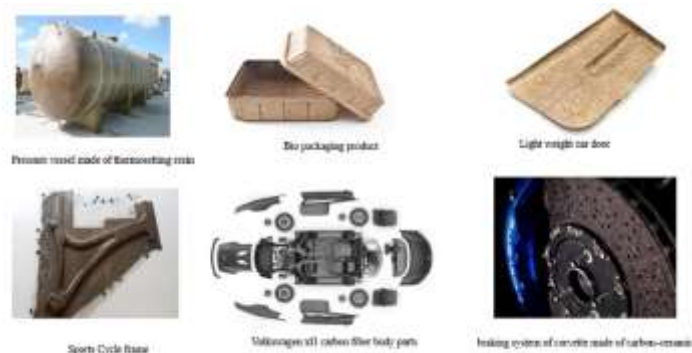


Figure I.10 : Application of natural fiber reinforced polymer composite.

Beyond automotive applications, NFRPCs are deployed across the electrical and electronics sectors (e.g., printed circuit boards and component assemblies), aerospace (fire-retardant panels and high-performance parts), sports and recreation equipment, marine (boats), machinery, office products, and more. Their widespread adoption reflects a combination of attributes low specific weight, high strength, low manufacturing cost, corrosion and wear resistance, complete biodegradability in appropriate systems, progressive surface conformity of molded parts, moderately high mechanical properties, and ready availability from renewable feedstocks that compare favorably with synthetic fiber–based composites [113]. In the construction industry, current use is concentrated on non-load-bearing interior components, which are less exposed to severe environmental stressors [114]. In parallel, the rise of green buildings has spurred interest in biocomposites as sustainable materials; as discussed earlier, the high stiffness-to-weight ratio, low density, and biodegradability of NFRPCs align well with these objectives [115]. Illustrative systems include sisal fiber–reinforced composites [116] and composites based on bamboo, sisal, and coir [117], which are employed in building panels, roofing sheets, door frames/shutters, transport components, packaging, window frames, helmets, panels, decking, railing, and fencing, as well as sports equipment such as tennis rackets, bicycle frames/forks/seat posts, and snowboards. Polymer nanofibers further expand the application space in biomedicine: they are used in burn and wound care, hemostatic materials, and device components with tailored functionalities. In

particular, electrospun or spray-spun biodegradable polymers deposited directly onto injured skin form fibrous mat dressings that support skin regeneration and accelerate healing [6]. Despite these advantages, NFRPCs face notable limitations including moisture uptake, restricted processing temperatures, and variability in raw-fiber quality that can constrain performance and application windows [118].

I.12 Hybridisation of natural FRP composites

I.12.1 Concept of natural FRP hybrid composites

Hybrid composites comprise mixtures of different fiber types within a single polymer matrix, enabling the advantages of one fiber to offset the limitations of another. Originally conceived as structural materials incorporating two or more reinforcements, hybrid systems combine the favorable attributes of synthetic and natural (plant-based) fibers through diverse manufacturing routes. Their application scope has broadened with the advent of advanced reinforcements such as carbon nanotubes and graphene [119,120]. Further performance gains can be realized by introducing nanoparticles, which strengthen the hybrid architecture. In natural-fiber FRP hybrids, the polymer matrix bonds the fibers, enhances load transfer, and improves surface quality; these matrices are generally categorized as thermosetting or thermoplastic resins. A detailed survey of polymers used in natural FRP systems is provided by Alagesan et al. [121]. Looking forward, hybrid composites can also integrate micro- and nanofillers, such as aluminium oxides, silicon nano/microparticles, carbon nanotubes, graphite, and fullerenes, as reviewed by Akpan et al. [122]. Such fillers typically improve mechanical properties by reinforcing interfacial bonding, while promoting better dispersion and matrix–fiber interactions. In summary, the principal categories of natural FRP hybrid composites distinguished by their reinforcing elements and orientations/arrangements are depicted in Figure I.11.

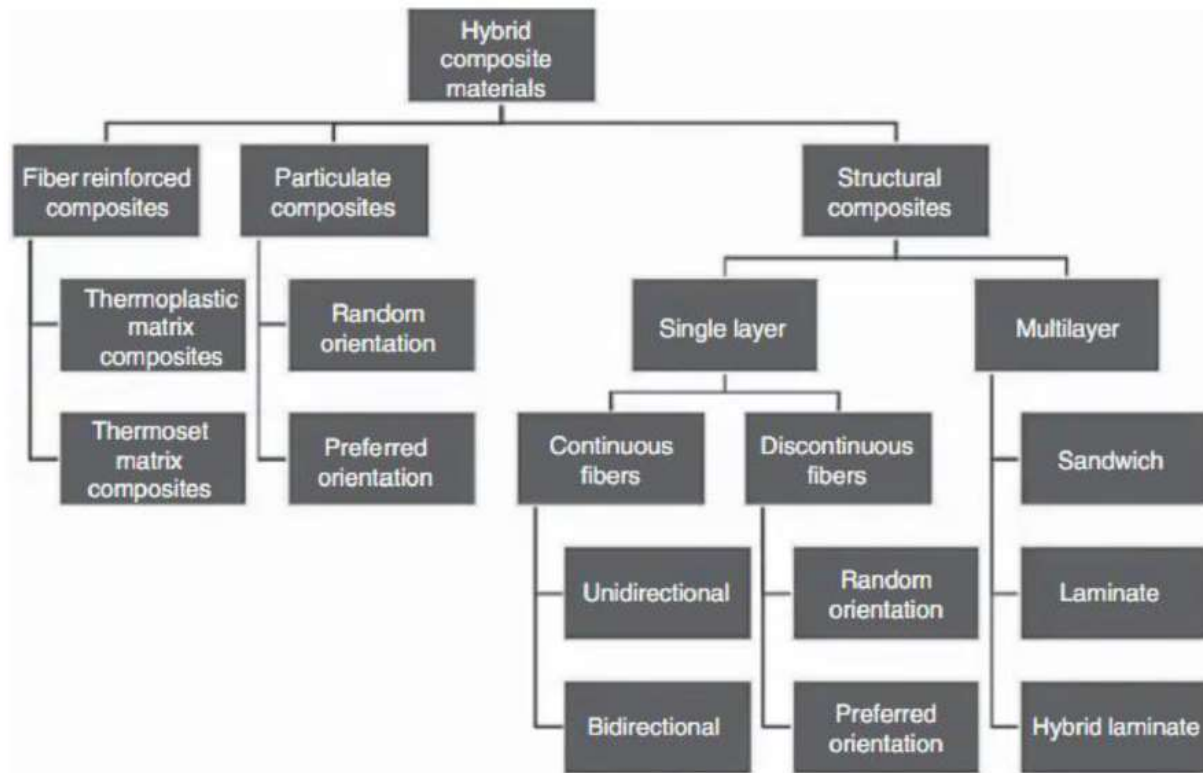


Figure I.11 : Various categories of natural FRP hybrid composites [121].

The fabrication of natural FRP hybrid composites typically begins with the preparation of fiber preforms, which are subsequently incorporated into a polymer matrix. A variety of processing routes can be employed, broadly classified by how fiber and matrix are combined into open-molding and closed-molding techniques. In open molding, the resin is exposed to the atmosphere during curing; in closed molding, it is not. Open molding is widely used due to reduced processing requirements and broad fiber compatibility, whereas closed molding is more efficient for producing three-dimensional (3D) laminates with lower material usage. Common open-molding methods include hand lay-up and spray-up. In hand lay-up, fibers are placed in an open mold and impregnated with matrix layer by layer until the target laminate thickness is achieved. In spray-up, chopped fibers and matrix are co-sprayed into the open mold to build the composite. Closed-molding methods include, but are not limited to, compression molding, extrusion, resin transfer molding (RTM), and injection molding. Extrusion employs bead-shaped thermoplastics that are mixed with fibers via rotating screws, compressed, and forced through a die. Injection molding uses either thermosetting or thermoplastic matrices to shape the composite, and typically involves high-viscosity melts and variable fiber arrangements along the mold cavity. Resin transfer molding (based on a thermosetting matrix and utilizing pultrusion-based practice in certain implementations) places dry fiber preforms into a clamped, two-part mold; molten resin is injected through

inlets to impregnate the preform, after which the part is cooled and removed for post-curing. Compression molding with either thermoplastic or thermosetting matrices mixes reinforcement and matrix and consolidates the charge under controlled temperature and pressure [123]. Variants include hot or cold compression as well as autoclave processing, wherein preforms are laid up in the mold and cured under vacuum and elevated temperature within the autoclave [124]. Across these routes, the cited manufacturing processes can be applied to produce natural FRP hybrid composites with enhanced properties, suitable for a broad range of structural and semi-structural engineering applications.

I.12.2 Hybridisation techniques

The formation of natural FRP hybrid composites entails combining different fibers/reinforcements within a single polymer composite system. Such hybrids are commonly classified based on reinforcement distribution into interply, intraply, and super-hybrid architectures. In interply hybridization, distinct constituents are stacked as separate plies at the laminate level; in intraply hybridization, constituents are combined within the same ply (e.g., parallel/commingled tows or fabrics) ; and in super hybridization, metal/composite layers and matrices are arranged in a prescribed sequence (e.g., fiber–metal laminates), as illustrated in Figure I.12.

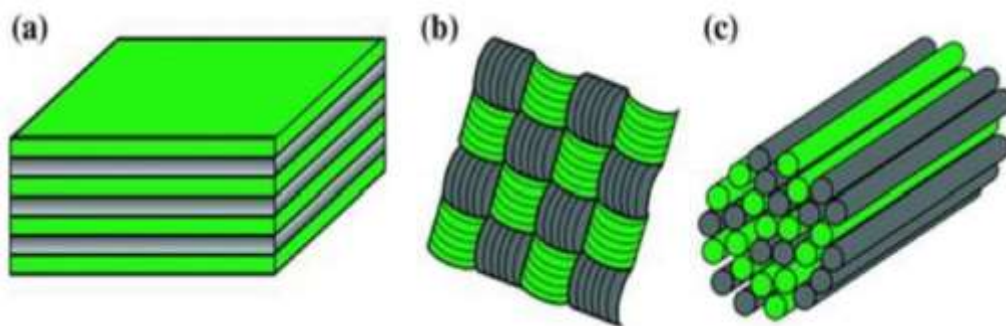


Figure I.12: Fibre arrangements within natural fibre-reinforced polymer (FRP) hybrid composites, illustrating: (a) interply, (b) intraply, and (c) intrayarn configurations [125,126].

Another key modality in natural FRP hybrids is dispersion hybridization, wherein the hybrid fibers are distributed within the primary fiber population according to prescribed patterns. Figure I.13 illustrates the principal dispersion schemes: (a) limited spreading, with each fiber class confined to separate layers; (b) increased layer count (finer interleaving of fiber classes); (c) dispersion at the fiber-bundle (tow) level; and (d) random fiber distribution [127].

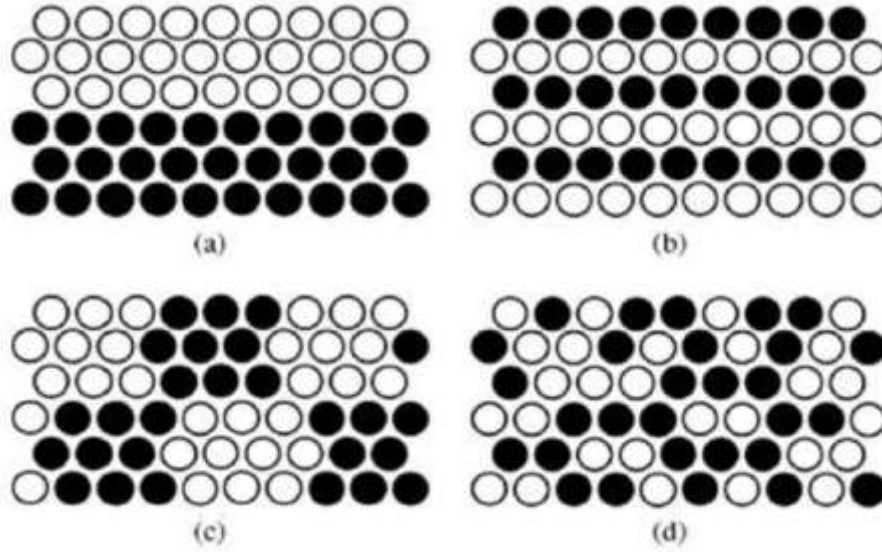


Figure I.13 : Amount of fibre spreading in (a) two layers, (b) alternating layers, (c) bundle-bundle dispersion and (d) irregular dispersion [126,127].

Moreover, the design of natural FRP hybrid composites commonly follows Puck's criteria, whereby the fracture strength and stiffness of the fibers must exceed those of the polymer matrix, while the matrix is selected to exhibit a greater elongation at break than the fibers [126]. Property enhancement via hybridization is then achieved by combining constituents in accordance with the rule of mixtures, as summarized in Eqs. (I.1) and (I.2).

$$P_H = P_{c1}V_{c1} + P_{c2}V_{c2} \quad (I.1)$$

Where, P_H represents resultant properties of natural FRP hybrid composites, P_{C1} and P_{C2} stand for corresponding components C1 and C2 of natural FRP hybrid composites, while V_{C1} and V_{C2} denote volume fractions.

$$V_{C1} + V_{C2} = 1 \quad (I.2)$$

This law primarily applies to static mechanical characteristics, including tensile and flexural properties of natural FRP hybrid composites. Dynamic properties, such as impact resistance, exhibit more variable results, depending on the geometric patterns applied during hybridization [128].

I.12.3 Properties and potentials of natural FRP hybrid composites

Natural FRP hybrid composites offer distinct advantages over monolithic systems, including the flexibility to tailor properties and the ability to select cost-effective fibers. Compared with monoliths, they commonly exhibit enhanced mechanical, thermal, chemical-resistance, and acoustic performance [129], while also providing lower cost, biodegradability, high strength, low weight, and fatigue resistance. Their physical and mechanical behaviors depend on

intrinsic fiber properties, fiber–matrix adhesion, and architectural/design variables such as fiber arrangement, volume fraction, length, and stacking sequence. Consistent with Edoziuno et al. [130], outcomes are further governed by the type of reinforcement, manufacturing technique, and filler–matrix interactions. Hybridization of natural fibers with synthetic fibers (e.g., carbon, glass) can raise strength and moisture/water resistance, although adverse effects may occur when the hybrid constituents possess markedly different specific mechanical characteristics [131]. The rule of mixtures provides first-order estimates of elastic properties in natural–synthetic hybrids, and tensile/flexural strengths are particularly sensitive in randomly arranged architectures. Applications span automotive, aerospace, electrical/electronic devices, building and construction, medical, and consumer sectors. In vehicles, natural FRP hybrids are used for seat backs, drawers, door linings, and door trim panels. German manufacturers (e.g., Volkswagen, Mercedes) employ cellulosic natural-fiber composites ; examples include coconut-fiber/latex seat composites and sisal-fiber/epoxy door panels [132]. Ford use kenaf-fiber/PP door panels in the Mondeo, and Toyota and Volvo likewise incorporate cellulose fibers in various components. Beyond polymers, bamboo fibers reinforce concrete elements, and sisal composites are used in roofing [132]. In aerospace, adoption of natural FRP hybrids is increasing to satisfy energy-absorption and safety requirements; glass and carbon fibers remain pervasive reinforcements in aircraft structures [121]. Additional exemplars include hemp-fiber door liners in automobiles and fiber-based aircraft cabin doors. Modified, twisted hybrids (e.g., Indian mallow/Roselle double-layer longitudinal yarn mats with wood sawdust filler) have been demonstrated for tri-wheel auto hubs, ceiling-fan blades, and side-mirror casings (Figure I.14a–c). Moreover, glass/jute–epoxy hybrids fabricated by Bajpai et al. [1] have been proposed as viable alternatives to industrial safety helmets.

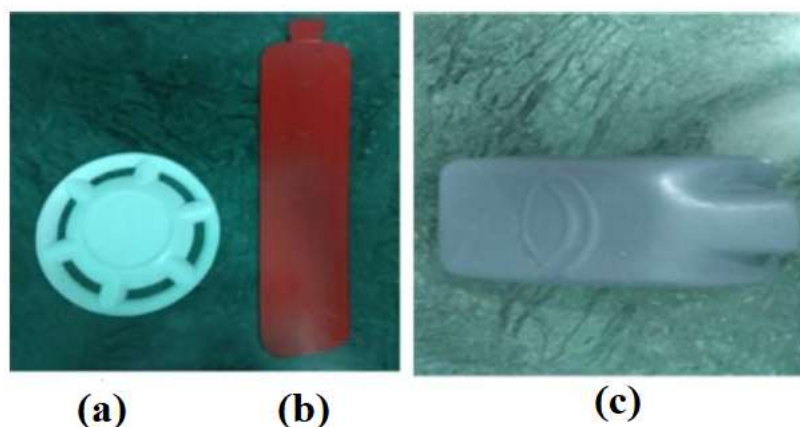


Figure I.14 : (a) Tri-wheel auto-wheel hub, (b) ceiling fan blade and (c) wheeler side mirror casing made from twisted natural FRP hybrid composites [133].

1.12.4 Challenges of natural FRP hybrid composites

The mechanical performance of natural FRP hybrid composites e.g., basalt fiber (BF)/polylactic acid (PLA) systems often remains modest ($\approx 60\text{--}120$ MPa), largely due to weak interfacial bonding in PLA/BF laminates. In parallel, plant-derived cellulosic fibers are hydrophilic (abundant surface OH groups), which promotes moisture uptake, fiber swelling, and consequent degradation of composite properties. These challenges can be addressed through various chemical, biological, and physical surface modifications, including acid/alkali etching, silane or maleated coupling, enzymatic hydrolysis, plasma discharge, and spray coatings. These treatments enhance fibre–matrix adhesion and reduce water absorption [134,135]. Progress also depends on thorough chemical characterization of emerging natural reinforcements and matrices to optimize compatibility and interfacial design in hybrid laminates. Processing presents additional constraints. The relatively low thermal stability of natural fibers means they degrade at elevated temperatures, adversely affecting mechanical behavior an effect that intensifies with higher fiber contents. Accordingly, curing parameters (temperature, pressure) must be selected with the thermal limits of each reinforcement in mind. Fiber breakage during compounding caused by collisions with tooling/molds further reduces effective aspect ratio ; therefore, fiber frictional resistance and gentle handling strategies should inform process design [135]. Lignocellulosic fibers are also susceptible to photo-, bio-, chemical-, and thermally induced changes [131] : while this aids end-of-life degradability, it can limit long-term durability (e.g., in soil contact). Another recurring concern is void formation from trapped air/particulates during fabrication, which degrades mechanical, thermal, and barrier properties; improved resin distribution, degassing, and air-gap removal help to control voids [136]. When paired with appropriate surface treatments, natural FRP hybrids commonly show superior mechanical properties relative to untreated counterparts, owing to improved interfacial load transfer [137]. Thus, these materials are viable for functional and selected structural applications. However, the field still lacks robust predictive models and standardized experimental designs to guide optimal selection of reinforcements (fillers/fibers) and matrices for targeted performance. Advancing predictive analytical modeling would enable cost reductions, lower water uptake, greater strength/toughness, improved environmental sustainability, and enhanced abrasion resistance, among other gains. Collectively, the aforementioned challenges define important research frontiers in composite technology that merit sustained investigation.

I.13 Properties of natural FRP hybrid composites

I.13.1 Mechanical properties of natural FRP hybrid composites

I.13.1.1 Tensile properties

Tensile properties are among the primary targets in the hybridization of natural-fiber FRP composites, chiefly because they can be enhanced by incorporating synthetic fibers. Natural plant fibers typically exhibit lower tensile strength than high-performance reinforcements such as carbon and glass ; consequently, introducing an appropriate fraction of these synthetic fibers into a natural-fiber system generally elevates tensile strength and modulus while retaining a more sustainable material profile. Prior studies on natural synthetic hybrids have therefore focused on optimizing the synthetic fraction to improve the tensile response of natural-fiber composites without unduly compromising environmental benefits. A consolidated comparison of tensile strength versus modulus for natural FRP hybrid composites reported in the literature is provided in Figure I.15.

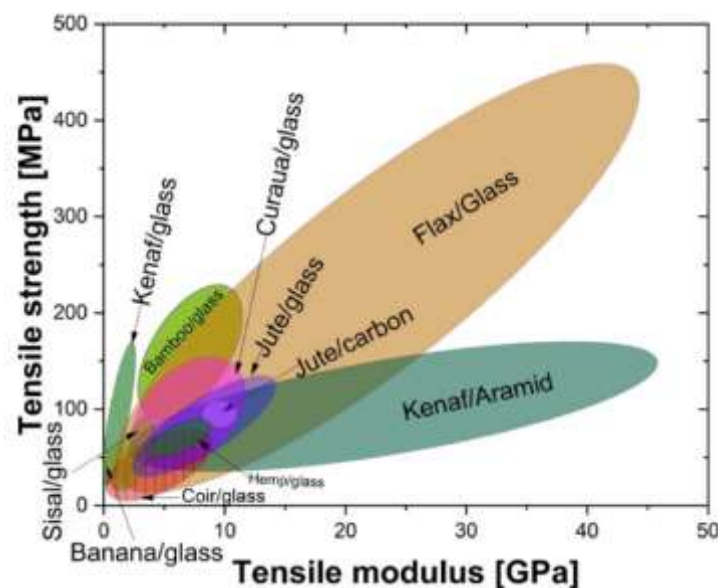


Figure I.15 : Evaluation of tensile characteristics of selected natural/synthetic FRP hybrid composite.

Nevertheless, tensile performance in natural FRP hybrids can deviate positively or negatively from values predicted by simple mixture rules due to synergistic effects within the hybrid system. Understanding these effects is essential for advancing natural-fiber composites to more complex applications. For example, hybridizing banana and coir fibers in polyethylene produced a measurable hybrid effect in both tensile strength and modulus: at equal volume fractions, the hybrid outperformed the single-fiber composites. This synergy is attributed to pairing low-elongation and high-elongation fibers: coir exhibits ~30% tensile elongation, whereas banana exhibits ~10%. Under tensile loading, the lower-elongation banana fibers

initially bear the load and fail first, after which the load is transferred to the higher-elongation coir fibers, which redistribute stresses and elevate tensile strength [138]. A similar but smaller effect was reported for jute/banana hybrids at equal weight fractions in epoxy, where jute (~2.5% elongation) acts as the low-elongation phase and banana (~10%) as the high-elongation phase ; the reduced elongation contrast plausibly accounts for the more modest improvement [139,140]. The mechanistic basis of such hybrid synergy remains incompletely resolved, further complicated by the interplay among fiber fractions, processing parameters, and layup architectures, which are difficult to isolate experimentally [141,142]. Moreover, the substantial design freedom available in hybrid systems increases the complexity of selecting architectures that deliver the desired response.

I.13.1.2 Flexural properties

Flexural strength refers to the maximum bending stress a composite can withstand when subjected to bending forces [143]. This property is influenced by several variables, including fiber material, particle size, filler content, and fiber orientation. A summary of the flexural properties of natural FRP hybrid composites is provided in Figure I.16. A comprehensive comparison of studies examining the impact of hybridization on the flexural performance of natural FRP hybrid composites indicates that outcomes are significantly influenced by factors such as fiber type, fiber modifications, layup strategy, matrix type and modifications, processing parameters, and fiber–matrix adhesion [144–147].

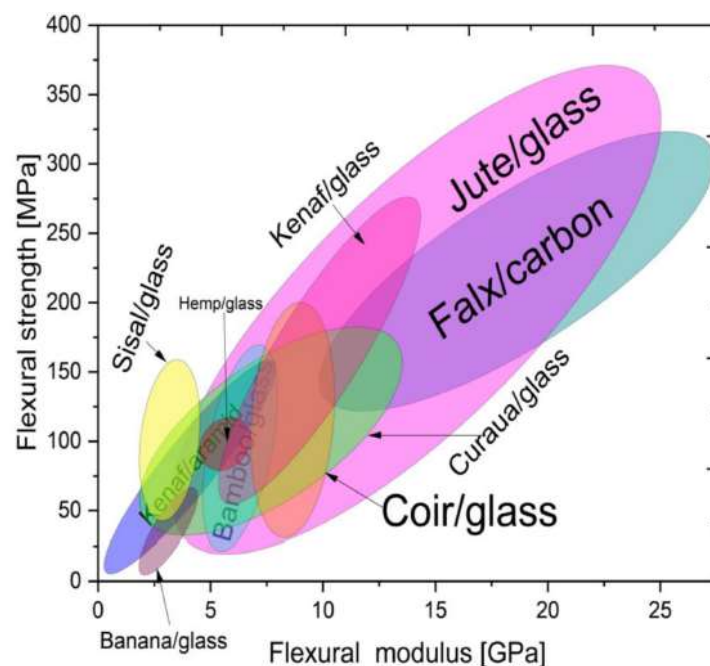


Figure I.16 : Evaluation of flexural responses of selected natural/synthetic FRP hybrid composites.

I.13.2 Fracture toughness of natural FRP hybrid composites

Materials in service must be capable of absorbing a specific amount of energy under load before failure occurs. In fibre-reinforced composites (FRPs), failure typically results from the propagation of cracks or defects within the material, which are often introduced during production, processing (e.g., surface finishing, drilling), and to a lesser extent, under structural loading [148-150]. Fracture toughness describes a material's ability to absorb energy prior to fracture and is directly related to its resistance to crack propagation. It is quantified by the critical intensity factor (K_{Ic}), while resistance to crack growth is assessed using the critical energy release rate (G_c) [151]. These parameters are determined using various methods, which depend on factors like loading mode, material type, composition, and stress conditions at the crack tip. Two prominent approaches in fracture mechanics for material failure analysis are linear elastic fracture mechanics (LEFM) and elastic-plastic fracture mechanics (EPFM). LEFM, which assumes elastic behavior near the crack tip, is most commonly applied to analyze failure in metal and polymer composites. Crack propagation occurs in three modes : Mode I (opening mode, under applied tensile loading), Mode II (shear mode, in-plane shear stress), and Mode III (tearing mode, out-of-plane shear stress). Mixed Mode, involving a combination of Modes I and II, is also considered [152]. Despite the commonality in fracture mechanics approaches, the specific mode and testing method can influence the failure analysis. Natural FRP hybrid composites can either be isotropic (e.g., short, non-woven fiber-reinforced polymers) or laminated (e.g., unidirectional fiber-reinforced laminates). Several methods are employed to determine their fracture behavior, with unidirectional or woven laminates being common in structural applications. The most common mode of fracture in laminated fiber-reinforced composites is delamination at the laminar-laminar interface [154,155]. Crack initiation, growth, and propagation are generally governed by the release of strain energy. Therefore, the delamination process can be characterized by measuring the strain energy released, considering the loading mode on the material. Several test methods are used to determine G_c for laminated composites, such as the double cantilever beam (DCB) test for Mode I, end-notched flexure for Mode II, and end-crack torsion for Mode III. The mathematical principles underlying these tests are well-documented in the literature [156]. Despite advancements, these methods have not yet been fully adapted for accurate natural FRP composite measurement, particularly for woven fiber mats. As natural FRP hybrid composites are increasingly used for structural and semi-structural applications, assessing their in-service functionality especially resistance to crack propagation is becoming critical. The inter-laminar fracture toughness of natural FRP composites has been extensively studied,

particularly using the DCB methodology [157-159]. Generally, the fracture toughness of natural fibre-reinforced polymer (FRP) composites is lower than that of synthetic fibre counterparts. However, various strategies, such as optimized fibre placement, fibre surface treatment, matrix toughening, through-thickness stitching, and the use of hyper-branched polymers, have been shown to enhance fracture toughness. A limited number of studies have specifically investigated the effect of hybridization on the fracture toughness of natural FRP hybrids. For example, hybridizing glass fabric–phenolic resin composites with flax fabric led to improved fracture toughness [160]. Using the double cantilever beam (DCB) method, the hybrid composite demonstrated significantly higher fracture toughness compared to pure glass-fibre composites. Additionally, the inter-laminar shear stress (ILSS) of the hybrid was found to exceed that of both flax and glass composites. These improvements were attributed to factors such as fibre surface roughness, fibre bridging, and entanglement at the flax–glass interface. Similar enhancements were observed in woven flax-glass fibre epoxy hybrids using the same testing method [161]. However, in some cases, hybrid composites (e.g., basalt and flax fibers) showed no improvement in critical energy release rate (tested in Mode I via DCB) compared to pure flax composites, primarily due to the lack of basalt fibers along the central axis of the composites [149]. Additionally, Mode II testing (via three-point end-notched flexure) revealed that hybridized composites had a 58% increase in critical energy release rate (G_{IC}) during initiation and 21% increase during propagation stages compared to their non-hybrid counterparts [150]. The intra-laminar hybridization approach was also studied by Pereira et al. [163], who reported 212% and 191% increases in G_c for woven jute-curaua and sisal-curaua fiber-epoxy hybrids, respectively, compared to pure jute and sisal composites. However, crack propagation was found to be unstable, with secondary cracks forming in the longitudinal direction, likely due to poor fiber-matrix adhesion, and fiber pull-outs. Matrix cracking was identified as the dominant failure mechanism, resulting from ineffective stress transfer at the fiber–matrix interface.

I.14 Hybrid composites of flax fiber

Natural fibers cannot match synthetic fibers in terms of mechanical performance, in sophisticated applications like aerospace, marine, and automotive that demand high performance from fiber-reinforced polymer composites. Flax fibers may be used in these applications while lowering the environmental effect of synthetic fibers due to hybridization. Audibert et al. [164] studied that flax/Kevlar/epoxy hybrid composites showed higher mechanical characteristics than pure flax/epoxy composite but lower than pure Kevlar fiber

reinforced epoxy composite. It showed similar compression weakness as Kevlar composite ; thus, the compression characteristics were lower than the tensile characteristics. Kureemun et al. [165] investigated the hybrid composite composed of PW flax, carbon, and epoxy's tensile and stiffness characteristics. By adding 8% carbon fiber to the composite, they found that the tensile strength could be raised by 50%. The characteristics attained were 30% better than those of aluminum. Additionally, if there is a significant variation in the out-of-plane standard stiffness of the mating fabrics, they drawn the opinion that the dispersion may have an impact on the tensile performance of interlaminar hybrids. Maheshwari et al. [166] investigated the mechanical characteristics of a hybrid composite made of flax, jute, hemp, and epoxy. In comparison to pure flax composite, it was determined that the hybridization greatly enhanced the tensile, flexural, and impact characteristics. In contrast to the pure fiber reinforced epoxy composites, the hybrid exhibited a lower level of hardness. FVF and stacking sequence are two crucial factors that influence the mechanical characteristics of hybrid composites. The order of the fiber stacking had less of an impact on the hybrid composites' tensile characteristics. However other characteristics, like flexural, provide a sound effect [167]. In hybrid composites, the amalgamation of the synthetic fiber layer as a skin might result in the best flexural characteristics [168]. Selver et al [167] studied an effect of different stacking sequences on tensile and flexural performance of canvas-woven (CW) flax/PW glass/epoxy hybrid composites. The stacking order has no discernible effect on the tensile characteristics. Glass fiber was used as a skin layer in the hybrid composite to increase its flexural strength. Zhang et al. [169] found a similar finding, indicating that the amount of glass fiber in the hybrid composite affected its tensile capabilities. This was enhanced by the addition of FVF. While the stacking sequence of the flax and glass fiber reinforced phenolic hybrid composite showed no influence on the tensile modulus, it did dramatically increase the tensile strength. Higher tensile and flexural characteristics are obtained in flax/carbon/epoxy hybrid composites (CFFFC and FCFCF) when carbon is utilized as a skin layer. [170] Abd et al. [171] investigated the mechanical characteristics of ternary hybrid composites composed of epoxy, glass, flax, and basalt. They discovered that the composites' tensile and flexural characteristics were significantly impacted by the stacking order. Fiore et al. [168] investigated the Epoxy hybrid composites' tensile and flexural strengths using twill weave flax and UD carbon fibers. When subjected to flexural loading, flax BD150 outperformed flax BD220 in the hybrid composite ; however, tensile loading produced the reverse effect. The hybrid epoxy composite made of flax BD150 and carbon fiber reinforced epoxy demonstrated strong tensile and flexural strength (288 and 160.4 MPa), which justifies it for use in

structural applications. The final composite characteristics are greatly impacted when additional natural fibers are synthesized with fiber reinforced thermoset composites. Because bamboo has fewer mechanical qualities than flax fiber, the thermoset composite showed a negative result from the hybridization of bamboo and flax fiber [172]. Therefore, using flax fiber in thermoset polymer composites with enhanced mechanical characteristics is the only way to reap the benefits of hybridization. But there are other benefits as well, including reduced density. Chandrasekar et al. [173] also discovered a similar result. They investigated how sugar palm fiber affected the flax/epoxy composite. In comparison to flax/epoxy composite, all evaluated hybrids showed worse tensile, flexural, and short beam performance.

I.15 Effect of environmental conditions on structure and properties of natural FRP hybrid composites

The structural application of natural FRP composites is constrained due to their limited environmental resilience, primarily stemming from the hydrophilic nature of natural fibers, which leads to moisture uptake and consequent reduction in mechanical performance [174]. This section examines the challenges associated with moisture absorption and exposure to diverse environmental conditions, and their effects on the mechanical, thermal, and physical properties of natural FRP composites.

I.15.1 Degradation in natural FRP composites under the influence of UV radiation

Exposure to ultraviolet (UV) radiation, moisture, and high humidity significantly influences the performance and degradation behavior of natural FRP composite structures. UV rays, which are electromagnetic radiation naturally generated by the sun, affect materials in various ways. The immediate consequence of UV exposure is surface discoloration, resulting from photo-degradation reactions. These reactions not only lead to the breakdown of chemical bonds within the polymer matrix but also induce physical changes, such as micro-cracking. At elevated temperatures, the degradation process can be further accelerated [175]. For natural FRP composites, UV exposure poses a substantial concern as it can drastically alter their durability, mechanical performance, and overall stability, making it a critical challenge for outdoor applications. Studies have shown that UV irradiation induces surface oxidation in these composites, which in turn generates thermal and mechanical stresses on both the surface and internal layers of the material. This results in stress concentration and shrinkage, which negatively impacts the composite's overall performance [176].

I.16 Rupture mechanisms and failure modes of biocomposites

The rupture and failure of biocomposites pose significant challenges to their widespread adoption in engineering and sustainable material applications. The combination of natural fibers and polymer matrices results in complex failure mechanisms, including fiber breakage, fiber–matrix debonding, progressive cracking, and, in some cases, delamination. These mechanisms are heavily influenced by factors such as fiber type, surface treatment, moisture content, and environmental conditions. Previous studies have emphasized the increased sensitivity of biocomposites to hygrothermal and mechanical aging, which accelerate damage accumulation and shorten the service life of the material [177,178]. Current research approaches to studying failure combine experimental testing (e.g., tensile, flexural, impact) with multi-scale modeling to predict durability and optimize formulations. As such, understanding failure modes remains a central area of research for ensuring the reliability and industrial applicability of biocomposites in structural applications.

I.17 Conclusion

This chapter has thoroughly explored the field of natural fiber-based composites, with a particular focus on alfa fibers and their role in the production of natural fiber-reinforced polymer composites (NFRPCs). We have discussed the entire manufacturing process and the factors influencing the mechanical performance of these composites, highlighting both their advantages and drawbacks. The applications of natural fiber composites, especially in various sectors, have also been reviewed, with attention given to challenges such as material degradation under UV radiation. The hybridization of natural fiber composites, along with the study of the mechanical properties of hybrid composites, represents a promising avenue to enhance their performance and expand their range of applications. However, it is crucial to continue exploring innovative treatment and manufacturing methods to overcome the limitations of natural fibers and optimize their integration into durable and high-performance composite materials. In summary, this bibliography emphasizes the growing significance of natural fiber-based composites, particularly those reinforced with alfa fibers, in the context of an industry striving for more sustainable and eco-friendly solutions. Continued research in this field is expected to broaden their use and optimize their performance while minimizing environmental impacts.

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Chapter I

References

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Chapter II

Experimental study of mechanical behavior of Polyester/Alfa composite

Chapter II outlines the materials and experimental methods used to develop and test polyester/alfa fiber composites. It describes the preparation and treatment of alfa fibers, their morphological analysis, and the composite fabrication process. Details on mold manufacturing, natural aging, and sample machining are provided. Finally, mechanical tests including flexural and toughness tests are conducted, followed by discussion of the results.

Chapter II : Experimental study of mechanical behavior of Polyester/Alfa composite

This chapter provides a comprehensive description of the experimental procedures implemented for the preparation, characterization, and mechanical analysis of polyester matrix composites reinforced with Alfa fibers. It begins by outlining the various fiber treatment stages, including washing, drying, grinding, sieving, and alkaline treatment, all aimed at enhancing the fibers' compatibility with the polymer matrix. The fabrication of molds and testing supports, as well as the specimen preparation techniques, are also detailed. Subsequently, the chapter presents the mechanical testing protocols, including three-point bending tests and fracture toughness tests (SENB), conducted in accordance with relevant ASTM standards. These tests were performed on both unaged composites and those subjected to three months of natural aging in a Mediterranean climate. In addition, morphological analyses of Alfa fibers were performed using an optical microscope to compare the structural features of coarse and fine fibers, and to relate these differences to their mechanical performance. The results and discussion section highlights the influence of fiber size, fiber content, and environmental aging on the overall mechanical behavior of the composites, offering insights into their potential application in bio-based and structural composite materials.

II.1 Materials

II.1.1 Matrix

In this study, the polymer matrix used is a polyester resin combined with a hardener (Figure II.1). This thermosetting system is widely employed in the manufacturing of composite materials due to its good mechanical properties, low cost, and ease of processing. Polyester resin is an unsaturated polymer which, when mixed with a hardener (typically based on methyl ethyl ketone peroxide), undergoes an exothermic polymerization reaction, resulting in a rigid and solid material. In this study, the polyester resin serves as the matrix, meaning the continuous phase of the composite, ensuring cohesion of the alfa fibers and transferring loads between them. The hardener is essential for initiating the chemical curing of the resin, thus enabling the formation of a final composite material with enhanced mechanical performance.



Figure II.1: Constituents of the matrix (resin and hardener).

II.1.2 Fibers

In October 2020, the Alfa fibers used in this study were collected from the Bougtob region, located in the El Bayadh Province in the southwestern part of Algeria (Figure II.2). This region is known for its natural abundance of *Stipa tenacissima* L. (Alfa grass), which offers desirable properties for use as a natural reinforcement in composite materials.



Figure II.2: Alfa (*Stipa tenacissima*) in natural conditions.

II.2 Preparation of Alfa fibers

II.2.1 Fiber Pre-Treatment

Firstly, the Alfa fibers were thoroughly washed with tap water to remove any surface impurities or contaminants. Following the cleaning process, the fibers were sun-dried for four days. This natural drying method helped to eliminate most of the residual moisture within the fibers, thereby facilitating the subsequent crushing process by making the fibers drier and easier to handle.

II.2.2 Oven Drying of Alfa Fibers

After the initial air-drying stage, the Alfa fibers were transferred to a vacuum oven to eliminate any remaining moisture in a controlled and efficient manner (Figure II.3). This step is essential to ensure an optimal reduction in water content while avoiding the risk of thermal degradation of the fibers. The drying was conducted at a temperature of 75 °C for a duration of 4 hours, providing uniformly dried fibers suitable for further processing.



Figure II.3: oven for drying Alfa fibers.

II.2.3 Mechanical Preparation and Grinding of Alfa Stems

To facilitate processing and promote fiber individualization, the Alfa stems were initially pre-cut into small segments measuring approximately 3 to 5 cm in length. This preliminary step is essential to optimize the mechanical grinding process and ensure uniform fragmentation of the fibers. The grinding was performed using a blade crusher, a machine equipped with high-speed rotating blades that efficiently reduce the size of plant material (Figure II.4). The intense shear forces generated during this process help to break down the lignocellulosic structure of the Alfa stems, enabling the effective separation of individual fibers. This mechanical treatment significantly increases the specific surface area of the fibers, enhancing their interaction potential with matrices in further applications such as in composite reinforcement or chemical surface modification. As such, the grinding process is a critical step in the valorization of natural fibers, ensuring homogeneous fragmentation while preserving the mechanical integrity and structural properties of the material.



Figure II.4: blade crusher.

II.2.4 Sieving and Morphological Classification of Alfa Fibers

Subsequent to the mechanical grinding stage, the Alfa fibers were subjected to a sieving process aimed at classifying them based on their diameter distribution (Figure II.5). This granulometric separation facilitates a more targeted use of fiber fractions in accordance with their morphological and mechanical properties, thereby enhancing the reproducibility and performance of subsequent applications. The sieving process yielded two distinct fiber populations :

- Fine fibers, with diameters ranging between 15 and 20 μm , exhibit greater flexibility and surface area, making them particularly suitable for applications requiring microstructural reinforcement, uniform dispersion, or smooth surface finishes.
- Coarse fibers, with diameters ranging from 70 to 80 μm , are characterised by their higher rigidity and load-bearing capacity, thus rendering them appropriate for use in applications demanding enhanced mechanical resistance, such as structural composites or bio-based engineering materials.



Figure II.5: Sieve.

II.2.5 Alkaline Treatment of Alfa Fibers

To improve the interfacial compatibility between Alfa fibers and polymer matrices, an alkaline treatment was conducted using sodium hydroxide (NaOH) (Figure II.6). The crushed fibers were immersed in a 5 wt% NaOH solution for 48 hours. This chemical treatment is known to remove surface impurities, dissolve lignin and hemicellulose, and increase surface roughness, thereby improving the fiber–matrix adhesion in composite materials. Following the immersion, the fibers were thoroughly rinsed with fresh water to eliminate any residual alkali. The treated fibers were then air-dried at room temperature for 48 hours prior to their use in further processing steps.



Figure II.6: Sodium Hydroxide.

II.3 Morphological Analysis of Alfa Fibers via Optical Microscopy

The characterization of natural fibers constitutes a fundamental step in evaluating their morphological, physical, and mechanical properties, which are critical for their integration into industrial and scientific applications. In this study, optical microscopy was employed to examine and document the structural features of both coarse and fine Alfa fibers. High-resolution optical micrographs were obtained using an OPTIKA HDMI Easy-4083.13E optical microscope. This instrument enabled the detailed observation of fiber surface texture, diameter variability, and general morphological integrity, providing valuable insights into the fiber quality and suitability for downstream processing, such as reinforcement in polymer composites or surface functionalization.

II.3.1 Microscopic Equipment and Imaging Capabilities

The OPTIKA HDMI Easy-4083.13E optical microscope is a high-performance optical analysis instrument equipped with an integrated digital camera, allowing for the acquisition of high-resolution micrographs (Figure II.7). Designed for precision imaging, this device offers excellent optical clarity and is particularly well-suited for the observation of microscopic structures in natural fibers and composite materials. The integrated system enables real-time visualization of samples, with images displayed directly on a computer or external monitor, thereby facilitating morphological analysis, documentation, and digital data archiving. Its robust imaging capabilities make it an effective tool for quantitative and qualitative assessment in material science studies.

II.3.2 Observation Methodology

The Alfa fiber samples were carefully prepared by placing them onto clean glass slides to ensure optimal positioning and stability during analysis. Observations were conducted using transmitted light illumination, which enhanced the contrast and visibility of the fibers' internal structures. A range of objective lenses available on the OPTIKA microscope was employed to achieve multi-scale magnification, enabling the detailed examination of surface texture, fiber diameter, and morphological characteristics. This methodology allowed for a comprehensive qualitative assessment of the fiber structure, contributing to a better understanding of their potential performance in material applications.

II.3.3 Morphological Comparison of Alfa Fibers

The obtained micrographs reveal significant differences between coarse and fine Alfa fibers (Figure II.8) :

-
- Coarse fibers: These exhibit a thicker and less homogeneous structure, with an irregular surface that may indicate the presence of impurities or structural defects.
 - Fine fibers: These appear more uniform and homogeneous, with better structural cohesion. These characteristics can influence the mechanical properties of the fibers and their potential use in composites or other industrial applications.

The use of the OPTIKA HDMI Easy-4083. 13E optical microscope allows for a detailed analysis of Alfa fibers, highlighting their morphological differences. This study is crucial for better understanding their structure and properties, which can be beneficial in fields such as biomaterials, textile engineering, and composite materials.



Figure II.7: OPTIKA optical microscope, model: HDMI Easy – 4083. 13E.

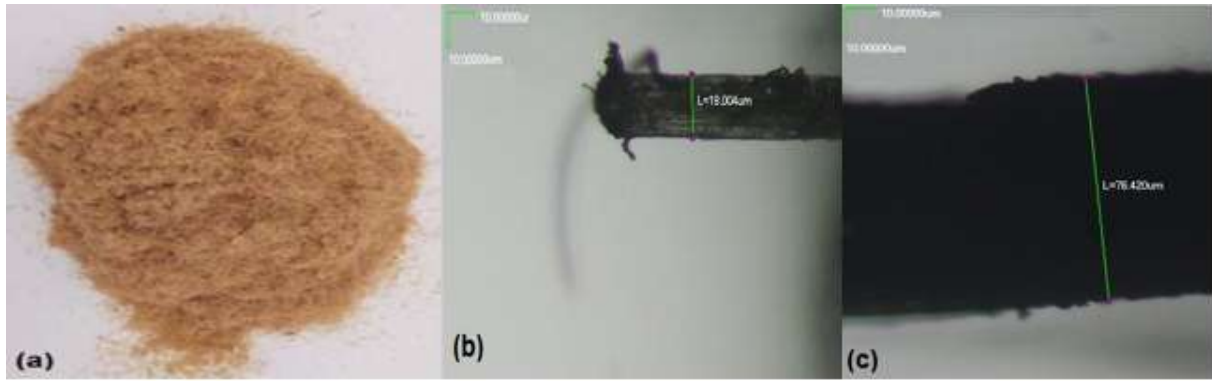


Figure II.8: Alfa fibers: (a) Crushed Alfa fibers, (b) Fine fibers, (c) Coarse fibers.

II.4 Manufacturing of Molds for Mechanical Testing

The mechanical test molds were manufactured at a gas bottle production facility located in Mascara (Algeria) (Figure II.9). These molds were specifically designed and fabricated to produce specimens conforming to standardized mechanical testing dimensions and requirements. The manufacturing process began with the selection of aluminum as the mold material, owing to its favorable mechanical strength, dimensional stability, and resistance to corrosion. Aluminum blocks were cut, milled, and drilled using precision machining techniques to create molding cavities corresponding to the desired specimen geometry. Special attention was given to the internal surface finish of the molds to ensure smoothness and uniformity, thereby enabling easy demolding and consistent specimen shape. The slightly textured surface observed on the final molds is a typical result of conventional machining processes and could be improved through post-processing operations, such as mechanical polishing, if required. A final dimensional inspection was carried out to verify that all molds conformed to the specified technical tolerances, ensuring their functional adequacy for the preparation of samples intended for flexural and other mechanical tests (Figures II.10 and II.11).



Figure II.9: Workshop Workstation: CNC Milling Machine for Industrial Manufacturing.



Figure II.10: Mold for three-point bending specimen.



Figure II.11: Mold for toughness specimen.

II.5 Preparation of polyester /alfa composite

As reinforcing fillers, two types of 5% alkali-treated Alfa fibers (coarse and fine sizes) were utilized. The Alfa fibers were vacuum-dried at 75°C for four hours to eliminate absorbed moisture and prevent void formation before the extrusion process. The first step in producing

complex composite specimens involved combining Alfa fibers with polyester resin (UPR) at four distinct fiber weight fractions (0, 10, 20, and 30 wt%). The resulting polyester resin/Alfa fiber mixture was then injected into a mold to create normalized flexural bending specimens. The dimensions of these bending specimens were 140 x 20 x 4.7 mm (Figure II.12). The specimens prepared for the toughness test are shown in Figure II.13.



Figure II.12: polyester/alfa composite specimens for bending test.

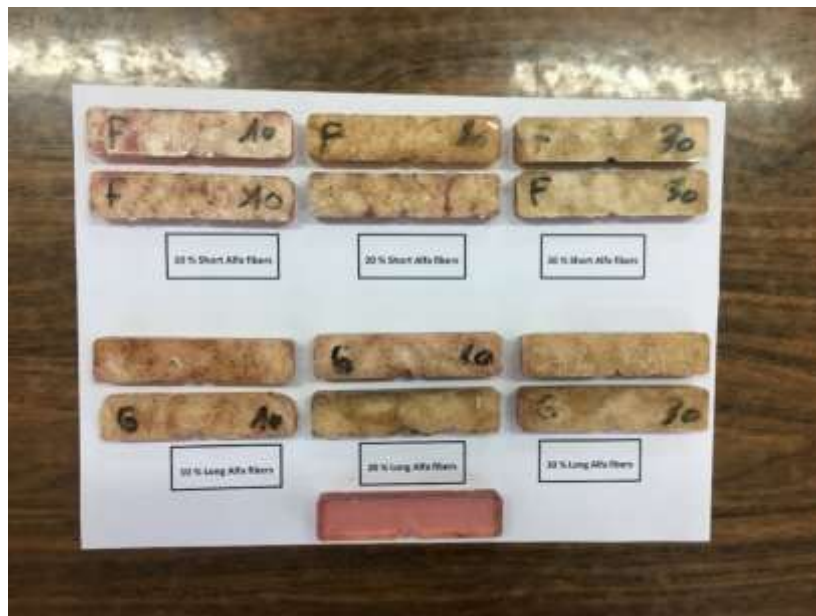


Figure II.13: different configurations of the Alfa polyester composite for the toughness test.

II.6 Natural Aging Conditions

To simulate real environmental exposure, the Mediterranean climate was taken into consideration, as it encompasses key aging parameters for Alfa/polyester composites, namely sunlight, ambient air, and relative humidity (RH). The natural aging experiment commenced on 23 July 2022 and lasted for a total duration of three months, concluding on 23 October 2022. The study was conducted in Mascara City, located in northern Algeria, a region characterized by significant diurnal and seasonal variations in climatic conditions. During the

exposure period, ambient temperatures fluctuated from approximately 15 °C in the early morning to peaks of 45 °C around 1:00 p.m.. Additionally, relative humidity levels ranged from 3% to 65%, reflecting the natural variability of the atmospheric conditions. These environmental factors play a critical role in the physical, chemical, and mechanical degradation mechanisms affecting natural fiber composites exposed to outdoor conditions.

II.7 Machining of Supports for the Three-Point Bending Test

The fabrication of supports used in the three-point bending test was conducted at the Mechanical Engineering Workshop of Mustapha Stambouli University in Mascara (Figures II.14 and II.15). The operation was performed using a conventional milling machine equipped with a Digital Readout (DRO) system, enabling precise control over the machining dimensions and tolerances. Raw aluminum blocks were selected as the base material due to their mechanical robustness, dimensional stability, and resistance to corrosion. These blocks were securely fixed in a vise to ensure positional stability during the machining process. A carbide end mill was employed to perform the material removal, ensuring both a smooth surface finish and dimensional accuracy in accordance with test standards. The presence of metal chips observed during the operation reflects the gradual material removal process aimed at achieving the required geometry. The use of the DRO system allowed for precise control of cutting depth and spatial positioning, ensuring that the final supports met the specifications necessary for reliable and reproducible mechanical testing. After machining, a dimensional verification was carried out to confirm compliance with the mechanical testing standards, thus guaranteeing the mechanical integrity and accuracy of the supports used during the bending tests.



Figure II.14 : cutting of rectangular and round profiles.



Figure II.15 : Manufacturing of Test Supports : Milling Operation with Digital Control.

II.8 Mechanical Testing

II.8.1 Preparation and Calibration of the Flexural Testing Machine

The preparation of a flexural testing machine, as presented in this study, is crucial to ensure the accuracy and reliability of experimental results. Before starting the test, it is essential to thoroughly verify the machine's setup, ensuring that all frame and support components are properly assembled and secured. This step aims to ensure that the equipment is stable and free from any defects that could compromise the quality of the tests. Once the machine is installed, adjusting the supports is a key step, which must be done according to the dimensions and

specific properties of the sample to be tested. It is critical that the sample is positioned and centered correctly on the supports to avoid any deformation or measurement errors that may result from misalignment. The choice of testing configuration, such as the type of flexure (three-point or four-point bending), should be made based on normative specifications and the study's objectives. The loading speed and test duration must also be adjusted according to the material and the desired test type. Before proceeding with the test itself, the machine must be precisely calibrated to ensure reliable measurements of applied forces and displacements. This calibration involves adjusting the force sensors and displacement sensors, ensuring their accuracy and proper synchronization. Once these preparations are completed, the sample is placed on the machine, and the test can begin, gradually applying the load to the sample. The computer system, connected to the machine, allows for real-time monitoring of the forces and deformations, while collecting the data necessary for analysis. This procedure ensures that the flexural test is conducted under optimal conditions, providing accurate and reproducible results.

II.8.2 Three-Point Bending Test Setup

The three-point bending tests were performed using a BED 100 universal tensile testing machine, which has a maximum load capacity of 1 kN. The device is equipped with a test speed control system, allowing precise regulation of the crosshead displacement rate in accordance with standard testing protocols. This configuration ensures consistent loading conditions and accurate measurement of flexural properties such as stress, strain, and modulus. The complete test setup is illustrated in Figure II.16, which shows the placement of the specimen on the supports and the centrally applied loading nose.



Figure II.16: Bend testing machine.

All three-point bending tests were performed at a crosshead speed of 6 mm/min, in accordance with the ASTM D790 standard [1], which governs the flexural testing of plastic and composite materials.

The specimens tested included:

- Neat (non-reinforced) polyester, and
- Polyester reinforced with Alfa fibers at three different weight fractions : 10%, 20%, and 30%.

Each composite formulation was evaluated under two distinct environmental conditions :

1. As-prepared (unaged) samples, and
2. Naturally aged samples, following 3 months of outdoor exposure in a Mediterranean climate.

Furthermore, for each fiber content, two fiber size categories were studied: coarse fibers and fine fibers, both of which had undergone alkaline treatment (5 wt% NaOH).

The flexural specimens had the following dimensions :

- Length: 140 mm
- Width: 20 mm
- Thickness: 4 mm
- Span length (distance between supports): 110 mm

The complete configuration of the flexural test setup is illustrated in Figure II.17.

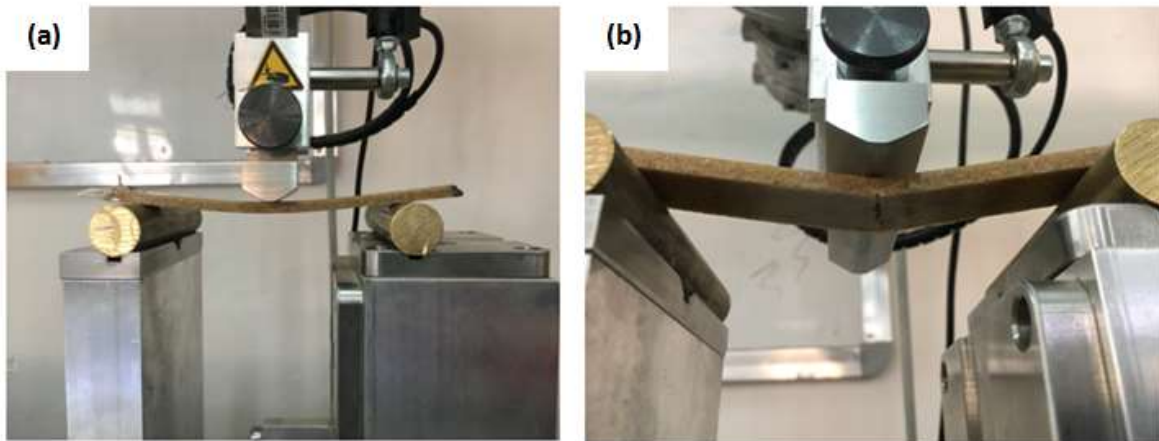


Figure II.17: Sequence of the experimental 3 points bending test until failure: (a) Initial state, (b) Final state.

In a three-point bending test for rectangular specimens, the resulting flexural stress and strain can be calculated using the following equations:

$$\sigma = \frac{3FL}{2bd^2} \quad (\text{II. 1})$$

Where:

- F is the applied load [N]
- L is the support span length [mm]
- b is the specimen width [mm]
- d is the specimen thickness [mm]

$$\varepsilon = \frac{6Dd}{L^2} \quad (\text{II. 2})$$

Where:

- D is the maximum deflection at the midspan [mm]

To describe the deflection behavior of the beam, the Timoshenko beam theory provides the following approximation :

$$w = \frac{Fl^3}{4Ebh^3} = \frac{Fl^3}{48EI} \quad (\text{II. 3})$$

Where:

- E is the Young's modulus [MPa or GPa]
- h is the beam height (equivalent to thickness d)
- I is the second moment of area $I = \frac{bd^3}{12}$

Equation 4 provides the resultant Young's modulus:

$$E = \frac{dF}{dw} \cdot \frac{l^3}{48I} \quad (\text{II. 4})$$

II.8.3 Toughness Testing via Single-Edge Notched Bending (SENB)

The toughness of the polyester and polyester/Alfa fiber composites was evaluated using the Single-Edge Notched Bending (SENB) method, in accordance with the ASTM D5045 standard [2]. This technique is designed to assess the fracture toughness of polymeric materials under mode I loading.

The tests were conducted using a three-point bending configuration on the BED 100 universal testing machine, previously used for flexural testing. The test speed was set at 0.2 mm/s, ensuring quasi-static loading conditions. The geometry of the SENB specimens is illustrated in Figure II.18, with the following dimensions :

- Length (L) : 80 mm (corresponding to 4w)

- Width (W) : 20 mm
- Thickness (B) : 10 mm
- Notch width (c) : 2 mm
- Notch depth (b) : 4 mm
- Span between supports : as defined in the standard (not specified here but typically 4×W)

The specimens tested included both neat polyester and polyester composites reinforced with 10%, 20%, and 30% Alfa fibers. For each composition, two fiber size categories (coarse and fine) were used. The mechanical behavior was evaluated under two conditions :

1. Unaged condition (as-prepared), and
2. After 3 months of natural aging in a Mediterranean environment.

The complete test setup is shown in Figure II.19.

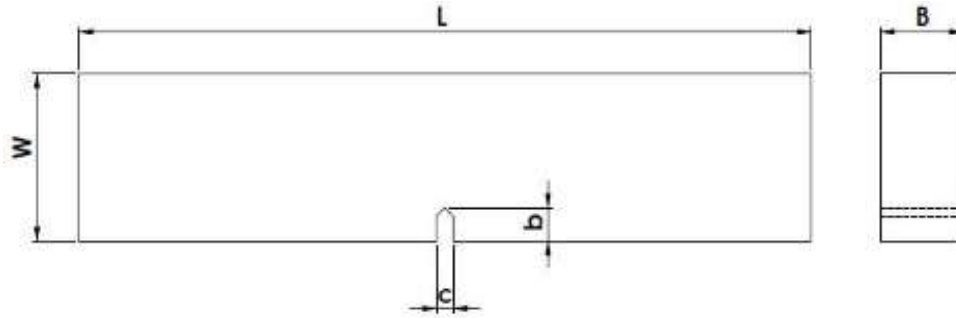


Figure II.18: Single edge notched bend (SENB) specimen.



Figure II.19: Composite specimens SENB during three-point bending test.

The critical stress intensity factor K_{IC} for Mode I fracture was calculated using the following standard formulation, as specified by ASTM D5045 :

$$K_{IC} = \frac{L.P_c}{b.\omega^{3/2}} \cdot f(\alpha) \quad (II.5)$$

Where:

- P_c is the maximum load applied to the specimen [N],

- L is the span length between the supports [mm],
- b is the thickness of the specimen [mm],
- ω is the width of the specimen [mm],
- $\alpha = \frac{\omega}{\alpha}$ the relative notch depth,
- $f(\alpha)$ is a dimensionless geometry correction factor.

The function $f(\alpha)$ is defined as follows:

II.9. Results and discussion

II.9.1 Three-point bending test

From the bending tests, stress-strain curves were plotted for polyester/Alfa composites reinforced with various fiber fractions, considering both fine and coarse Alfa fiber sizes. The mechanical properties of the composite, including Young's modulus, ultimate stress, and ultimate strain, were derived from these curves. These curves provide essential insight into the mechanical behaviour of the composites under flexural loading. In particular, they allow for a comparative analysis between the fine and coarse Alfa fiber reinforcements, highlighting how fiber size and content influence stiffness and strength. The determination of Young's modulus gives an indication of the rigidity of the material, while the ultimate stress and strain values reflect its strength and deformation capacity before failure. Such parameters are critical for assessing the suitability of natural fiber-reinforced composites in structural applications. Moreover, these findings align with previous studies that emphasize the influence of fiber characteristics and volume fraction on the mechanical performance of polymer composites.

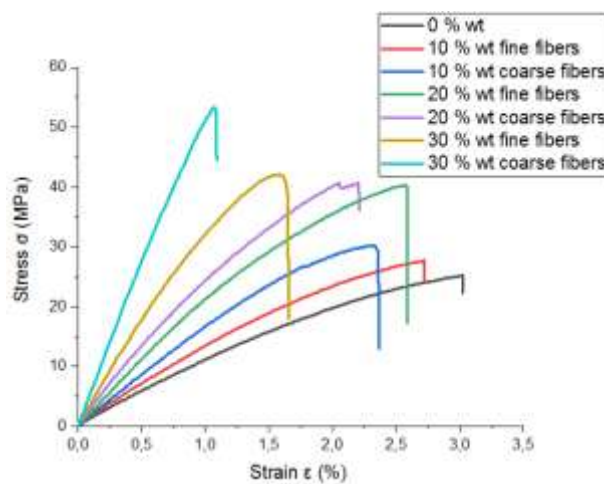


Figure II .20: Stress-strain polyester/Alfa composite with different fractions of reinforcement (0, 10, 20, 30 wt%) for both sizes of Alfa fibers (fine and coarse).

Figure II.20 presents the stress-strain curves for the bending response of polyester resin reinforced with varying fractions of Alfa fibers (0, 10, 20, and 30 wt%). The curve for pure polyester resin serves as a reference for comparison. As found in this figure, the mechanical resistance of polyester considerably increased with increasing the volume of reinforcement in the composite. This trend clearly indicates the reinforcing effect of Alfa fibers in enhancing the structural performance of the polyester matrix. Some found the addition of the Alfa fibers in polyester resin increased its mechanical strength [3,4]. However, the failure strength of resin is extremely sensitive to the bending stress. This sensitivity highlights the critical role of fiber reinforcement in delaying failure under flexural loads. It is also evident that the effect of coarse Alfa fibers is more pronounced than that of fine fibers on the strength of the polyester/Alfa composite. The ultimate stress of the non-reinforced polyester is approximately 25 MPa, while the inclusion of 30 wt% coarse Alfa fibers increases this value by 110%, compared to a 68% increase for the fine Alfa fibers. These findings suggest that fiber morphology, particularly size and surface area, plays a crucial role in stress transfer efficiency between the matrix and the reinforcement. This augment of bending strength confirms that the coarse fibers act as a better reinforcement. The improved performance of composites with coarse fibers could be attributed to their ability to provide better interfacial bonding and mechanical interlocking. This significant increase can be attributed to the increase in stiffness of the composite due to the reduction in mobility of polymer chains. As the amount of fiber in composites made from Alfa fibers grows, so does their flexural strength. This confirms the positive correlation between fiber content and load-bearing capacity under flexion. The enhanced strength of the polyester/Alfa composite may be attributed to the superior mechanical properties of the Alfa reinforcement. These natural fibers offer an eco-friendly and low-cost alternative to synthetic fibers, without compromising mechanical performance. In a similar study, Ben Brahim and Ben Cheikh investigated the influence of Alfa fiber orientation and fiber volume fraction on composite mechanical properties [4]. However, the reinforcement results in a decrease in the ultimate strain of the polyester/Alfa composite, with the magnitude of this reduction being influenced by both fiber content and fiber size. The ultimate strain of pure polyester is 3.2%, which drops to 1.6% for the composite reinforced with 30 wt% fine Alfa fibers, and further to 1% for the composite reinforced with 30 wt% coarse Alfa fibers. This reduction in ductility indicates a transition from a more plastic to a more brittle behaviour with increasing fiber content. This reduction in ultimate strain is primarily attributed to the high stiffness of the Alfa fibers, which results in a decrease in the plasticity of the polyester matrix. While the strength of the composite increases, the reduced

strain at break may limit the applicability of these composites in applications that require high flexibility.

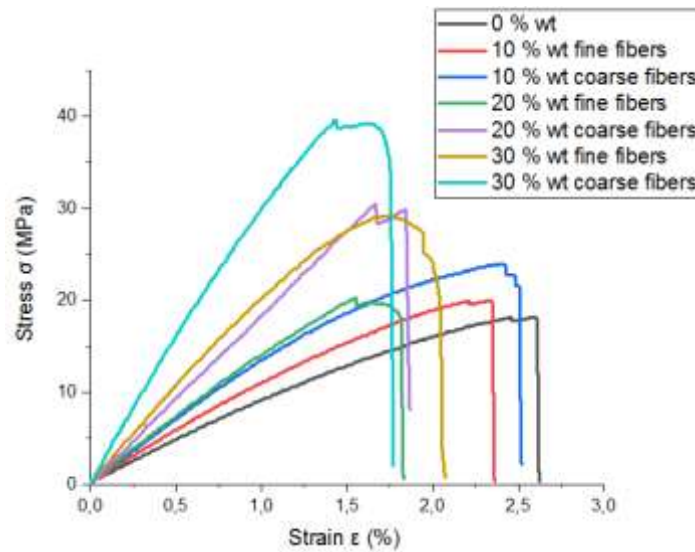


Figure II. 21: Effect of three months atmospheric ageing on the mechanical behaviour of the different categories of the polyester/Alfa composite.

The stress-strain curves for different polyester/Alfa composite and pure polyester affected by atmospheric aging were shown in Figure II.21. For the comparison of ultimate stress and strain of the different polyester/Alfa composite categories before and after atmospheric aging conditions. These curves provide a clear visual representation of how environmental exposure alters the mechanical performance of the materials. Relative to non-affected composite by atmospheric aging, the reduction of the ultimate stress lies between 25% and 45%. This decline demonstrates the vulnerability of natural fiber-reinforced composites to external environmental conditions, particularly humidity and temperature fluctuations. We note that the ultimate stress of the composite reinforced with 30 wt% of coarse and fine Alfa fibers are about 54 MPa and 42 MPa, respectively. Therefore, the values were reduced to 41 MPa and 29 MPa after atmospheric aging (Figure II.22). This degradation highlights a significant loss in structural integrity, especially in the fine-fiber composites, indicating a higher sensitivity to aging. Subsequently, thermal and humidity aging are considered as the major causes of long-term failure of composites exposed to the atmosphere. These forms of aging contribute not only to a reduction in strength but also to the weakening of the internal cohesion of the material system. Atmospheric aging leads to several consequences, including the degradation of the matrix's mechanical properties, differential swelling due to concentration gradients, and damage at the matrix/reinforcement interface [5]. The loss of mechanical performance can thus be linked to both chemical and physical changes occurring at the microscale within the

composite. When composites are exposed to various environmental conditions, particularly moisture, the cellulosic fibres undergo swelling. This hygroscopic nature of Alfa fibers becomes a critical factor in composite durability under outdoor exposure. Consequently, the shear stresses will rise at the fiber/matrix interface. Thereby, the debonding risk of the interface augment. This interface weakening reduces the load transfer efficiency and may lead to premature failure under mechanical loads. Joseph et al. found that the composites after water absorption induced the increase of the mechanical properties' degradation of natural fibers [6]. Such observations confirm that moisture absorption is one of the key factors compromising the mechanical stability of natural fiber composites. In addition, the diminution of thermal stability can be led to the degradation of polymer, fibers and polymer/fiber interface loosening [7]. These thermal effects contribute to long-term degradation, accelerating crack formation and the loss of adhesion between the constituents.

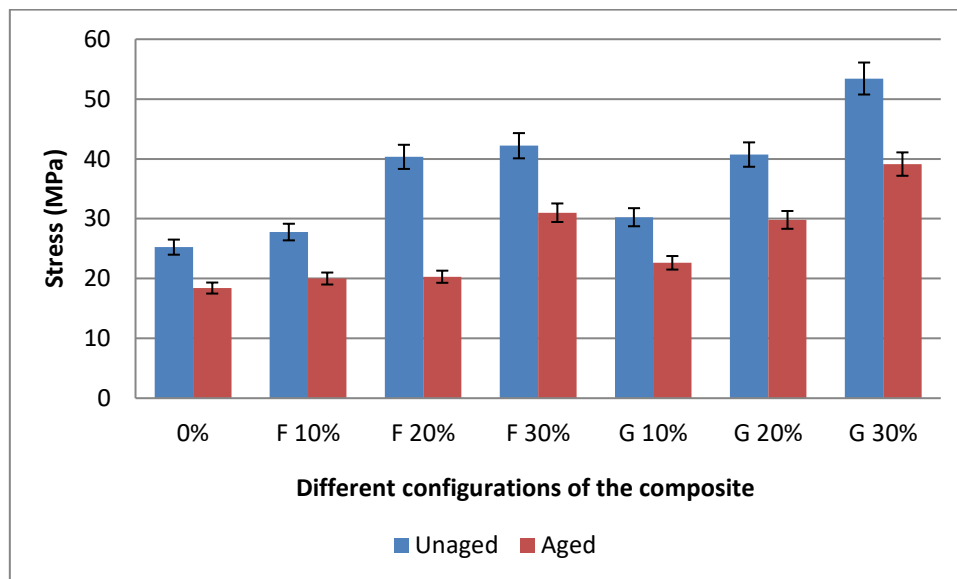


Figure II. 22: Effect of three months atmospheric ageing on the bending strength of the polyester/Alfa composite with different fractions of reinforcement (0, 10, 20, 30 wt %) for both sizes of Alfa fibers (fine and coarse).

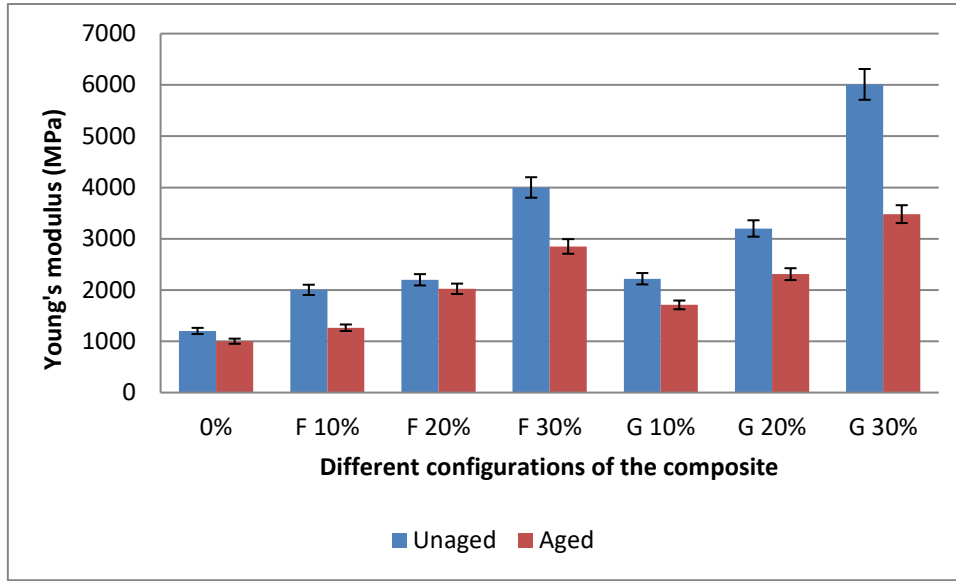


Figure II. 23: Young's modulus variation of different Polyester/Alfa composites
(Coarse and fine size).

Figure II.23 illustrates the evolution of Young's modulus of unaged and aged composites with respect to fiber content. As shown in this figure, an important Young's modulus increase with fiber addition was observed for all composite types. This trend indicates a notable improvement in stiffness, confirming the reinforcing effect of Alfa fibers on the polyester matrix. This significant increase can be attributed to lower porosity and an indication of a strong interphase (Figure II.24). A well-bonded interphase between the fiber and the matrix allows for more efficient stress transfer, which directly contributes to the improved rigidity of the composite. Cavities whose genesis is connected to structural faults begin in the amorphous phase when the material is mechanically loaded [8]. These microstructural defects, often initiated under mechanical stress, may compromise long-term mechanical performance, especially if the interfacial bonding is weak. However, the reduction of porosity observed with fiber reinforcement suggests a denser and more homogeneous internal structure, which plays a positive role in enhancing the mechanical properties.

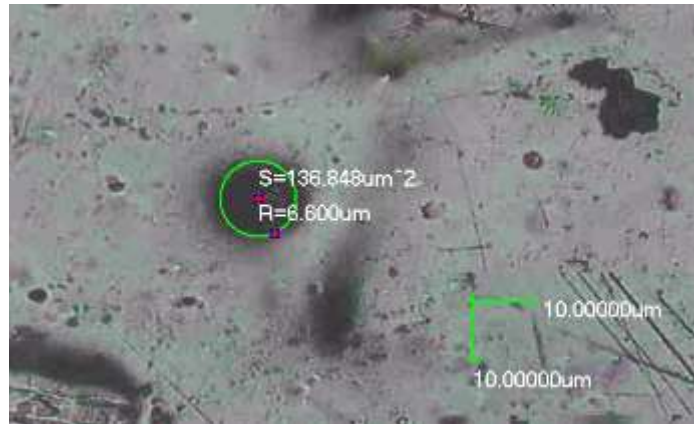


Figure II.24: Microvoids observed experimentally in the polyester/Alfa composite.

The stress concentration can be located in the defects and leading to the creation, initiation and propagation of the crack in the material [9]. These stress concentrations act as starting points for micro-cracks, which can grow under repeated or sustained loading, ultimately compromising the structural integrity of the composite. Furthermore, the composite with coarse fibers exhibits a much higher Young's modulus than the one with fine fibers and neat polyester resin. This clearly shows that the morphology and size of the reinforcement fibers strongly influence the stiffness and mechanical response of the final composite. These findings demonstrate that variations in Young's modulus are caused by atmospheric aging. Aging modifies the internal structure and weakens the fiber-matrix interface, resulting in decreased stiffness across all composite types. Indeed, atmospheric aging induces a decrease in Young's modulus for all aged composites and this variation was amplified by Alfa fiber content. The more the fiber content increases, the more the composite becomes susceptible to environmental degradation, due to the hygroscopic nature of the natural fibers. Moreover, the reduction of Young's modulus is less than 1% for the pure polyester caused by the natural ageing, and was reached 40% for the composite reinforced with 30 wt% of coarse Alfa fibers. This stark contrast highlights the trade-off between mechanical performance and environmental durability when natural fibers are introduced. Numerous studies have detailed the linear evolution of the longitudinal Young's modulus with the volume filling ratio [10–12], where relationships between the filling percent and the composites mechanical properties were shown. These studies confirm that mechanical reinforcement through fiber addition follows predictable trends, though environmental factors can disrupt this linearity over time.

II.9.2 Toughness Test

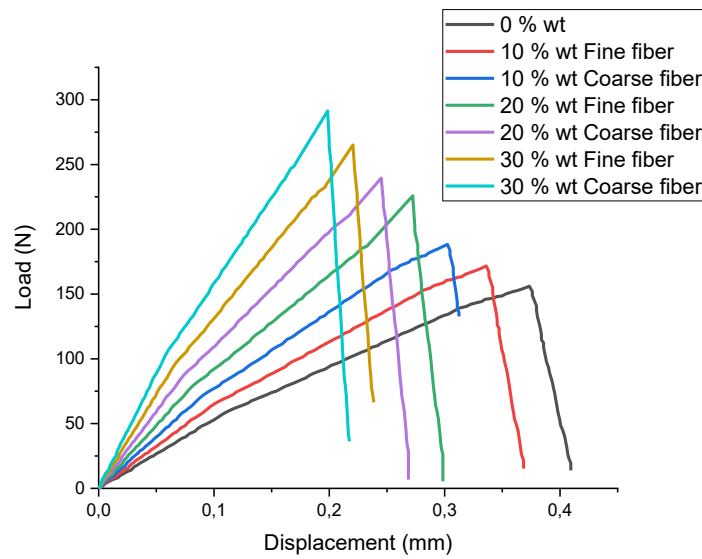


Figure II.25: Load-displacement unaged polyester/Alfa composites curve with different fractions of reinforcement.

Toughness presents a material property which states the material's capability to resist crack propagation. It is characterized by the mechanical field of the fracture process (considers the presence of defects) and appears when it recalls that the fracture is a failure mode governed by cracking (created, initiated and propagated). In other words, toughness reflects the energy a material can absorb before fracturing, especially in the presence of structural imperfections or notches. It is a key parameter when evaluating the durability and damage tolerance of fiber-reinforced composites. Load-displacement curves from the Mode I fracture test (Figure II.27) for unaged and aged polyester/alfa composites with varying reinforcement fractions (0, 10, 20, 30 wt%) are presented in Figures II.25 and II.26, respectively. These curves offer valuable information on the crack initiation and propagation phases under tensile stress perpendicular to the crack plane (opening mode). Comparing unaged and aged specimens allows for a better understanding of how environmental exposure alters the fracture toughness of the composites. As reinforcement content increases, changes in curve shapes and peak loads provide insights into the toughening or embrittlement mechanisms active in the material.

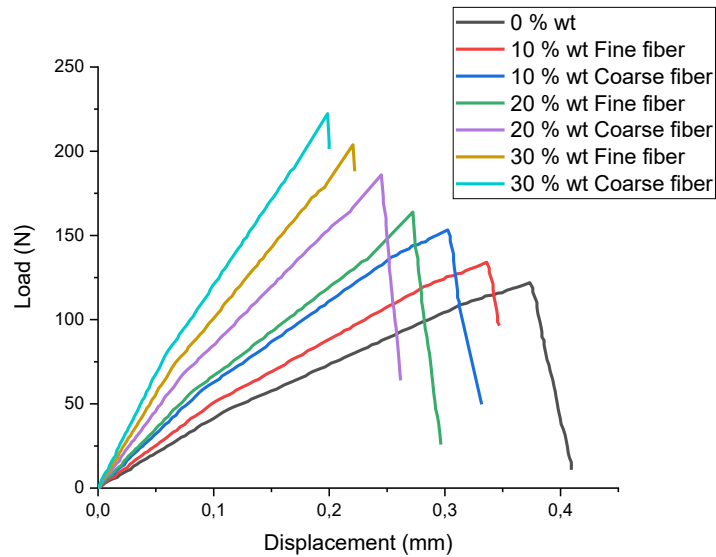


Figure II.26: Load-displacement aged polyester/Alfa composites curve with different fractions of reinforcement (0, 10, 20, 30 wt %) for both sizes of Alfa fibers (fine and coarse).

The fracture toughness (K_{IC}) and fracture energy (G_{IC}), were measured for the polyester and epoxy resins, respectively, are $0.30 \text{ MPa}\cdot\text{m}^{0.5}$ and 90 J/m^2 , which are typical values for these resins [14]. These baseline values serve as reference points for evaluating the improvements brought by natural fiber reinforcement. Several elements and characteristics have been shown to affect the mechanical properties of polymers. For instance, polymers have a poor impact and fatigue strength and comparatively low fracture toughness. Their intrinsic brittleness makes them vulnerable to sudden failure under impact or cyclic loading. Porosity may have an impact on fracture toughness, despite what some researchers have claimed [15]. In fact, internal pores often act as stress concentrators that reduce energy absorption capacity. Higher levels of porosity may cause microcracking, since many works have been shown that pores incite to increase stress intensity and initiate cracks [16]. These microcracks can rapidly propagate under load, leading to a decrease in both strength and durability. A disadvantage of thermosetting resins is, however, their mediocre impact resistance which makes them brittle, and their low fracture toughness facilitates the propagation of cracks within the material. This limitation represents a major challenge in applications requiring high toughness and damage tolerance. The reinforcement added in the matrix, as polyester or epoxy, can improve its stiffness and toughness [17]. Natural or synthetic fibers can serve as effective barriers to crack propagation by distributing stress and absorbing fracture energy. For example, the comparison of pure polyester and reinforced with sugarcane bagasse fiber by a volume fraction of 30% into a polyester matrix composite increases the notch toughness of Charpy impact tested

composites by 33% [18]. This example underlines the growing interest in agricultural waste as a low-cost, eco-friendly reinforcement alternative. In addition, the toughness of the polyester reinforced with 30 wt% of ground nutshell is improved by 60% compared to the polyester resin toughness [19]. Such significant improvements confirm the efficiency of natural reinforcements in modifying the mechanical response of polymeric matrices. However, when compared to the neat matrix (polyester resin) toughness, the toughness of the polyester reinforced with 30 weight percent Alfa fiber is enhanced by 80%. This remarkable increase places Alfa fiber among the most effective natural reinforcements for toughness improvement, validating its potential in structural bio-composites.

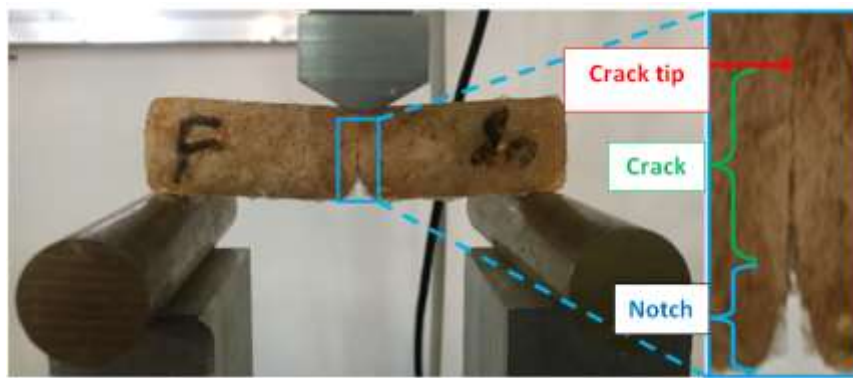


Figure II.27: Crack propagation under mode I fracture.

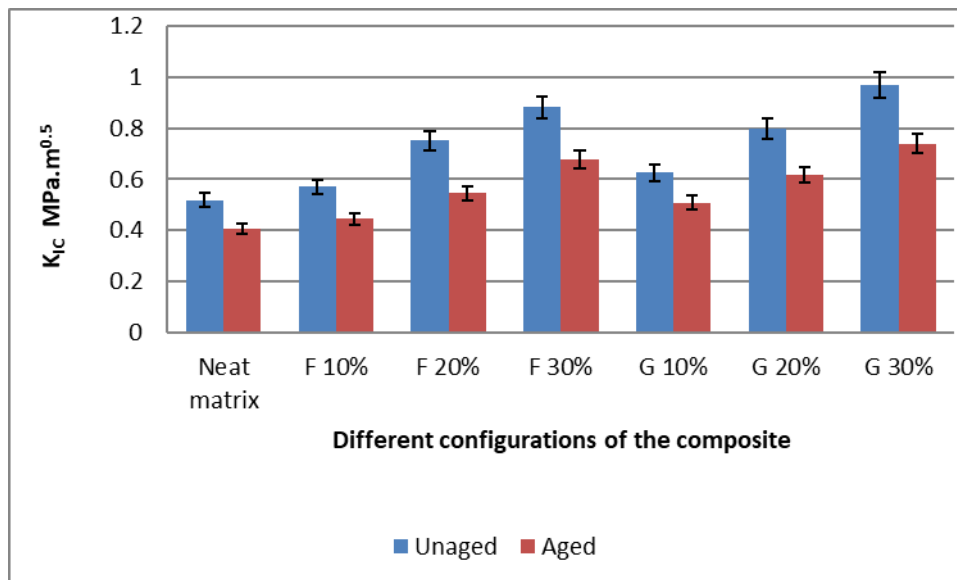


Figure II.28: Variation of fracture toughness (K_{IC}) of different Polyester/Alfa composites (Coarse and fine size).

II.9.2.1 Effect of Natural Aging on the Fracture Toughness

On the other hand, according to the results shown in Figures II.26 and II.28, the toughness properties of the polyester/Alfa composites can be affected by environmental aging. This sensitivity to aging reflects the complex interaction between environmental factors and

the composite's microstructure. For all composite categories, the cracking resistance of the specimens was significantly reduced after three months of aging in a specific environment. This decline highlights the loss of energy absorption capacity and the increased brittleness of the material over time. As test results, the toughness of the composite reinforced with 30 wt% of coarse and fine Alfa fibers are about $0.94 \text{ MPa}\cdot\text{m}^{0.5}$ and $0.85 \text{ MPa}\cdot\text{m}^{0.5}$, respectively. After an aging duration of three months in a specific environment, the values were reduced to $0.74 \text{ MPa}\cdot\text{m}^{0.5}$ and $0.68 \text{ MPa}\cdot\text{m}^{0.5}$. This represents a reduction of approximately 21% and 20%, confirming that even relatively short-term exposure can degrade fracture resistance. As a result, the mechanical behaviour of polymer composites deteriorates with increasing aging duration [20]. This degradation limits the long-term applicability of natural fibre composites in outdoor or humid environments. The mechanical and physical properties of various polymer composite materials are significantly affected by aging conditions, such as temperature, pressure, humidity, and curing conditions [21]. These parameters not only influence the polymer matrix directly but also modify the fibre-matrix bonding and internal stresses. Consequently, humidity and thermal aging are considered the primary factors responsible for the degradation of composites exposed to environmental conditions over time. Moisture penetration and thermal cycles act synergistically, accelerating microstructural damage. The consequences of atmospheric aging include the degradation of the matrix's mechanical properties, damage at the matrix/reinforcement interface, and differential swelling due to concentration gradients [22]. These effects compromise the overall mechanical cohesion of the composite, weakening both stiffness and toughness. The cellulose fibers in the composites will expand when they are subjected to various environmental factors, particularly moisture from the surroundings. This expansion induces internal stresses and distorts the matrix-fiber interface. As a result, shear stresses increase at the matrix/fibre interface, thereby raising the risk of interfacial debonding. Interfacial debonding disrupts stress transfer, thereby reducing the material's load-bearing capacity and its resistance to crack propagation. Joseph et al. found that water absorption exacerbates the degradation of the mechanical properties of natural-fibre composites [6]. This highlights the critical role of water absorption in accelerating composite aging and failure. Additionally, a reduction in thermal stability can lead to the degradation of the polymer, fibres, and the loosening of the polymer/fibre interface [7]. This weakening of thermal resistance contributes to the accelerated embrittlement of the material over time. The effects of atmospheric aging lead to a degradation of the mechanical properties of the polyester/alfa composite. These changes are gradual but cumulative, eventually compromising the reliability of the material under service conditions. The

degradation of properties can be attributed to the sensitivity of the polyester resin to ultraviolet (UV) rays, the biodegradability of alfa fibres, and, notably, the presence of defects introduced during the composite preparation process. Such defects whether microvoids, incomplete curing, or poor dispersion can act as aging accelerators, facilitating crack initiation and propagation.

II.10 Conclusion

This study demonstrated that alfa fibres can effectively reinforce polymer matrices, with coarse fibres providing superior strengthening compared to fine fibres. Increasing the fibre content enhanced the mechanical properties of the composites. However, after three months of atmospheric aging in a specific environment, a significant deterioration in the mechanical behaviour of the composites was observed, highlighting the substantial influence of environmental aging and filler content on their performance.

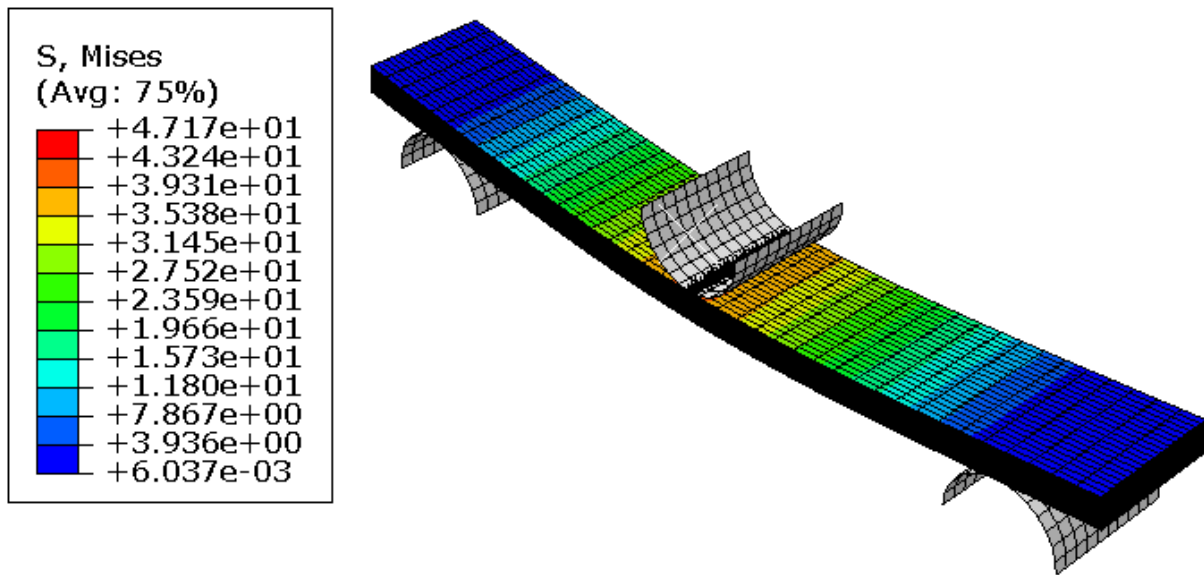
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Chapter II

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Chapter III

Finite element analysis of mechanical behavior of Polyester/Alfa composite

This chapter complements the experimental investigations of Chapter II through a numerical analysis using the finite element method in ABAQUS. It aims to validate the three-point bending tests and explore mechanical aspects not accessible experimentally, such as stress distribution and the evolution of the J-integral. The first part focuses on numerical validation and stress distribution, while the second analyses the variation of the J-integral in an SENB specimen.

Chapter III: Finite element analysis of mechanical behavior of Polyester/Alfa composite

III.1. Introduction

This chapter follows on from the experimental investigations presented in Chapter II and aims to deepen the understanding of the mechanical behavior of the studied biocomposites through a numerical approach based on the finite element method, implemented using ABAQUS software. The main objective of this chapter is twofold: first, to numerically validate the three-point bending tests performed experimentally, and second, to analyze mechanical aspects that are not easily accessible through experimentation alone, such as stress distribution and the variation of the fracture energy integral.

This chapter is structured into two complementary parts :

- The first part is dedicated to the numerical validation of the three-point bending tests described in the previous chapter. To this end, numerical models were developed to replicate the experimental conditions. A comparison between the simulated and experimental results is carried out to assess the accuracy and reliability of the proposed numerical model. In addition, this section focuses on the distribution of Von Mises stresses within the different biocomposites, allowing for the identification of critical zones and the mechanisms of stress localization that may lead to damage.
- The second part focuses on the variation of the J-integral for a Single Edge Notched Bending (SENB) specimen, which was previously examined experimentally in Chapter II. This analysis makes it possible to evaluate the fracture resistance of the various biocomposites and to examine the evolution of fracture energy as a function of notch geometry and material properties.

Through this numerical approach, it becomes possible to enhance the interpretation of the experimental results, validate the formulated hypotheses, and provide additional insight into the flexural behavior and fracture resistance of the studied materials.

III.2. Introduction to XFEM

One of the major scientific advancements of the 20th century was the invention of the computer, which greatly accelerated developments in computational mechanics. Techniques like the Finite Element Method (FEM) allowed for the simulation of complex engineering problems. However, traditional FEM faces challenges in modeling internal defects such as cracks, voids, and inclusions, due to mesh constraints. To overcome these limitations,

Belytschko and Moës introduced the Extended Finite Element Method (X-FEM) in the late 1990s. X-FEM enhances FEM by incorporating discontinuous functions into the shape functions, making the simulation of discontinuities independent of the mesh. This makes it ideal for problems involving fracture mechanics, localized deformation, and heterogeneous materials. X-FEM can handle complex geometries (like crack branching), requires less refined meshes, and integrates known analytical solutions to achieve high accuracy. Unlike other advanced methods such as the Boundary Element Method or meshless methods, X-FEM is built on the established FEM framework, ensuring compatibility with commercial software like ABAQUS and LS-DYNA.

III.3. Key Features and Advantages of X-FEM

- 1- Crack Modeling Without Remeshing: X-FEM allows cracks to initiate and propagate inside elements without the need for remeshing, even for complex crack geometries. This reduces computational costs significantly.
- 2- Enriched Elements for Accuracy: Elements near cracks are enriched with extra degrees of freedom, enabling accurate capture of stress singularities using a coarse mesh.
- 3- No Need for Field Variable Mapping: Unlike traditional FEM with remeshing, X-FEM avoids the need to remap field variables during crack propagation.
- 4- More Versatile than Boundary Element Method: X-FEM handles multi-material and nonlinear problems (geometric and contact) more effectively than the Boundary Element Method.
- 5- Easy Integration and Scalability: X-FEM can be conveniently implemented in commercial software and supports parallel computing, enhancing efficiency.

III.4. Finite Element Analysis

Considering the mechanical testing, finite element models of bend specimens were developed to analyze the fracture and stresses-strain curves of the polyester resin reinforced with different volumes of two sizes of alfa fibers (coarse and fine), the analysis was performed using the ABAQUS/explicit program [1].

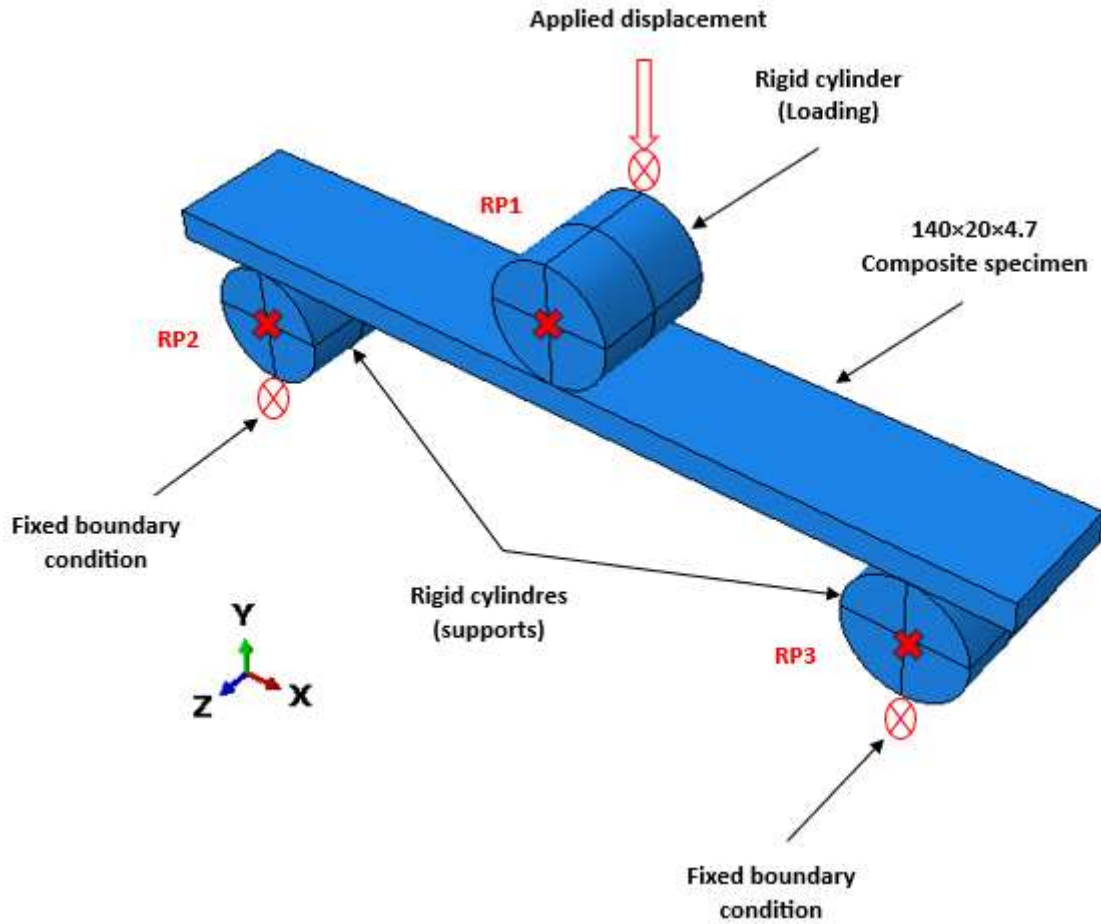


Figure III.1: Loading and boundary conditions applied to the model.

III.4.1. Validation of Numerical Bending Model

Numerical modeling is considered a validation of the experiment findings. Numerical computes are carried out in three dimensions, using the ABAQUS software, based on finite element development. Among the variety of elements available in the ABAQUS documentation, the boundary conditions have been defined in such a manner as to reproduce as well as possible the real conditions of the test. In fact, there are three boundary conditions applied to the model, the contact lines of the sample with the supports are blocked in translation along x, y, and z ($U_x=U_y=U_z=0$). The displacements of the punch are locked along x and z ($U_x=U_z=0$), with the application of a displacement in the y direction. All models were loaded to failure using 0.001 mm displacement increments in shear.

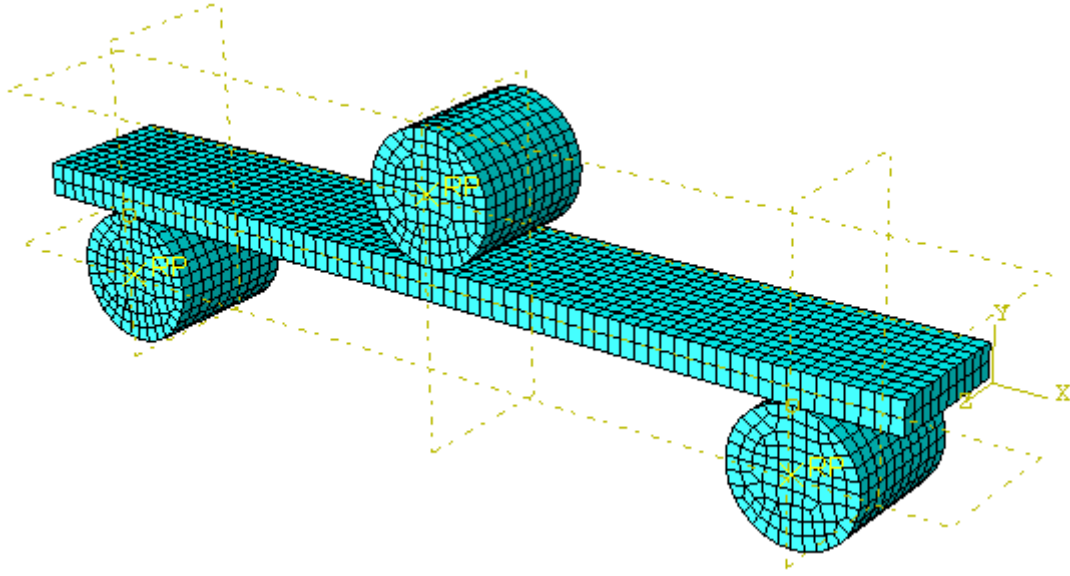


Figure III.2: The finite element mesh model (sample, punch, and supports).

For all cases under bend loading conditions, the finite element analysis established the distribution of von Mises stresses and shear stresses vs strain in the composite.

III.4.2. X-FEM and Fracture Criteria

To pretend the crack nucleation and growth numerically we applied the X-FEM in the polyester/alfa composites. The criterion of the maximum principal stress (σ_1) was chosen for the damage initiation as specified in Eq. (III.1) [2]. However, when the maximum principal stress becomes bigger than the tensile strength of the composite material, the crack presence can be seen, and the factor f^e refers to the stress ratio in an element (e):

$$f^e = \frac{\sigma_1^e}{\langle \sigma_{\max}^* \rangle} \quad (\text{III. 1})$$

Fracture energy G_c shows the criterion of crack propagation. In addition, supplementary numerical parameters are necessary to ensure the computation convergences which are 0.01 for Damage Initiation Tolerance and $10e-05$ for the viscosity coefficient and the dissipated energy fraction. This value allowed the calculation in a reasonable time without influencing the final result (the viscous dissipation energy remains much lower than strain energy during the whole calculation). Hence, only 1400 elements of the specimen mesh have been enriched, the head being subjected to a concentrated force which enrichment would cause inadequate cracking.

III.4.3. Finte Element Model

A numerical simulation of the bending test was performed in order to replicate laboratory test results. Numerical analysis was performed using the finite element method (FEM) with Abaqus explicit. Three-dimensional finite element models of specimens made of alfa/polyester composite were created based on mechanical testing in order to assess the stress fracture in polyester reinforced with varying volumes of alfa fibers for benchmarking purposes. Flexural simulations were conducted on Alfa composite materials using finite element analysis (FEA) software ABAQUS 2022. This software was utilized to generate the model and refine the finite element specifics, including contact properties, boundary conditions, and meshing the details of numerical modeling.

III.4.3.1. Geometrical Model

The dimensions used in these simulations were identical to those employed in the experiments conducted in the current study. The geometrical model is composed of a plate composite, of the following size : length, $L = 140$ mm, width, $w = 20$ mm, thickness, $e = 4.7$ mm. Three pins 19 mm in diameter were in contact with the specimen to apply the three-point bending loading. The supports and puncher were modeled using discrete rigid elements. Figure 5 displays the finite element model.

III.4.3.2. Mechanical Properties of Materials

The mechanical properties of the composite were derived from the experimental results obtained and are shown in Table III.1 and Table III.2. The Maxps damage model is used to simulate material damage when the material properties are simplified to linear elasticity. The puncher and the two supports are designed to be rigid without regard to the setting of its material properties. Table III.1 and Table III.2 shows the material attributes that were used, which were expressed in terms of yield strength, yield strain, Young's modulus, and Poisson's ratio for all related components. All materials were assumed to exhibit homogeneous materials.

III.4.3.3. Boundary Conditions and Load Application

For reasonable computational time, a stable time increment of 1×10^{-9} was used. The creation of operation points RP-1, RP-2, and RP-3 facilitated more accurate setting of the "load" and "boundary conditions." Three special points were defined and assigned to three rigid bodies in the model. The punch was linked to point RP-1 and configured as a "rigid body." The left and the right supports were similarly configured as a "rigid body" and bound to the points RP-2

and RP-3 respectively. A reference point (RP-1) was established in the middle of the FE model's top surface, as shown in Figure III.1. The direct cyclic was the step process. The boundary condition of "Displacement/ Rotation" was assigned to the composite model's bottom surface. The interaction between the loading puncher and the specimen is assumed to have general contact, as is the interaction between the supports and the specimen. The puncher was allowed to move freely along the z-axis, which is perpendicular to the face of the composite plate. An imposed displacement "U" is applied longitudinally to the specimen. In fact, there are three boundary conditions applied to the model, the contact lines of the sample with the supports are blocked in translation along x, y, and z ($U_x=U_y=U_z=0$). The displacements of the punch are locked along x and z ($U_x=U_z=0$), with the application of a displacement of 10 mm in the y direction ($U_y=10$ mm), in order to simulate the loading. The composite component was loaded with displacement increments of 0.01 mm in shear until it failed.

III.4.3.4. Finite Element Mesh

Based on a mesh convergence study, hexahedral elements of type C3D8R are used to mesh the generated model (Figure III.2). For two-dimensional contact analysis, however, ABAQUS advises the use of C3D8R hexahedral elements for the combination. The element type of each component is set to C3D8R. The Alfa composite specimen's "approximate global size" was set to 1 mm, while the puncher and supports were set to 2 mm. A study on mesh convergence was carried out, processing a number of runs with constant boundary conditions. There are 1400 elements and 2343 nodes in the entire finite element model.

III.4.3.5. Numerical Results

The numerical analysis determined the bending stresses versus strain and von Mises stresses distribution in the composite for all cases under pure bend loading conditions.

Table III.1 : Mechanical properties of polyester/alfa composite reinforced with fine fibers.

	Unaged composite				Aged composite			
	σ_{ultime} (MPa)	ϵ (%)	E(MPa)	ν	σ_{ultime} (MPa)	ϵ (%)	E(MPa)	ν
Neat matrix	25.24	3.02	1202	0.3	18.40	2.57	1001	0.3
10 wt % fibers	27.77	2,72	2003	0.3	20	2,32	1265	0.3
20 wt % fibers	40.34	2,57	2200	0.3	20.3	1,55	2024	0.3
30 wt % fibers	42.2	1,58	4000	0.3	31	1,72	2850	0.3

Table III.2: Mechanical properties of polyester/alfa composite strengthened with coarse fibers.

	Unaged composite				Aged composite			
	σ_{ultime} (MPa)	ϵ (%)	E (MPa)	ν	σ_{ultime} (MPa)	ϵ (%)	E (MPa)	ν
Neat matrix	25.24	3.02	1202	0.3	18.40	2.57	1001	0.3
10 wt % fibers	30.25	2,33	2220	0.3	22.63	2,08	1710	0.3
20 wt % fibers	40.72	2,20	3200	0.3	29.8	1,62	2310	0.3
30 wt % fibers	53.43	1,07	6010	0.3	39.13	1,58	3480	0.3

III.5. J-Integral Evaluation

A numerical method of the J-integral was utilized in the finite element model used in this study as a criterion of crack propagation and a measure of fracture mechanics safety. Based on the virtual crack extension/domain integral techniques, ABAQUS/Standard provides a procedure for these J-integral assessments. The finite element method offers a practical way to carry out virtual tests and examine how the damaged plate behaves in different configurations. It has been one of the most used numerical tools in the fracture mechanics field since the early 1960s. ABAQUS/CAE, a commercial nonlinear finite element package developed by SIMULIA Inc., was used to do the study. The beginning of the cracking was determined using the contour integral approach, which was also required as an output parameter to compute the J-integral at the crack tip. The theoretical concept of the J-integral was developed, independently, in 1967 by Cherepanov and in 1968 by Rice [3]. When the variation in elastic energy is greater than the variation in surface energy, there is propagation of the crack, which corresponds to a reduction in the free energy of the system. In an infinitely dimensioned plate of ideally elastic material containing a crack, it propagates when:

$$\sigma = \sigma_r = \sqrt{\frac{2 E \gamma_s}{\pi a}} \quad (\text{III.2})$$

With E = Young's modulus, σ_r = breaking stress and a = crack length.

$2E\gamma_s$ Corresponds to the energy required to create a cracking unit surface. It is in fact a critical energy that we note: G_{Ic} in J m^{-2} .

We can therefore rewrite (3) as follow:

$$\sigma = \sigma_r = \sqrt{\frac{EG_{Ic}}{\pi a}} \quad (\text{III.3})$$

The two criteria K_{Ic} and G_{Ic} characterize the sudden propagation of a crack. These two criteria are linked by the Relationship :

$$K_{Ic} = \sqrt{\frac{EG_{Ic}}{1-\nu^2}} \quad (\text{III.4})$$

With ν = Poisson's ratio

For isotropic materials exhibiting a marked ductile/brittle transition, the J-integral can be directly related to the ductile failure mode. In the case of plane deformation under loading conditions corresponding to Mode I, the relationship is given by :

$$J_{IC} = G_{IC} = K_{IC}^2 \left(\frac{1-\nu^2}{E} \right) \quad (III.5)$$

With G_{IC} : the relaxation factor of the critical strain energy, K_{IC} : the stress intensity factor in ductile failure under loading in mode I, ν : the Poisson ratio and E the Young's modulus of the material. The energy release associated with crack growth can be quantified using this integral when considering a path Γ that encloses the crack tip and has starting and ending points that rest on the two fractured faces. It has the following definition:

$$J = \int_{\Gamma} \left\{ w_e n_1 - \sigma_{ij} n_j \frac{\partial u_i}{\partial x_1} \right\} ds \quad (III.6)$$

Where w_e , an open contour around the tip of the crack, represents the elastic deformation energy density. Along the crack's axis, this is supposed to be rectilinear. It notices by: The normal outside the contour, $\sigma_{ij} n_j$: The contour's stress and the corresponding displacement: u_i (Figure III.3).

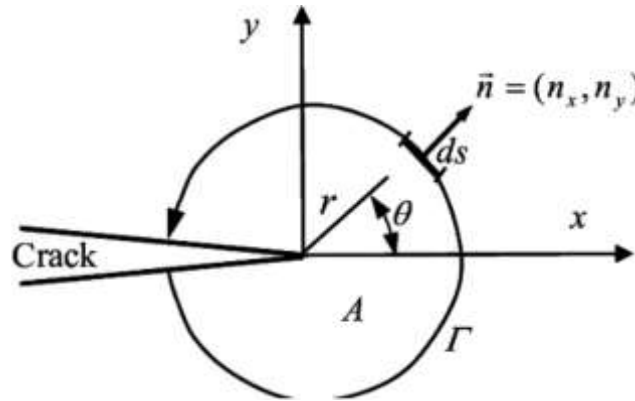


Figure III.3 : Contour integration path around crack tip.

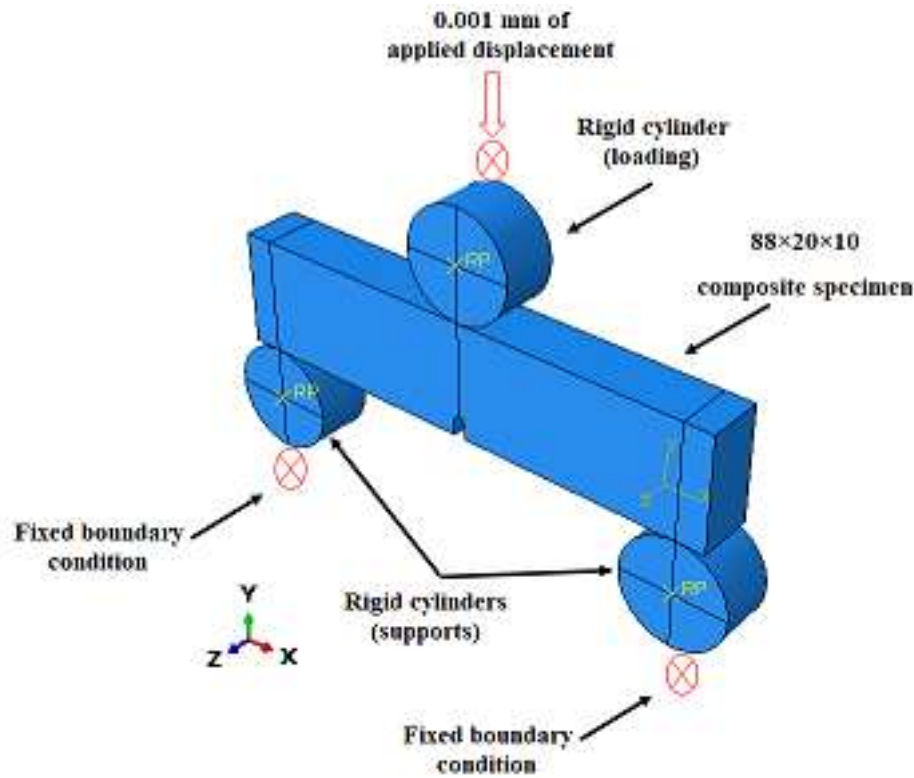


Figure III.4: Loading and boundary conditions applied to the model.

III.5.1. Materials Properties and Loading Conditions

According to the mechanical testing, finite element models of SENB specimens were made to investigate the fracture and stress–strain curves of polyester resin reinforced with different mass rates of two sizes of alfa fibers (coarse and fine). The geometry of the bending mechanism is shown in Figure III.4. The analysis was performed using the ABAQUS/Explicit program. It was anticipated that all materials would behave in a nonlinear, homogeneous, elastic/plastic manner (Table III.1 and III.2). The boundary conditions were defined in such a manner as to reproduce, as closely as possible, the real conditions of the test. In fact, there are three boundary conditions applied to the model: the contact lines of the sample with the supports are blocked in translation along x, y, and z (i.e., $U_x = U_y = U_z = 0$). The displacements of the punch are locked along y and z ($U_y = U_z = 0$). In shear, all models were loaded to failure using 0.001 mm displacement increments.

III.5.2. Introduction to Notches and Fracture Initiation

Many materials contain physical discontinuities, commonly referred to as notches. When a notched material is subjected to load, small fissures may begin to form. These fissures can merge with other microcracks, leading to the development of a larger macroscopic crack,

which propagates until the material ultimately fractures. Establishing rupture criteria for various stress conditions is essential for accurately predicting the performance of composites.

III.5.3. Fracture Analysis Using the J-Integral

By computing the J-integral at the crack front, the fracture behavior of the SENB specimens was investigated (Figure III.5).

III.5.4. Finite Element Mesh and Convergence Study

Based on a mesh convergence analysis, the generated model is meshed with 8-node linear brick (Hex) elements of type C3D8R. ABAQUS, however, suggests that elements for the combination for three-dimensional contact analysis be of type C3D8R. A study on mesh convergence was carried out, processing a number of runs with constant boundary conditions. There are 29,546 nodes and 25,790 elements in the entire finite element model.

III.5.5. Mesh Refinement Near the Crack Front

The mesh around the crack front has been sufficiently refined to predict the plastic zone and to calculate the opening mode of the J-integral with good precision (Figure III.6).

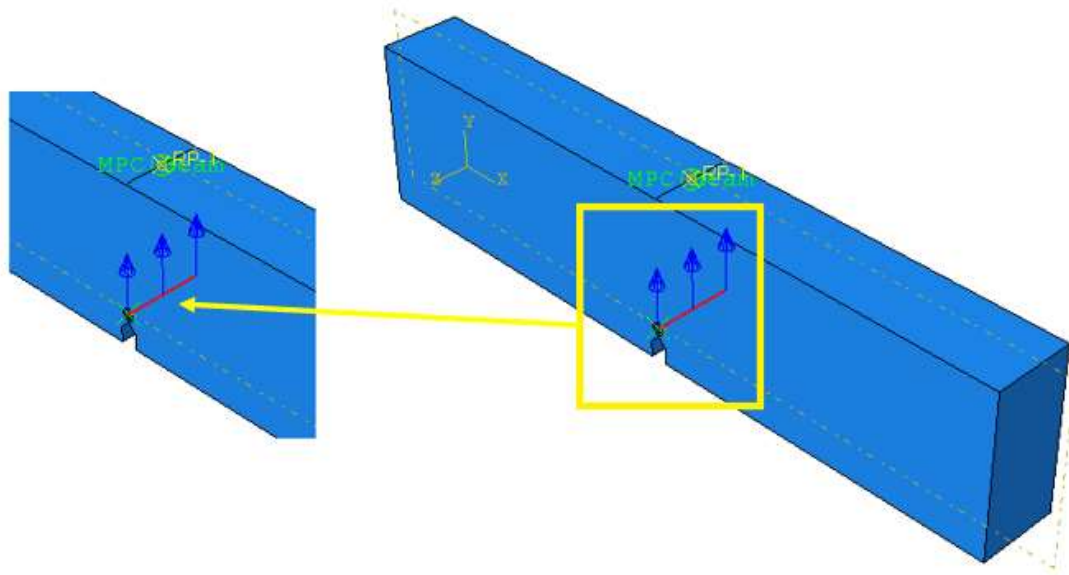


Figure III.5: 3D model with the direction of the fracture.

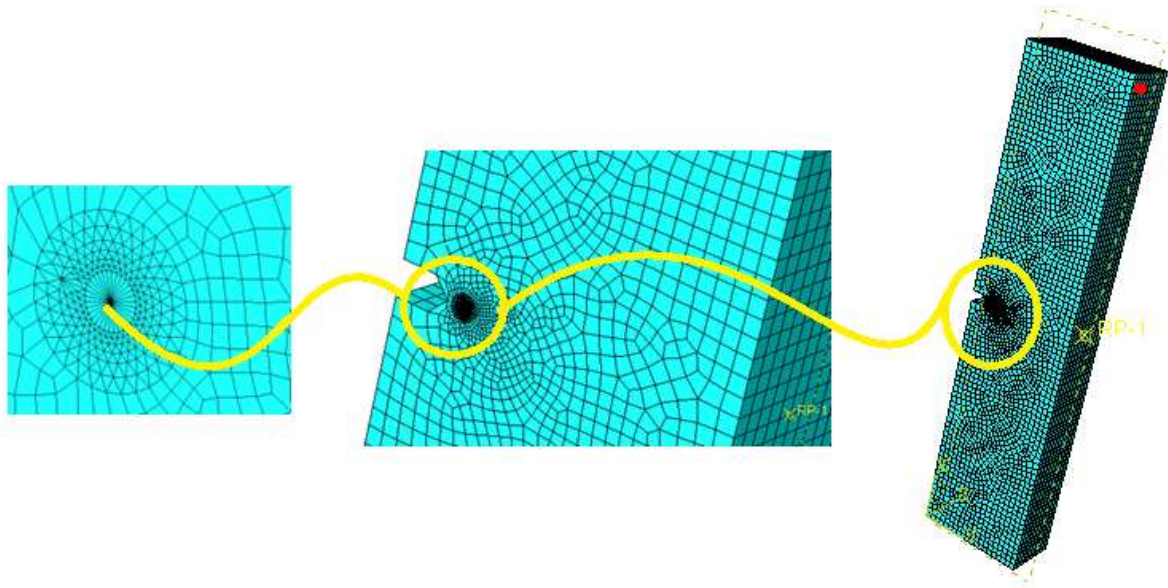


Figure III.6: The finite element mesh model: the SENB specimen, the mesh around the crack emanating from the notch, and the mesh around the crack tip (from right to left).

III.6. Results and discussion

III.6.1. Numerical Analysis and Validation

To examine the specifics of the composite Alfa polyester's bending resistance ability, its deformation mode during the bending process is shown in Figure III.7, where the general case's experimental and numerical deformation states were compared. Clear fracture in Zone (a) (marked with red lines) can be seen both in the experiment and simulation. They align rather well with one another. These details of composite Alfa polyester further demonstrate the numerical model's applicability and validity. The comparison between the FE and experimental findings (stress-strain curves) of both alfa/polyester composite cases (fine and coarse alfa fibers) reinforced with different fractions of fibers (0, 10, 20, and 30 wt%) is illustrated in Figure III.8. The experiment findings and FE results are in good agreement, as seen in the figure, with an estimated average relative inaccuracy of less than 15% in the perceived strength. The continuum damage mechanics-based model appears to be sufficient for simulating the failure of the alfa/polyester composite.

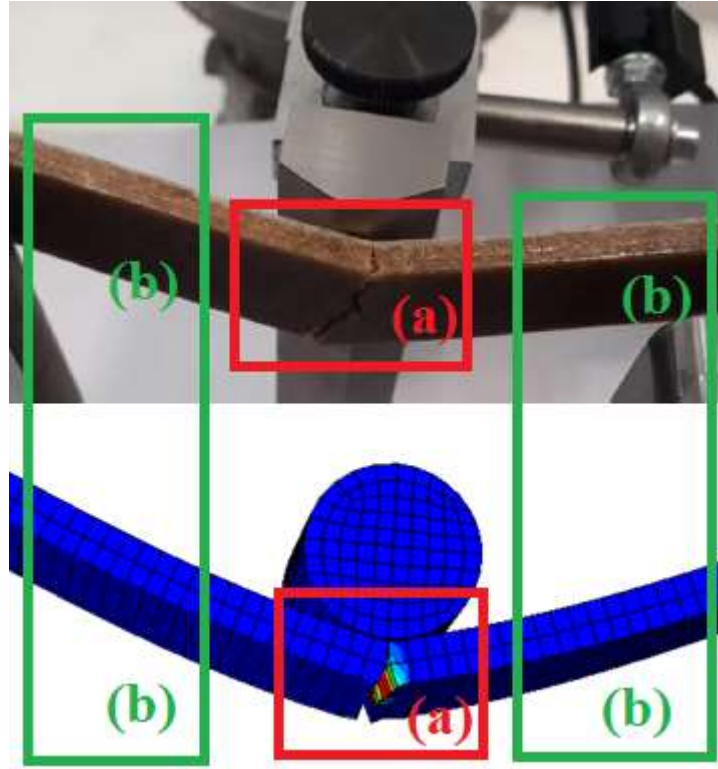


Figure III.7: Central area failure: experimental and numerical.

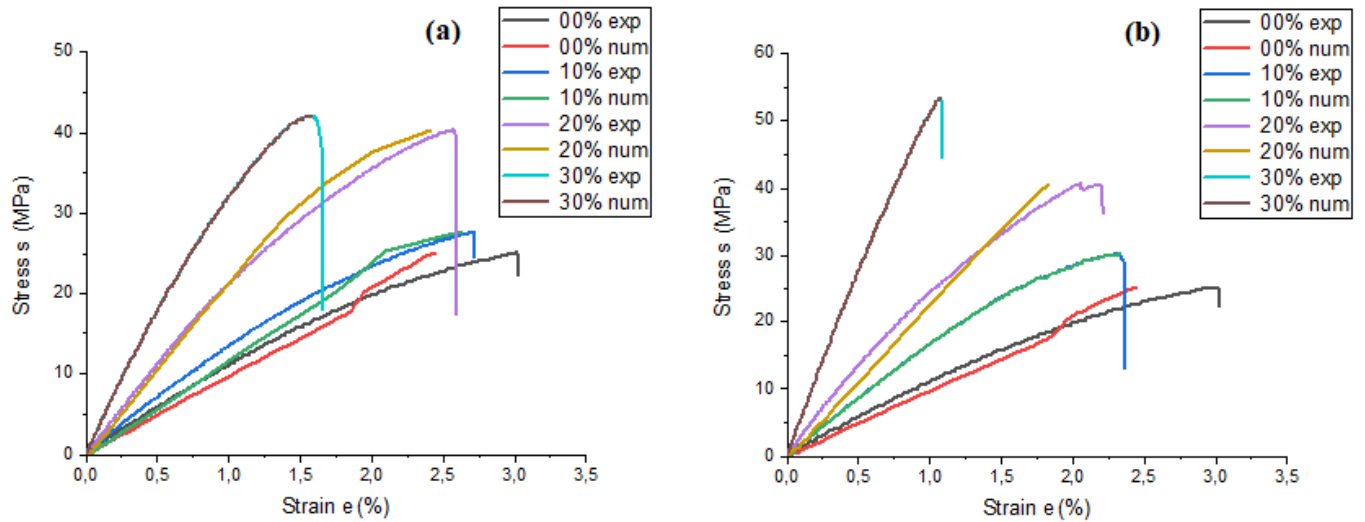


Figure III.8: The experimental and numerical stress-strain curves of unaged Alfa/polyester composite with different fractions of reinforcement (0, 10, 20, 30 wt%) for both sizes of alfa fibers: (a) fine fibers and (b) coarse fibers.

Figure III.9 presents a comparison between the predicted and experimental stress-strain curves for all cases of aged alfa/polyester composites under bending loading conditions. The mechanical responses observed in the experiments are well represented by the numerical data, with the average relative error in the apparent stress estimated to be less than 15%. These

composites' experimental and numerical curves show the same shape with a tolerable error. The existence of voids, discontinuities, and porosity in the fiber and matrix might cause this error. Higher strength and fracture energy are typically associated with mostly shear loading due to bend loading conditions, which tends to cause numerous damages such as fracture of the polyester and alfa fiber as well as decohesion of the polyester/alfa fiber interface. Data from the flexural analysis can be used to forecast the failure behavior of composite materials, suggesting that the constitutive principles chosen for damage initiation and evolution appear to be plausible.

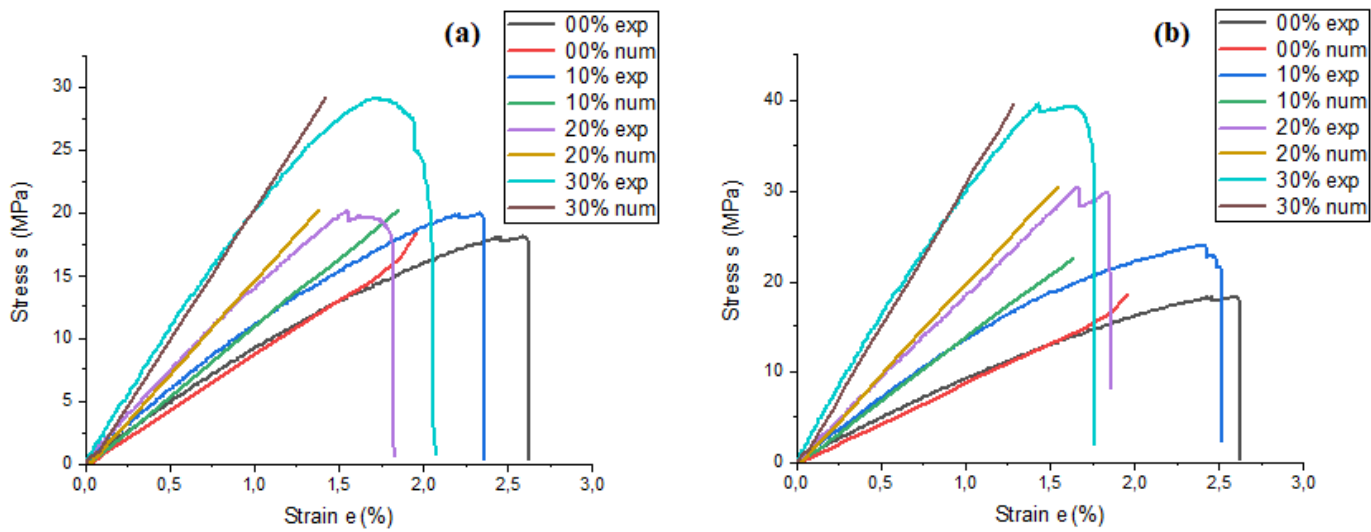


Figure III.9: The experimental and numerical stress-strain curves of aged Alfa/polyester composite with different fractions of reinforcement (0, 10, 20, 30 wt %) for both sizes of alfa fibers: (a) fine and (b) coarse.

III.6.2. von Mises Stress Distributions

Figure III.10 illustrates the von Mises stress distributions along the longitudinal axis of the composite specimen. According to this figure, for all cases of composites, it has been seen that the highest von Mises stress is noted in the middle of the specimen. It seems that the von Mises stress values found in the composite reinforced with coarse fibers are greater than the stress values in the composites reinforced with fine fiber. The analysis of the von Mises stress distribution at different weight fractions (0%, 10 %, 20 %, 30 %) can be seen in this figure, revealing that the curve of the von Mises stress at 30% is different from those at the other fractions. Without reinforcement, the polyester has a maximum stress of around 23 MPa., in polyester reinforced with 30% fine and coarse alfa fiber, this value increases to 38 MPa and 50 MPa. The association between the effects of the percentage of alfa fiber and its size increases the strength of the alfa/polyester composite.

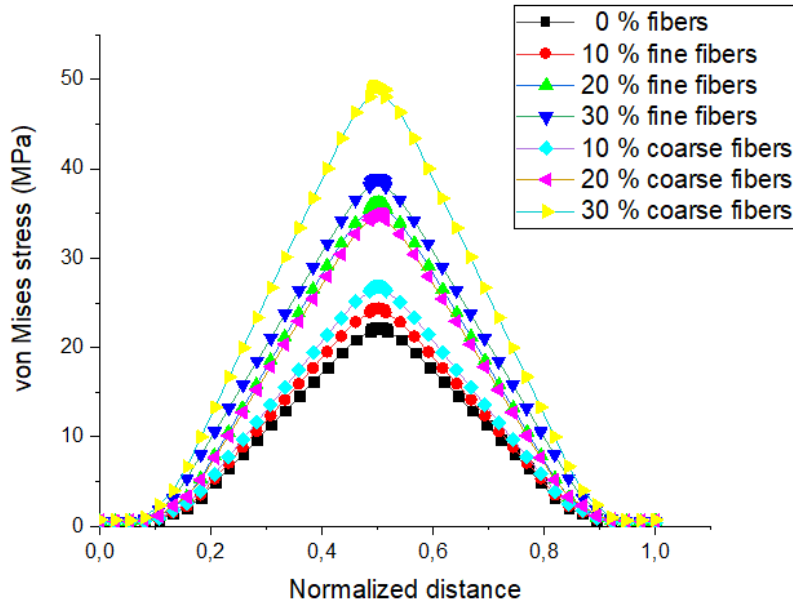


Figure III.10: von Mises stress distributions: Young polyester/Alfa composite with different fractions of reinforcement (0, 10, 20, 30 wt%) for both sizes of Alfa fibers (fine and coarse).

By comparing the values of the maximum von Mises stresses in the unaged and aged tests, it can be observed that natural aging has a significant effect on the degradation of the mechanical properties of alfa/polyester composite. For pure polyester (without reinforcement), three months of aging decreased the stress concentration by more than 30%; this reduction reaches 28%, in the case of the alfa/polyester composite reinforced with 30% of coarse alfa fibers (Figure III.11).

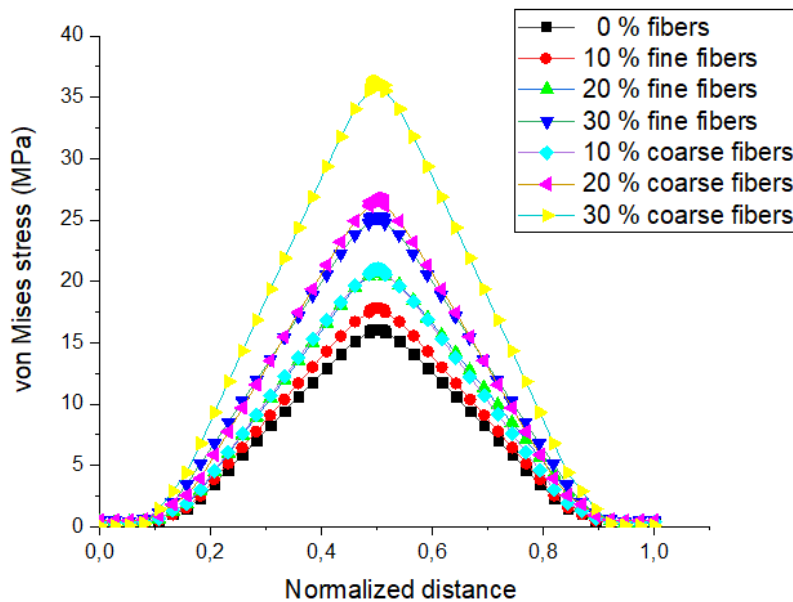


Figure III.11: von Mises stress distributions: Aged polyester/Alfa composite with different fractions of reinforcement (0, 10, 20, 30 wt%) for both sizes of Alfa fibers (fine and coarse).

III.6.3. J-Integral Evaluation

III.6.3.1. Effect of the Fiber Proportion, Fiber Size and the Crack Length

The change in the value of the J-integral along the crack's length for an unaged polyester composite reinforced with fine and coarse alfa fibers under Mode I loading is shown in Figure III.12. One of the principal geometrical characteristics of fracture is the crack length. The J-integral was computed for different crack lengths, starting from $a = 1$ mm and increasing up to 6 mm. In all categories of composites, it has been observed that the value of the J-integral increases as the crack length grows. The results further indicated that the fiber content has a considerable impact on the J-integral. This figure demonstrated that as the percentage of alfa fibers in the polyester/alfa composite increased, the J-integral decreased. This implies that the energy at the crack tip is significantly reduced, and the fatigue life of the structure can be enhanced. Shear forces cause the load to be transferred into the matrix through the reinforcement. The increase in reinforcement content in the composite decreases the shear stress at the crack tip.

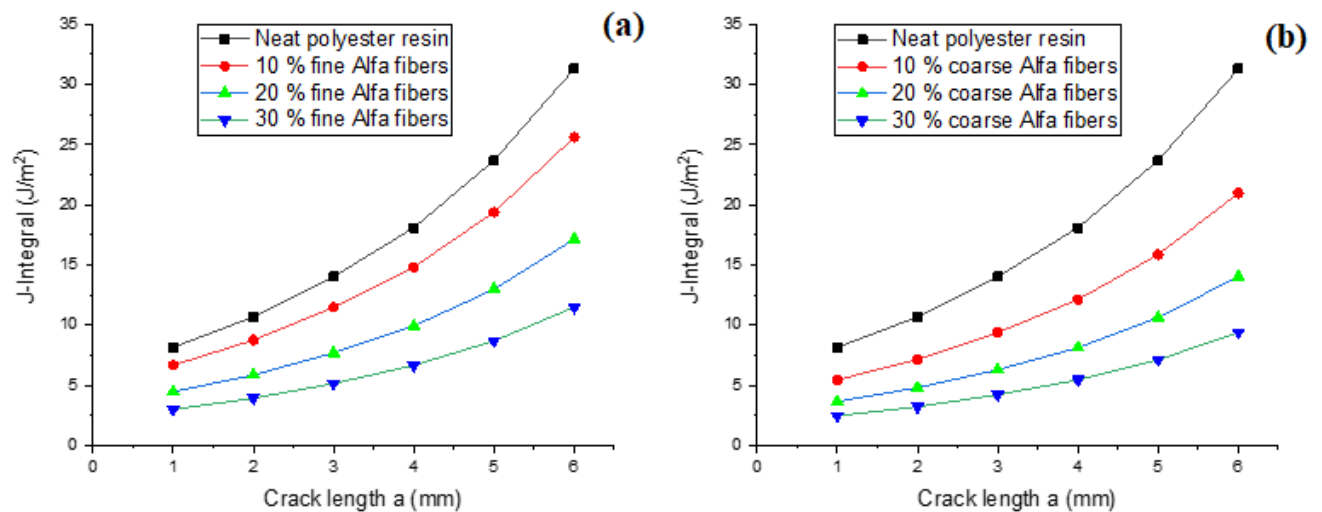


Figure III.12: J-integral versus crack length for young composite alfa polyester with fine (a) and coarse fibers (b).

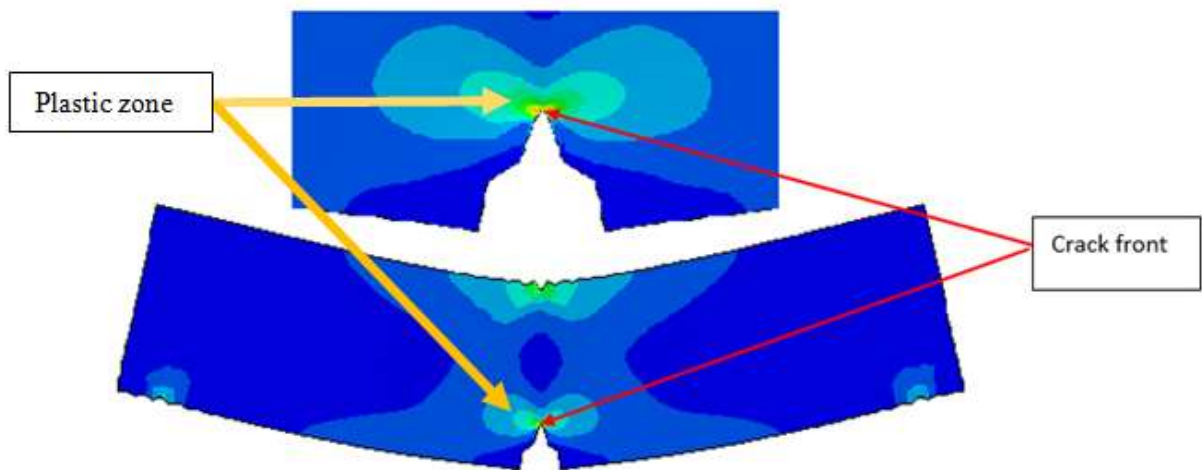


Figure III.13: A three-dimensional representation of the fractured composite.

Consequently, the plastic zone around the crack tip decreases (Figure III.13), which is consistent with a reduction in the J-integral. As the fiber ratio in the composite increases, crack propagation decreases. Thus, it can be concluded that adding more alfa fibers to a composite significantly enhances its reinforcing properties and increases its resistance to crack propagation. On the other hand, the fiber size is expected to have important effects on the values of the J-integral in composites. The coarse fiber size in the composites provides greater resistance to fracture and crack opening than the fine fibers.

III.6.3.2. Effect of Natural Aging

Figure III.14 illustrates how the J-integral varies based on the size of the crack in the polyester/alfa composite affected by atmospheric (1-6 mm of crack length with increment of 1mm).

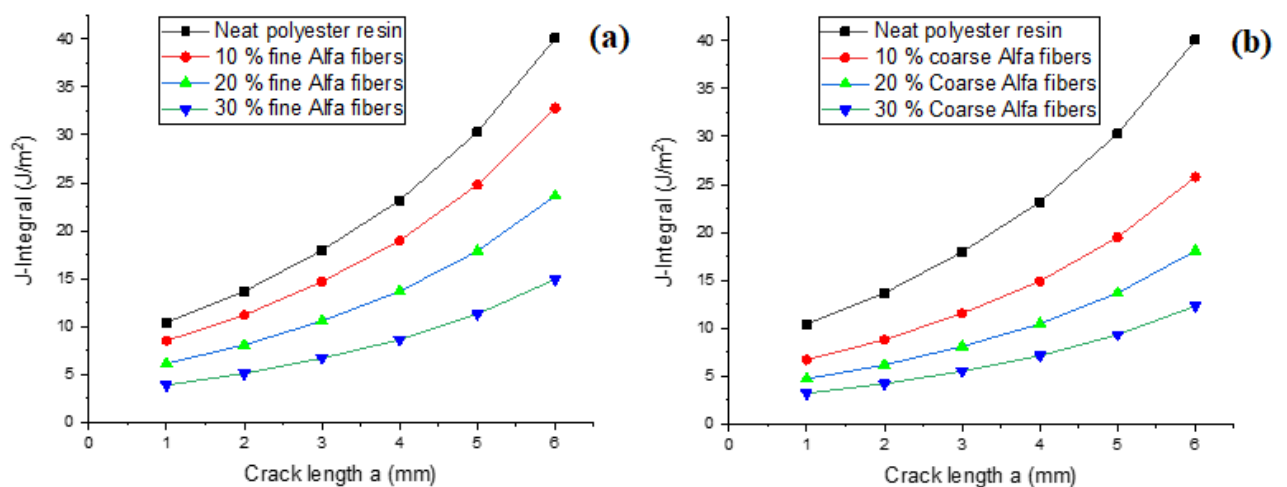


Figure III.14: J-integral versus crack length for aged composite alfa polyester with fine (a) and coarse fibers (b).

For all cases of composite, the J-integral considerably increased after three months of aging in a particular environment. The lowest values were noted for the composite reinforced with 30 wt. % of coarse and fine alfa fibers when the crack length was $\alpha = 1$ mm. It has been observed that the J-integral of the composite reinforced with 30 wt.% of coarse and fine alfa fibers is about 3.20 and 3.88 J/m², respectively. These values increased to 12.31 and 14.93 J/m² for a crack length $\alpha = 6$ mm. The values of the J-integral at the crack tip were about 25% higher than those of the unaged polyester/alfa composite. These results confirm that atmospheric aging degrades the mechanical properties of composites, particularly their fracture resistance.

III.7. Conclusion

At the end of this chapter, the numerical analysis conducted using ABAQUS has served to complement and deepen the understanding of the experimental results previously obtained. The simulations contributed, on the one hand, to the validation of the three-point bending tests by accurately reproducing the observed experimental behavior, and on the other hand, to a better understanding of the stress distributions and fracture mechanisms in the various biocomposites studied.

The main findings derived from this chapter can be summarized as follows:

- The simulation results showed strong agreement with the experimental results.
- The J-integral increases with the crack length across all composite categories, reflecting a growing energy release rate at the crack tip as the crack propagates.
- Increasing the percentage of alfa fibers in the polyester matrix leads to a decrease in the J-integral, indicating reduced energy available for crack propagation and thus improved fracture resistance of the composite.
- Composites reinforced with coarse alfa fibers exhibit lower J-integral values compared to those with fine fibers, suggesting better resistance to crack opening and propagation.
- Higher fiber content reduces the shear stress at the crack tip and consequently decreases the size of the plastic zone, which supports the observed reduction in the J-integral.
- After three months of atmospheric aging, the J-integral values significantly increase (by up to 25%), indicating a notable degradation in the mechanical properties, particularly in fracture resistance.

These results highlight the significant influence of fiber characteristics and environmental aging on the fracture behavior of biocomposites. The numerical modeling approach has

proven to be an effective tool for analyzing complex mechanical phenomena and predicting the evolution of fracture parameters. This study lays the groundwork for further investigations into the optimization of biocomposite materials for enhanced durability and structural performance.

References

Chapter III

References

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Chapter IV

UV aging and mechanical performance of Flax hybrid composites

This chapter explores the impact of accelerated UV aging on the flexural performance of flax/epoxy composites and their hybrids with synthetic fibres. It addresses the degradation issues caused by UV exposure, particularly at the matrix–fibre interface. The study tests the hypothesis that hybridisation with glass or carbon fibres improves durability. Flexural properties were assessed after 300, 600, and 1000 hours of UV exposure through three-point bending tests.

Chapter IV : UV aging and mechanical performance of Flax hybrid composites

IV.1. Introduction

In recent years, natural fibre-reinforced polymer composites have attracted growing interest due to their biodegradability, low cost, and favourable mechanical performance. Among these, flax fibre-reinforced epoxy composites have emerged as promising candidates for structural and semi-structural applications in automotive, construction, and sports industries. However, their long-term durability, particularly when exposed to environmental factors such as ultraviolet (UV) radiation, remains a critical limitation for outdoor use. Although several studies have demonstrated the potential of hybridising natural fibres with synthetic ones (such as carbon, Kevlar, or glass) to improve mechanical performance, the influence of UV exposure on the long-term behaviour of these hybrid composites remains insufficiently explored. The main problem lies in the photo-degradation of the polymer matrix and fibre–matrix interface, which leads to a loss of structural integrity and service life over time. This study hypothesises that hybridisation of flax fibres with UV-resistant synthetic fibres can mitigate the negative effects of accelerated UV aging and improve the mechanical durability of the resulting composites. In particular, it is expected that composites reinforced with glass and carbon fibres will retain better flexural properties after UV exposure compared to those reinforced with flax alone or flax/Kevlar combinations. To test this hypothesis, an experimental investigation was conducted to evaluate the flexural behaviour of flax/epoxy composites and their hybrid variants after 300, 600, and 1000 hours of accelerated UV aging. Mechanical performance was assessed through three-point bending tests, focusing on parameters such as flexural modulus, ultimate strength, and deformation at failure. The following section details the materials used, the sample preparation, the aging protocol, and the testing procedures.

IV.2. Materials and methods

IV.2.1. Materials

In this study, various materials were used to fabricate the hybrid composites. The reinforcement materials included commercial fabric sheets made of carbon fibers (4 Harness Satin Weave type HexTow®, Hexcel Composites, Stamford, CT, USA) (Figure IV.1), glass fibers (Unidirectional type, Composites Canada, Mississauga, ON, Canada) (Figure IV.2),

flax fibers (Unidirectional type FlaxPly®, Lineo NV, Belgium) (Figure IV.3), and Kevlar fibers (Plain Weave type Kevlar® 49 style 5500, BFG Industries, Greensboro, NC, USA) (Figure IV.4). The matrix material was an epoxy resin system composed of Araldite® LY 1564 resin and Aradur® 22962 hardener, supplied by Huntsman Advanced Materials (The Woodlands, TX, USA) (Figure IV.5).



Figure IV.1 : Carbon Fiber Spools.

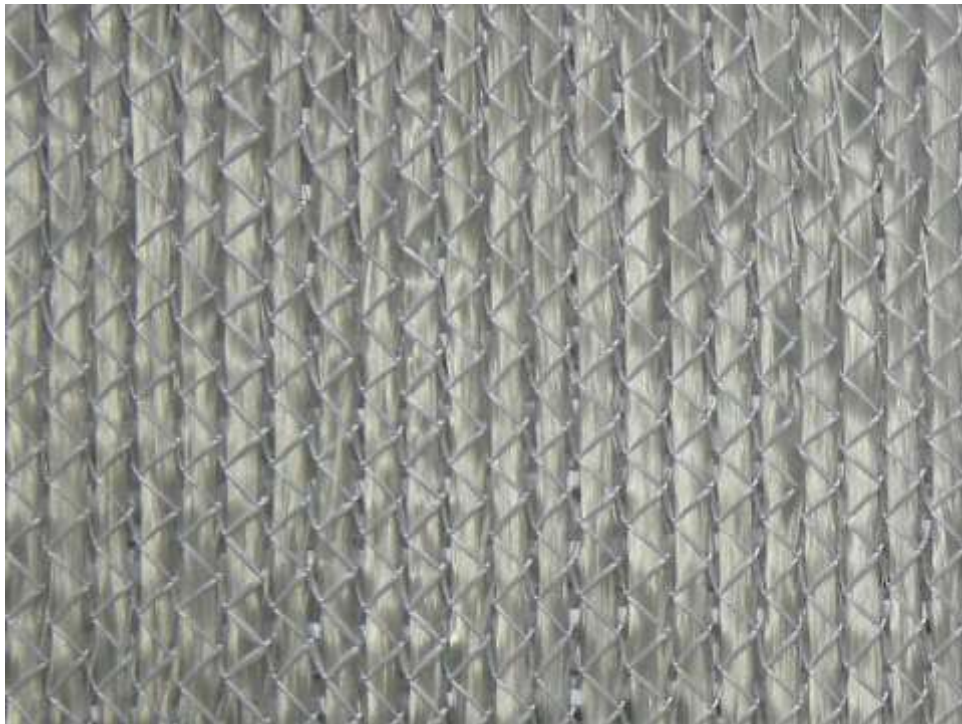


Figure IV.2 : Unidirectional Glass Fiber Fabric.



Figure IV.3 : FlaxPly® Natural Flax Fiber Fabric.



Figure IV.4 : Kevlar® 49 Fabric Roll.



Figure IV.5 : Araldite® Epoxy Resin and Aradur® Hardener.

IV.2.2. Hand lay-up

Epoxy matrix composites reinforced with flax fibres, as well as their hybrid variants, are developed in the Laboratory of Biomaterials and Biomechanics (LBB), Toronto Metropolitan University in Canada. The composite specimens were fabricated using the hand lay-up method (Figure IV.6). This process consisted of manually placing the flax fibre layers and hybrid reinforcements (carbon, Kevlar, or glass) into a flat mould. An epoxy resin was then applied to each layer using brushes to ensure proper impregnation. The stacking sequence was completed layer by layer, and the laminates were left to cure at room temperature under slight pressure for 24 hours. This method was selected for its simplicity, cost-effectiveness, and suitability for producing the required composite plates.

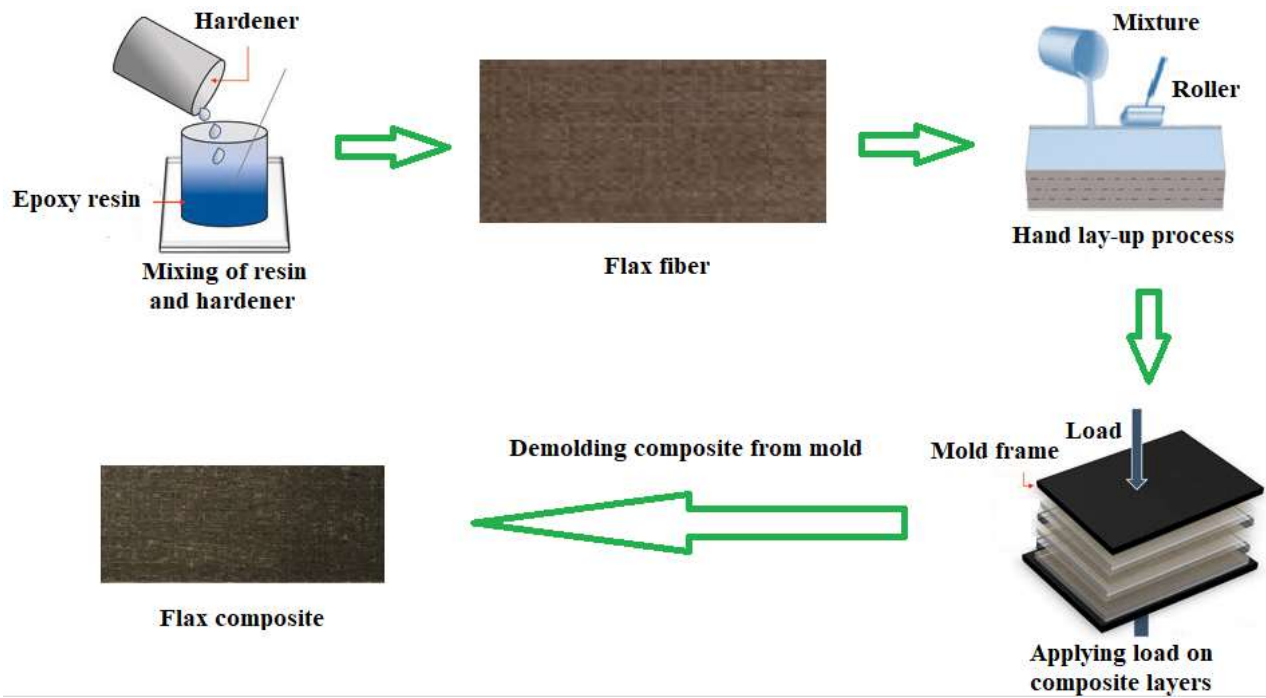


Figure IV.6 : Hand lay-up process.

IV.2.3. Hybrid Composite

Table IV.1 provides a summary of the different configurations that were manufactured and tested.

Table IV.1 : Summary of the different configurations manufactured and tested.

Specimen	Number of flax layers	Number of synthetic layers
Flax/epoxy (FE)	12	
Carbon/flax/epoxy (CFE)	12	2
Glass/flax/epoxy (GFE)	12	2
Kevlar/flax/epoxy (KFE)	12	2



Figure IV.7 : hybrids composites specimens for bending test.

IV.2.4. Accelerated Aging Test

Accelerated artificial aging was performed using a chamber in the form of a box, equipped with germicidal UV-C fluorescent lamps (GE S875, 28W, emitting at 254 nm) for a total of 300,600,1000 h (Figure IV.8). The test was conducted at room temperature, with the specimens placed vertically to ensure adequate exposure to UV-C radiation. The changes in the flexural properties of the tested samples were evaluated after 300,600 and 1000 h.



Figure IV.8 : UV-C fluorescent lamps (GE S875, 28W).

IV.2.5. Three-point flexural test

The effects of UV aging on the mechanical properties of the biocomposites laminates have been evaluated through flexural tests. These tests have been carried out on the materials immediately after their production (virgin material), and after 4 weeks of accelerated aging. Bending tests were performed using a BED 100 tensile testing machine equipped with a 1 kN load cell (Figure IV.9), in accordance with the ASTM D790 standard [1] at a load speed of 1 mm/min. Three samples of each type were tested to verify the repeatability of the results.

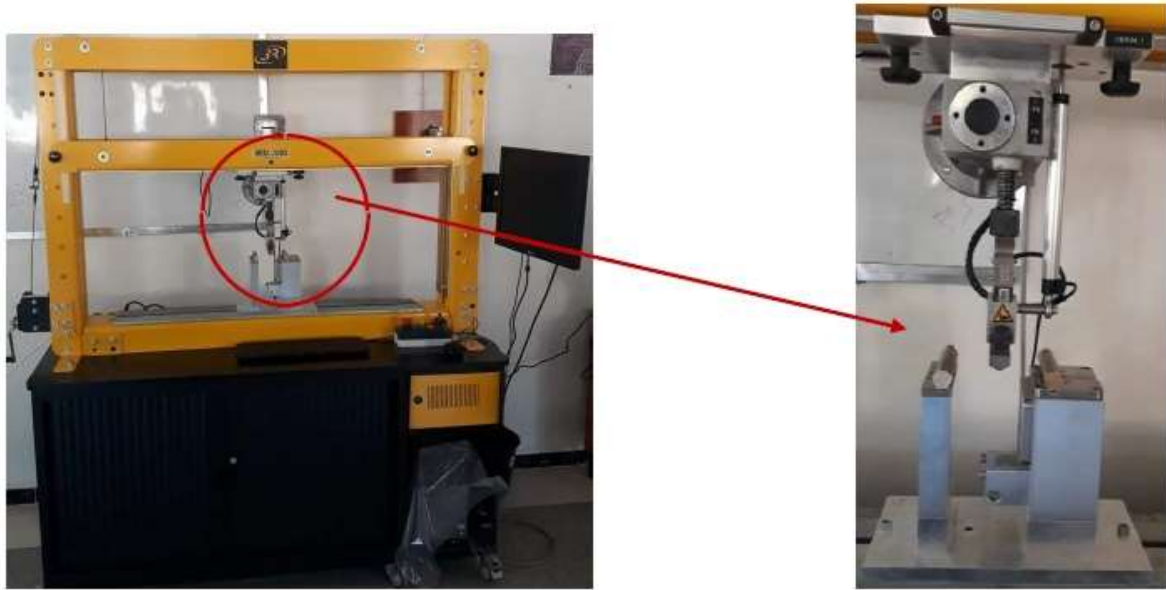


Figure IV.9 : Bend testing machine.

The bending specimens measured 250 mm in length, 10 mm in width, and 4 mm in thickness, with a support span of 110 mm (Figure IV.10).

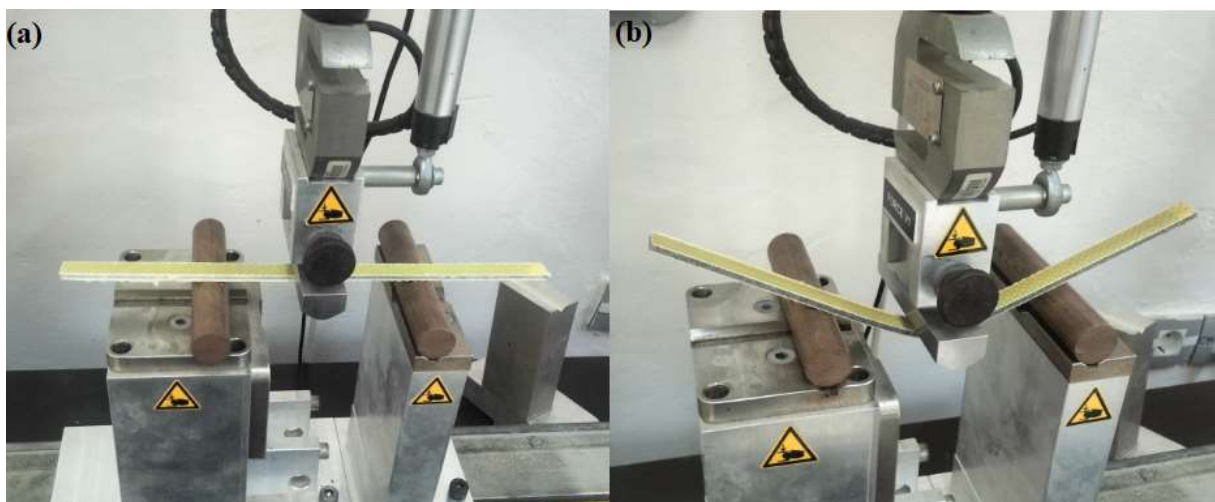


Figure IV.10 : Sequence of the experimental 3 points bending test.

The stress and strain resulting from a rectangular specimen subjected to a three-point bending load are calculated using the formulas presented in Equations (IV.1) and (IV.2) below.

$$\sigma = \frac{3FL}{2bd^2} \quad (\text{IV. 1})$$

$$\varepsilon = \frac{6Dd}{L^2} \quad (\text{IV. 2})$$

In these equations, F represents the applied load (N), L is the support span length, b is the specimen width, d is its thickness, and D denotes the maximum deflection at the beam's center. The deflection w can be estimated using Timoshenko beam theory, as expressed in Equation (IV.3).

$$w = \frac{Fl^3}{4Ebh^3} = \frac{Fl^3}{48EI} \quad (\text{IV. 3})$$

Equation (IV.4) is used to calculate the resultant Young's modulus of the material.

$$E = \frac{dF}{dw} \cdot \frac{l^3}{48I} \quad (\text{IV. 4})$$

IV.3. Results and discussion

This section presents and analyses the experimental results of three-point bending tests conducted on epoxy matrix composites reinforced with flax fibres, as well as their hybrid variants. The stress–strain curves obtained before and after ultraviolet (UV) exposure are used to assess the influence of UV ageing on the mechanical behaviour of these materials. This analysis highlights the effects of UV-induced degradation on mechanical properties, particularly flexural stiffness and strength, depending on the nature and configuration of the reinforcements used.

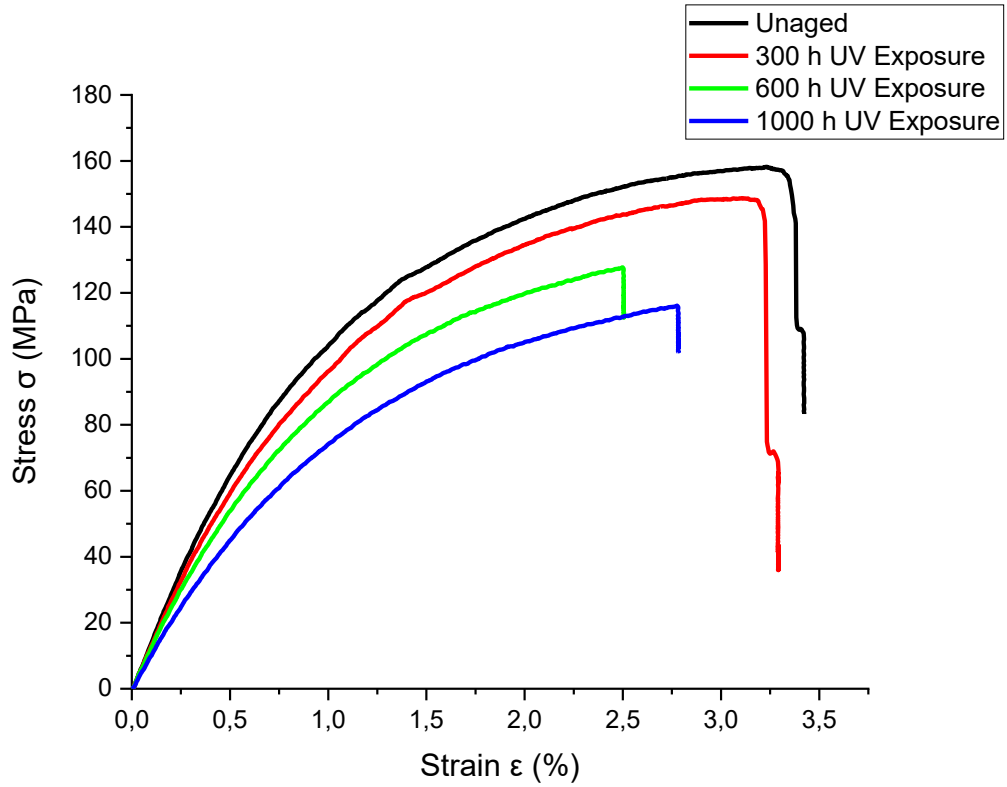


Figure IV.11 : stress-strain curve of Flax epoxy hybride composite.

Figure IV.11 illustrates the flexural stress–strain behaviour of flax epoxy composites subjected to different durations of ultraviolet (UV) ageing: unaged, 300 h, 600 h, and 1000 h, using a three-point bending test configuration. The unaged specimen exhibits the highest flexural strength, reaching a maximum stress of approximately 165 MPa and a strain at break beyond 3.5%. As the duration of UV exposure increases, there is a clear and progressive decline in both flexural strength and strain. After 1000 hours of UV ageing, the maximum flexural stress drops to below 120 MPa, while the strain at failure reduces to around 2.5%. This degradation reflects the sensitivity of flax epoxy composites to UV-induced ageing. The reduction in flexural performance is mainly attributed to photo-oxidative degradation of the epoxy matrix, which leads to polymer chain scission, embrittlement, and deterioration of the fibre/matrix interfacial bonding [2]. In addition, flax fibres, being lignocellulosic and hydrophilic in nature, are particularly prone to UV degradation, resulting in surface cracking and decreased mechanical interlocking with the matrix [3]. The flexural stress–strain curves also reveal a change in failure mode. While the unaged material shows a more ductile response with gradual failure, the UV-aged samples demonstrate more brittle behaviour with a steeper drop post-peak, particularly after 600 h and 1000 h of exposure. This shift indicates a decrease in energy absorption capacity, consistent with matrix microcracking, fibre/matrix

debonding, and fibre degradation reported in similar studies [4]. These findings confirm that prolonged UV exposure adversely affects the flexural properties of flax epoxy composites. Therefore, for structural applications requiring long-term outdoor performance, it is essential to incorporate UV stabilisers, protective coatings, or hybridisation with UV-resistant fibres such as glass or carbon [5].

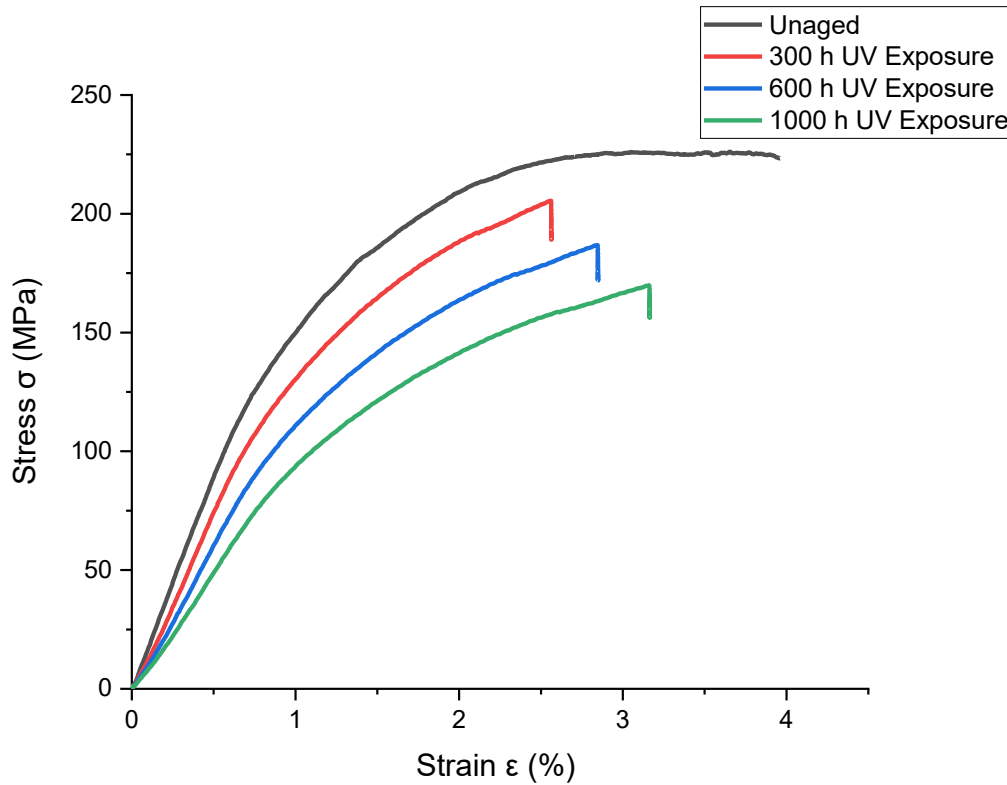


Figure IV.12 : stress-strain curve of Flax Kevlar epoxy hybride composite.

Figure IV.12 presents the flexural stress–strain response of flax/Kevlar epoxy hybrid composites subjected to accelerated ultraviolet (UV) ageing for 0 h (unaged), 300 h, 600 h, and 1000 h. The unaged sample exhibits the highest mechanical performance, with a flexural strength exceeding 230 MPa and strain at break above 3.5%. With progressive UV ageing, both flexural strength and ductility significantly decrease. After 1000 h of UV exposure, the flexural strength drops to below 170 MPa, and the failure strain is reduced to approximately 2.5%, indicating a more brittle response and reduced energy absorption capacity. This decline is primarily due to photo-oxidative degradation mechanisms, including matrix embrittlement, polymer chain scission, surface oxidation, and fibre/matrix debonding. The epoxy matrix becomes more prone to microcracking under bending stress, and the natural flax fibres, composed of cellulose, hemicellulose, and lignin, are particularly sensitive to UV exposure [6,7]. However, the integration of Kevlar fibres provides a partial protective effect. Kevlar

exhibits high mechanical strength, superior toughness, and excellent resistance to photochemical degradation. These characteristics contribute to enhancing the composite's resistance to environmental ageing, limiting the extent of UV-induced damage in the hybrid structure [8,9]. Compared to pure flax epoxy composites, flax/Kevlar hybrids demonstrate greater retention of mechanical integrity under long-term UV exposure, confirming the effectiveness of hybridisation strategies. The load is better redistributed between fibres, and the Kevlar component helps to maintain a more stable interfacial region, thereby reducing early failure and crack propagation [10]. The change in failure mode from ductile in the unaged sample to brittle in UV-aged samples further confirms the progressive loss of matrix toughness and interface strength. These results underscore the importance of combining natural and synthetic fibres in hybrid systems to improve ageing resistance for outdoor or semi-structural applications. To further enhance UV durability, protective surface coatings, UV stabilisers, or nano-reinforcements should be considered [11,12].

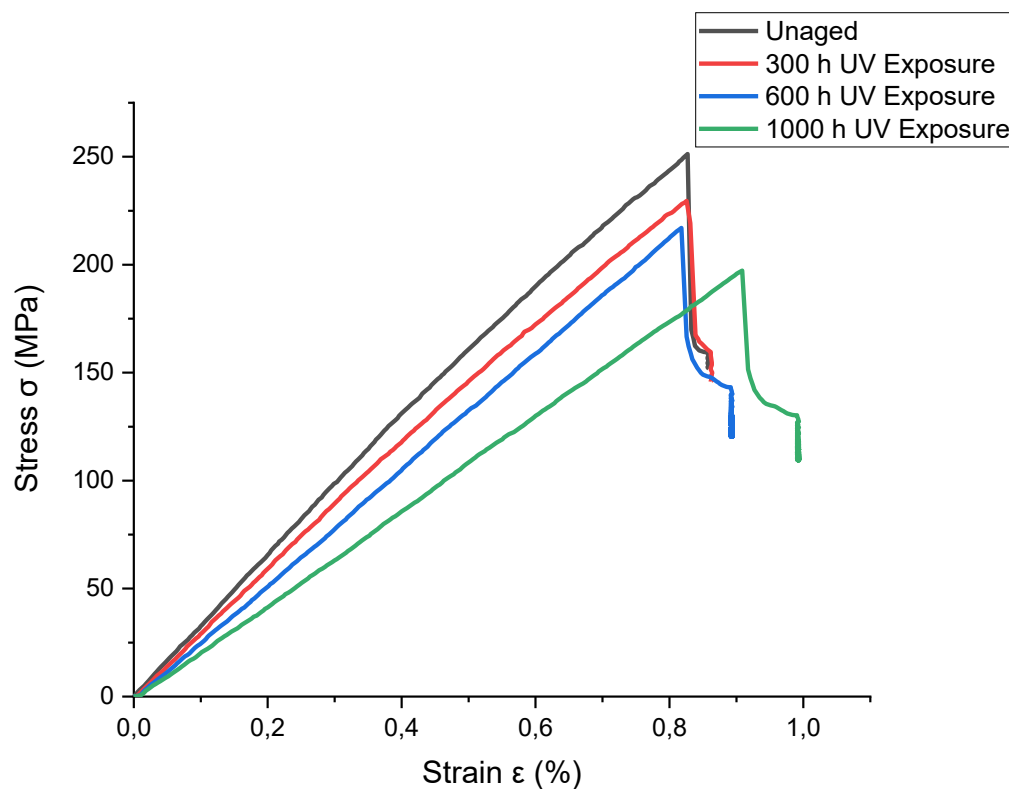


Figure IV.13 : stress-strain curve of Flax Carbon epoxy hybride composite.

Figure IV.13 illustrates the flexural stress–strain behaviour of flax/carbon epoxy hybrid composites subjected to different durations of UV exposure: 0 h (unaged), 300 h, 600 h, and 1000 h. The unaged composite shows the highest flexural strength, reaching approximately 250 MPa, and exhibits a strain at break close to 0.85%. Upon UV exposure, the mechanical

performance progressively degrades. After 1000 h of UV aging, the flexural strength decreases to around 180 MPa, and the strain to failure is also significantly reduced to below 0.9%, indicating brittleness and reduced ductility. The decline in flexural performance is primarily attributed to photo-induced degradation of the polymer matrix and fibre–matrix interface. UV radiation leads to oxidative scission, microcrack formation, and deterioration of the bonding between flax fibres and the epoxy resin. Although carbon fibres are chemically inert and highly UV-resistant, the natural flax fibres are susceptible to photodegradation, which results in microstructural damage and loss of integrity under bending stress [13,14]. Interestingly, this hybrid composite retains a better performance over prolonged UV exposure compared to pure flax epoxy systems. This improved durability is due to the reinforcement effect of carbon fibres, which exhibit high modulus, excellent fatigue resistance, and UV insensitivity. They effectively restrict the propagation of cracks, maintain load transfer efficiency, and reinforce the matrix despite partial degradation of the flax component [15,16]. The near-linear elastic behaviour observed in all curves up to the yield point reflects the stiffness and rigidity imparted by the carbon fibres. Post-yield, the reduction in flexural strength and the onset of brittle failure are more pronounced with increased exposure time, confirming the ageing-induced embrittlement of the polymer matrix [12].

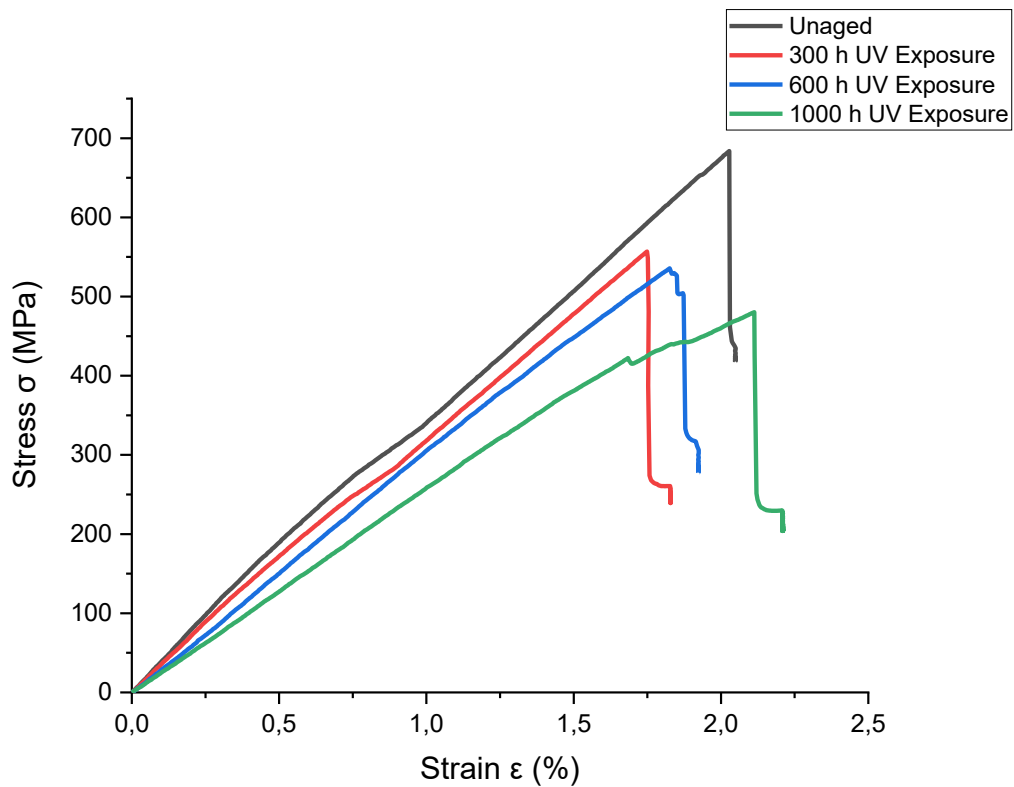


Figure IV.14 : stress-strain curve of Flax Glass epoxy hybride composite.

Figure IV.14 presents the flexural stress–strain curves of a glass/flax epoxy composite subjected to UV aging for 0 h (unaged), 300 h, 600 h, and 1000 h. In the unaged state, the composite exhibits the highest flexural strength, approximately 700 MPa, and a strain at break of around 2%. As the duration of UV exposure increases, a noticeable degradation in both strength and strain-to-failure occurs. After 1000 h of UV irradiation, the flexural strength drops to roughly 480 MPa, and the maximum strain decreases, indicating a loss of ductility and the onset of brittle fracture mechanisms. The degradation is attributed to UV-induced photochemical reactions within the polymer matrix and at the fibre–matrix interface. These reactions lead to microcrack initiation, matrix embrittlement, and a reduction in interfacial adhesion, especially affecting the natural flax fibres, which are more susceptible to UV radiation than the synthetic glass fibres [12,17]. However, compared to pure flax-based composites, the hybridisation with glass fibres significantly mitigates the detrimental effects of UV aging. Glass fibres are UV-stable, inert, and possess high tensile strength, which helps maintain structural integrity and reinforces the weakened regions caused by flax degradation. This hybrid structure ensures load redistribution, preventing catastrophic failure and enhancing post-aging performance [18,19]. The stress–strain curves exhibit a linear trend up to the yield point for all exposure durations, highlighting the composite’s initial stiffness. The steep slope observed for the unaged sample corresponds to a high modulus of elasticity. As aging progresses, this slope gradually decreases, indicating a loss of stiffness due to photo-oxidative degradation and fibre–matrix decoupling. These findings confirm the benefits of hybridisation using glass fibres in improving the long-term performance of natural fibre composites under UV-rich environments. Nevertheless, further enhancement could be achieved through surface treatments, UV-stabilised epoxy matrices, or nano-reinforcements that delay photodegradation [11,20].

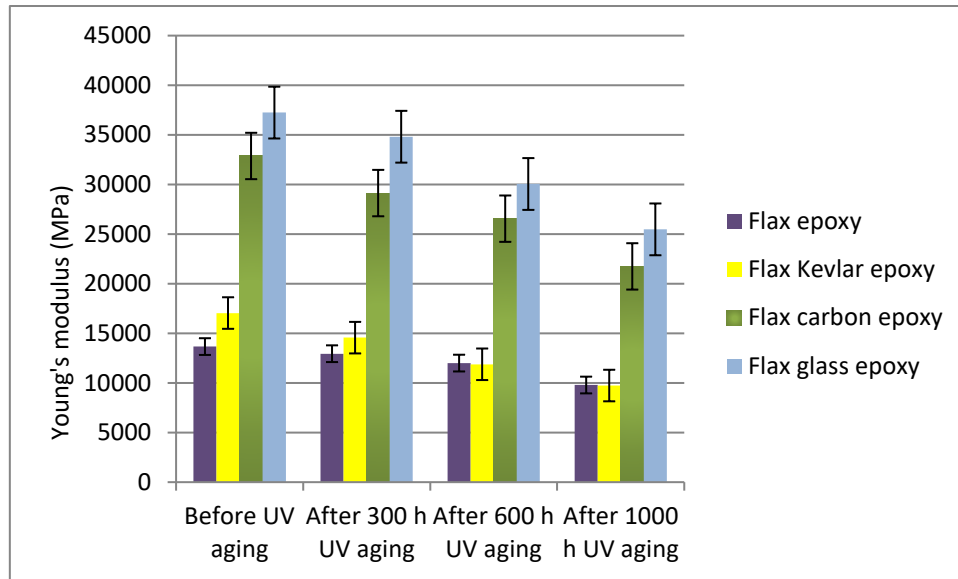


Figure IV.15 : Young's modulus variation of different hybrid composites.

Figure IV.15 illustrates the evolution of the Young's modulus (expressed in MPa) of different epoxy-based composites reinforced with natural or hybrid fibres (flax, flax/Kevlar, flax/carbon, and flax/glass), subjected to accelerated ultraviolet (UV) ageing for 0 h, 300 h, 600 h, and 1000 h. In general, a progressive decrease in Young's modulus is observed for all formulations as UV exposure time increases, reflecting a degradation in the mechanical properties of the material. This phenomenon is mainly attributed to the photo-oxidation of the epoxy matrix, which leads to polymer chain scission, as well as possible degradation of the fibre/matrix interface [2]. Among the tested composites, the one based solely on flax fibres (Flax epoxy) exhibits the lowest initial Young's modulus (around 14,000 MPa) and the most significant reduction in stiffness after 1000 h of UV exposure, dropping below 10,000 MPa. This pronounced sensitivity may be explained by the hydrophilic and photosensitive nature of flax fibres [3], combined with the limited UV resistance of the epoxy matrix [21]. The introduction of hybrid fibres significantly improves the mechanical resistance to ageing. The Flax/Kevlar/epoxy composite displays intermediate performance, with slightly higher stiffness than pure flax and moderate degradation, reflecting a stabilising effect of Kevlar, known for its excellent thermal and chemical resistance [22]. The Flax/carbon/epoxy composite, on the other hand, demonstrates far better mechanical stability, with initial Young's modulus values close to 32,000 MPa and a well-preserved performance after 1000 h of UV exposure. This is attributed to the chemical inertia and dimensional stability of carbon fibres [23]. Finally, the Flax/glass/epoxy composite stands out with the highest mechanical properties, exhibiting an initial modulus of around 38,000 MPa and a relatively moderate loss

in stiffness after 1000 h of exposure, where the modulus remains above 26,000 MPa. This excellent UV ageing resistance is due to the stability of glass fibres under UV radiation and their good compatibility with the epoxy matrix, ensuring a durable interface [24,25].

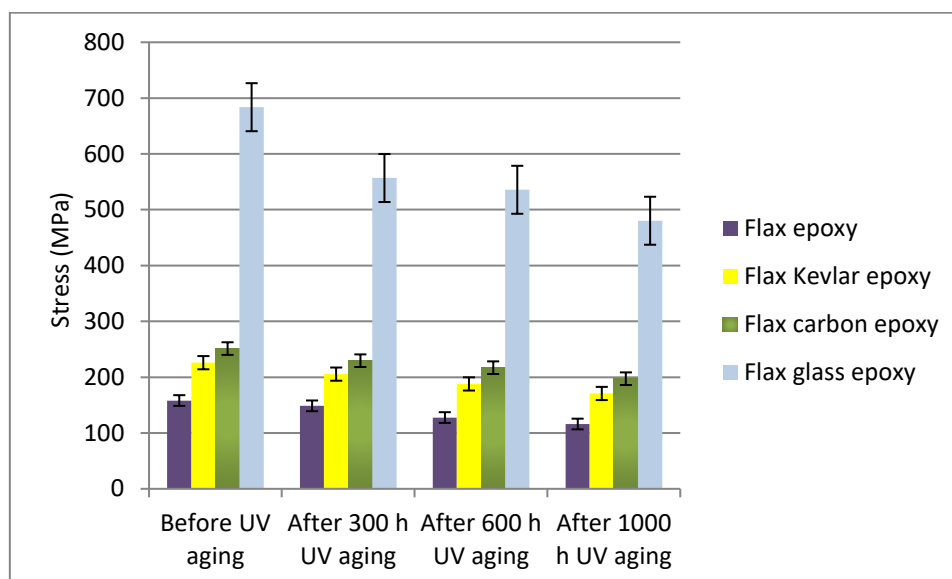


Figure IV.16 : Bending strenght variation of different hybrid composites.

The experimental study conducted on epoxy matrix composites reinforced with flax fibers, either alone or hybridized with synthetic fibers (Kevlar, carbon, or glass), aimed to evaluate the impact of UV aging (300 h, 600 h, 1000 h) on their behavior under three-point bending. The results show a clear decrease in maximum stress with increasing UV exposure time (Figure IV.16), indicating the progressive degradation of the epoxy matrix and the fiber/matrix interface under ultraviolet radiation. Before aging, hybrid composites exhibit significantly higher mechanical performance compared to pure flax/epoxy composites. The flax/glass/epoxy composite shows the highest maximum stress (around 700 MPa), followed by flax/carbon/epoxy (~250 MPa), flax/Kevlar/epoxy (~230 MPa), and flax/epoxy (~180 MPa). This ranking confirms the beneficial effect of hybridization with synthetic fibers, which enhances the overall stiffness and strength of the material. After 1000 hours of UV exposure, significant degradation is observed in all materials, though to varying extents. The flax/glass/epoxy composite remains the most efficient (~480 MPa), highlighting the excellent UV stability of glass fibers and their ability to maintain good adhesion with the matrix. The flax/carbon/epoxy composite also maintains reasonable performance (~200 MPa), due to the intrinsic resistance of carbon fibers to photodegradation. In contrast, the flax/Kevlar/epoxy composite shows a sharper drop in performance (~190 MPa), likely due to the sensitivity of Kevlar to photo-oxidative degradation, as reported by Chauhan et al. [26]. The flax/epoxy composite displays the most significant degradation, with maximum stress reduced to ~120

MPa after 1000 h. These results confirm that UV aging affects not only the polymer matrix but also the critical interfaces and natural fiber regions. Hybridization with synthetic fibers, particularly glass, helps to mitigate these negative effects due to their superior chemical and mechanical stability under UV exposure. Several recent studies support these findings. For instance, Nasser et al. [27] demonstrated that flax/glass hybridization improves long-term mechanical stability under UV conditions. Rizvi et al. [28] also showed that adding carbon or glass fibers delays crack initiation mechanisms triggered by photochemical degradation. Finally, Basarir et al. [29] emphasized the importance of selecting suitable hybrid fibers when designing durable composites for outdoor applications.

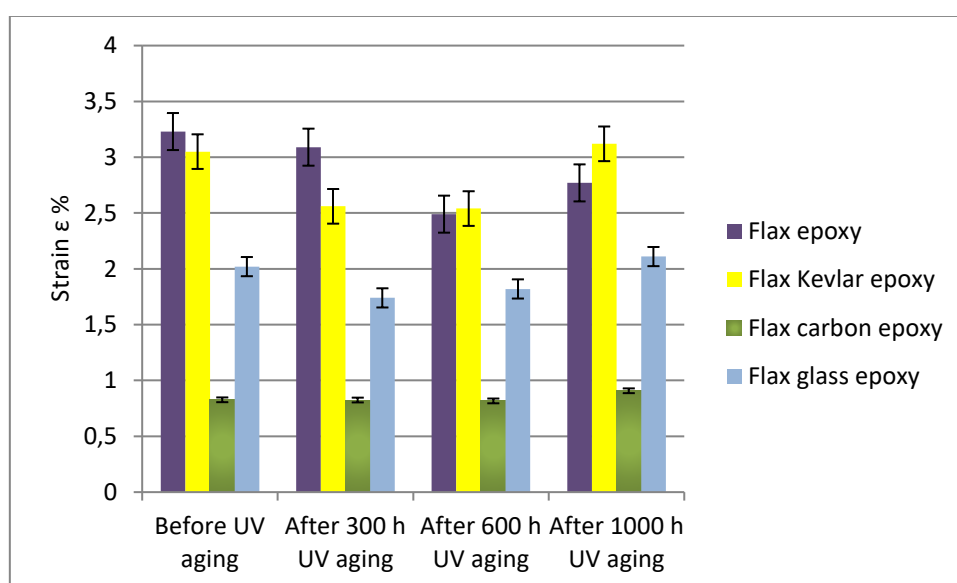


Figure IV.17 : Bending strain variation of different hybrid composites.

Table IV.2 : Flexural Performance of Unaged and UV-Aged Flax-Based Epoxy Composites : A Comparative Analysis.

Materials	Conditions	Flexural properties		
		Eb [Mpa]	σb [Mpa]	εb [%]
Flax/epoxy	Unaged	13664	158.18	3.23
	UV aging for 300 h	12946	148.68	3.09
	UV aging for 600 h	12000	127.71	2.49
	UV aging for 1000 h	9790	116.10	2.77
Flax/glass/epoxy	Unaged	37244	684	2.02
	UV aging for 300 h	34810	556.81	1.74
	UV aging for 600 h	30048	535.73	1.82
	UV aging for 1000 h	25477	480.29	2.11

Flax/carbon/epoxy	Unaged	32871	251.25	0,827
	UV aging for 300 h	29133	229.57	0.825
	UV aging for 600 h	26554	217.02	0.817
	UV aging for 1000 h	21744	197.02	0.908
Flax/kevlar/epoxy	Unaged	17044	225.98	3.05
	UV aging for 300 h	14563	205.49	2.56
	UV aging for 600 h	11880	187.93	2.54
	UV aging for 1000 h	9738	170.84	3.12

Eb: Bending modulus; σ_b : Flexural strength; ϵ_b : Deformation at failure.

Table IV.2 summarises the evolution of the flexural properties of various flax-based epoxy composites before and after UV aging at different exposure durations (300 h, 600 h, and 1000 h). For all composite types flax/epoxy, flax/glass/epoxy, flax/carbon/epoxy, and flax/Kevlar/epoxy a general decline in mechanical properties is observed with increasing UV exposure time. The flexural modulus (Eb), flexural strength (σ_b), and deformation at failure (ϵ_b) decrease progressively as UV exposure increases, indicating degradation of the polymer matrix and fibre–matrix interface under UV irradiation. Notably, the flax/glass/epoxy composite exhibits the highest flexural strength in the unaged state (684 MPa) and retains the highest value even after 1000 h of aging (479 MPa), confirming the superior resistance of glass fibres to UV degradation. The flax/carbon/epoxy composite also shows better durability than the flax/epoxy composite, while the flax/Kevlar/epoxy hybrid maintains a relatively high ϵ_b value (3.12%) after 1000 h, suggesting improved ductility and damage tolerance. Overall, the data underline the benefit of fibre hybridisation in enhancing UV resistance and mechanical performance in flax-reinforced composites.

IV.4. Conclusion

The study highlights the challenges associated with the exposure of flax/epoxy composites and their hybrid counterparts to accelerated aging conditions, particularly UV radiation. The findings offer valuable insights into the mechanical behaviour of these composites based on their composition and suggest pathways for optimisation in outdoor applications. The main conclusions drawn from this work are as follows:

- UV aging has a significant detrimental effect on the mechanical performance of natural fibre composites, notably reducing their structural integrity and durability.

-
- The type of fibre reinforcement plays a decisive role in the stability of composites under UV exposure.
 - Hybrid composites reinforced with high-performance fibres, such as carbon and glass, demonstrate superior resistance to UV-induced degradation compared to flax-only composites.
 - Flax/carbon epoxy composites show promising mechanical stability and are well-suited for semi-structural applications in UV-intensive environments.
 - Flax/glass hybrid composites provide an optimal balance between environmental sustainability and long-term mechanical performance, making them especially appropriate for outdoor applications.

The benefits of hybridisation are evident, but further improvements could be achieved by incorporating surface treatments, UV-stabilised epoxy matrices, or nano-reinforcements to further delay photodegradation.

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Chapter IV

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Conclusion and challenges

Conclusion and challenges

This thesis has explored the potential and limitations of natural fibre-reinforced polymer composites, with a particular focus on biocomposites based on alfa and flax fibres. Through a combination of experimental investigations and numerical simulations, the study has addressed key challenges related to mechanical performance, environmental durability, and structural optimisation.

In the first part of the study, alfa fibres—particularly coarse ones—proved effective in reinforcing polyester matrices, significantly improving mechanical properties when used at optimal content levels. However, environmental ageing, particularly atmospheric exposure over a three-month period, resulted in substantial degradation of these properties. This finding underscores the vulnerability of lignocellulosic fibres to ageing agents and the importance of considering environmental effects in material design.

The numerical simulations carried out using ABAQUS further validated the experimental bending tests and provided critical insights into stress distributions and fracture mechanisms. Notably, the J-integral analysis demonstrated that increased fibre content and coarser fibres contribute to greater resistance to crack propagation, while ageing leads to a marked increase in the J-integral, signalling reduced fracture toughness.

The final part of the thesis focused on flax/epoxy composites and their hybrid variants reinforced with synthetic fibres (carbon, Kevlar, and glass) subjected to accelerated UV ageing. The results confirmed the severe impact of UV radiation on mechanical integrity, with flax-only composites exhibiting the highest degradation. In contrast, hybrid composites—especially those incorporating carbon or glass fibres—showed significantly improved UV resistance and mechanical stability. These outcomes confirm the strategic benefit of hybridisation for enhancing the long-term performance of natural fibre composites in outdoor applications.

Overall, the findings of this work highlight the dual importance of optimising natural fibre characteristics (type, content, and morphology) and improving resistance to environmental ageing for developing durable, high-performance biocomposites. Hybridisation with synthetic fibres emerges as an effective approach to bridging the gap between sustainability and durability. Additionally, numerical modelling has proven to be a powerful tool for understanding complex mechanical behaviours and guiding material design.

This research contributes to advancing sustainable composite technologies and offers practical guidelines for the design of biocomposites suited to real-world applications,

especially in conditions where mechanical performance and environmental exposure are critical factors.

Publications

Annex 1



Investigation of the Fracture Behavior of Alfa/Polyester Composite Using Experimental and Finite Element Methods

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Abstract The importance of creating and using green items made of natural fibers and natural fiber composites rather than conventional goods has increased as awareness of sustainability has grown. *Stipa tenacissima* L., often known as alfa grass, is a tussock grass that is endemic to North Africa and southern Europe. In this work, alfa fiber is used as reinforcement. This study investigated the effects of atmospheric aging through three months on the mechanical properties of alfa fibers composites. Short fibers/polyester composites have been synthesized and characterized successfully with various reinforced ratios of natural fibers (i.e., 0, 10, 20 and 30 wt.%). Using experimental and finite element approaches, this work aims to investigate the fracture analysis of composite materials. The critical stress intensity factor (K_{IC}) and J-integral have been evaluated. The results showed that the alfa coarse fibers with 30 wt.% were capable to enhance the mechanical properties of the polyester/alfa composite. This investigation also revealed that the mechanical properties of composite were notably deteriorated regarding the relative atmospheric aging.

Keywords Fracture toughness · Mode I bending · SENB specimen · Alfa/polyester composite · Integral J

Introduction

When the crack is positioned perpendicular to the applied load, mode I illustrates how the fracture propagates under typical in-plane loading. The fracture toughness values of materials as a function of crack length are often determined using specimens with compact tension (CT) and single-edge-notched bending (SENB). There is now a lot of interest in the creation of high-performance biocomposites made of renewable resources for various uses. Bio-composites are composite materials reinforced by natural fibers, such as agave, hemp and flax, nested inside an ecologically benign or renewable polymeric matrix [1, 2]. The start and propagation of cracks in fiber-reinforced plastic (FRP) composites vary somewhat from traditional isotropic materials due to their anisotropy. Given that fiber volume ratios in FRP composites vary greatly depending on the application and that the fibers and matrix have a broad range of elastic characteristics, the investigation of fracture initiation and propagation becomes case-specific. Many researchers have looked at the fracture behavior of composites reinforced with natural fibers. Zanichelli et al. studied the fracture behavior of a mortar reinforced with date palm fibers [3]. Prasad et al. evaluated the interlaminar fracture toughness of modes I and II while examining the effects of matrix alteration on the interlaminar properties of epoxy composites reinforced with flax fiber [4]. The mixed mode I/II/III translaminar fracture of a natural fiber-reinforced composite system was examined by Zeinedinia et al. [5]. Arul et al. evaluated the mechanical properties and

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Fig. 1 Alfa in natural conditions



fracture toughness of polyester matrix composites reinforced with SLS fiber and coir [6]. The fracture toughness of asphalt concrete reinforced with carbon and kenaf fibers is studied by S. Pirmohammad et al. [7]. The fracture toughness behavior of polypropylene composites reinforced with coconut fiber was investigated by Kumar et al. They discovered that natural coconut coir fiber-reinforced composites outperformed pure polypropylene in terms of mechanical properties and fracture toughness ratings [8]. There are very few references that attempt to study the fracture behavior of alfa fiber-reinforced composites [9–11]. To the best of the authors' knowledge, no studies on influence of atmospheric aging on the mechanical properties of alfa fiber polyester composites were found in the literature. Therefore, the aim of this article is to experimentally investigate and quantify the effect of exposure to environmental aging on the mechanical properties and mode I fracture toughness of alfa/polyester composite reinforced with different mass rates (0, 10, 20 and 30 wt.%) and two categories size (fine and coarse) of short alfa fibers. On the other hand, according to the findings of the experiment, three parameters, such as the proportion of alfa fibers, the size of the fibers and the length of the cracks, are varied to determine the J-integral of alfa/polyester composites before and after natural aging. These natural plant fibers are abundant in North Africa, and are available at very low prices compared to glass fibers.

Experimental Investigation

Materials and Specimen Preparation

Preparation of Alfa Fibers

Alfa fibers were harvested from the region of Bougtob El Bayadh (South-West area of Algeria). Their middle parts were cut in bundles of length 150 mm (Fig. 1). The fibers

were first washed by running tap water over them. It was then first sun-dried for four days. This environmental procedure will eliminate most of the moisture from the alfa fibers, which will make them easier to crush. Following that, they were dried for four hours at 75 °C in a vacuum oven. Alfa stems were then chopped into tiny pieces that were between three and four centimeters in length, and they were crushed using a blade crusher (Fig. 2a). To enhance the fibers' individualization, a blade crusher was used as a mechanical treatment. Secondly, the fibers are sieved with a filter in order to obtain fine size lies between 15 and 20 µm of diameter, see Fig. 2b, and coarse size lies between 70 and 80 µm of diameter, see Fig. 2c. The OPTIKA optical microscope (Model: HDMI Easy-4083. 13E) was used to take optical micrographs of alfa fibers. The micrographs were compared and analyzed to tap out the morphological features such as the size of the alfa fibers. Finally, sodium hydroxide was used in an alkaline treatment to increase the fiber compatibility. The crushed fibers were immersed in NaOH of concentration solution 5 wt.% for 48 h. After immersion, the fibers were rinsed with distilled water to remove the excess of NaOH, and then let it dry for 48 h at room temperature before using.

Bio-Composites Preparation

A suitable hardener accelerator is mixed with polyester resin to create the matrix used in this work. Thus, the alfa fibers were used as fillers for reinforcement. To ensure that all absorbed moisture is removed and that void formation is prevented, alfa fibers were dried using a vacuum at 75 °C for four hours prior to extrusion. Alfa fibers and polyester resin are mixed in three different mass rates (10, 20 and 30 wt.%) and two categories of short fibers size (coarse and fine) in order to elaborate toughness specimens according to ASTM D5045 specifications [12] and 3-point bending specimens according to ASTM D790 standard [13].

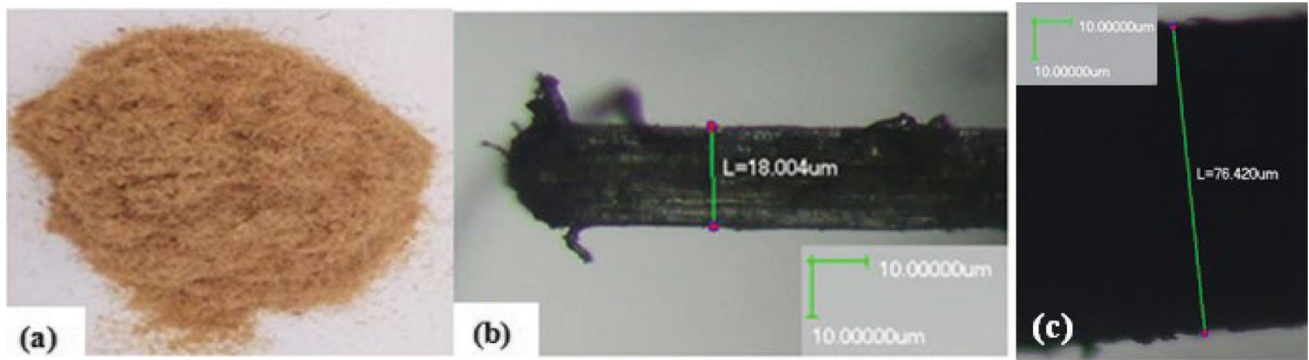


Fig. 2 Preparation of alfa fibers: (a) crushed alfa fibers, (b) fine fibers, (c) coarse fibers

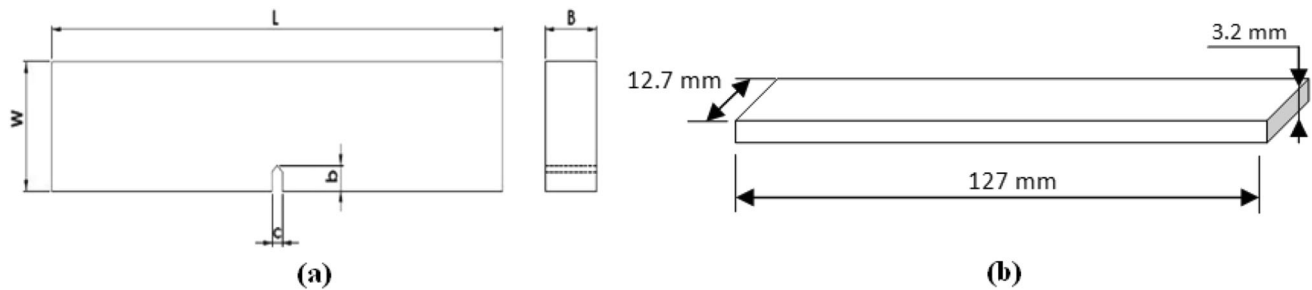


Fig. 3 (a) Setup geometry and dimensions of tested single-edge-notched bending (SENB) samples and (b) 3-point bending specimen

Natural Aging

The environments of Mediterranean weather were considered. However, the sunlight, air and relative humidity (RH) are the key parameters involving in alfa/polyester composite aging processes in the atmosphere. The experiment was started on July 22nd, 2022 for exposure duration of 3 months in Mascara city (North of Algeria). Temperatures range from 15 °C in the morning to 45 °C at 1 p.m. from July 22nd to October 23th and the relative humidity fluctuated between 3 and 65%. Thus, all of sample types were exposed to natural weather environment. Therefore, specimens were removed from the natural environment to perform characterization tests.

Test Setup

In this study, single-edge-notched bending (SENB) and 3-point bending tests were performed according to ASTM D5045-14 Standard [12] and ASTM D790 [13], respectively (Fig. 3a and b). The test setup and sample geometry are shown in Figs. 3 and 4.

The tests were carried out based on 3-point bending geometry. The 3-point bending specimens have sections of 3.2 mm × 12.7 mm and a length of 127 mm. Single-edge-notch-bend (SENB) specimens have a length of 80 mm (4 w) and the thickness was set to 10 mm (B), the notch has a

square shape, the width (c) of the notch was 2 mm, and the notch-depth (b) was set to 4 mm. All the tests were performed at the speed of 0.2 mm/s using tensile machine (BED 100) equipped with a capacity load cell of 1 KN (Fig. 4). The specimens of the non-reinforced polyester and polyester reinforced with different mass rates of alfa fibers (10, 20 and 30 wt.%) were tested under two different conditions: the first under normal conditions and the second after natural aging for 3 months. All the tests were carried out at least five times and were performed at a temperature of 23 °C and 50% relative humidity.

According to the standard test methods for plane-strain fracture toughness and strain energy release rate of plastic materials ASTM D5045-14, the following equations were used to get the critical stress intensity factor (K_{IC}) for the mode I fracture toughness test [12].

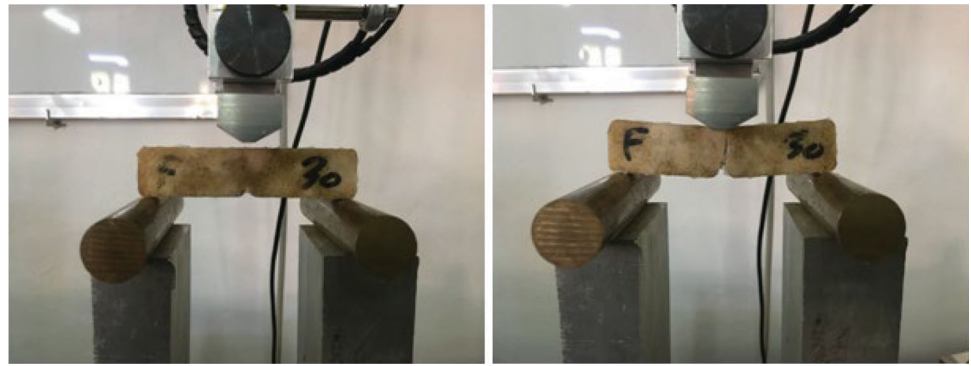
$$K_{IC} = \frac{LPc}{bw^{\frac{3}{2}}} f(\alpha) \quad (\text{Eq 1})$$

$$\text{where } \alpha = \frac{a}{w}$$

$$f(\alpha) = \frac{3}{2} \sqrt{\alpha} \frac{1.99 - \alpha(1 - \alpha)(2.15 - 3.93\alpha + 2.7\alpha^2)}{(1 + 2\alpha)(1 - \alpha)^{3/2}} \quad (\text{Eq 2})$$

where Pc and α are maximum load carried by the specimen and geometric factor, respectively.

Fig. 4 Composite specimens SENB during 3-point bending test



Finite Element Modeling and Analysis

J-Integral Evaluation

A numerical method of the J-integral was utilized in the finite element model used in this study as a criterion of crack propagation and a measure of fracture mechanics safety. Based on the virtual crack extension/domain integral techniques, ABAQUS/Standard provides a procedure for these J-integral assessments. The finite element method offers a practical way to carry out virtual tests and examine how the damage plate behaves in different configurations. It has been one of the most used numerical tools in the fracture mechanics field since the early 1960s. ABAQUS/CAE, a commercial nonlinear finite element package developed by SIMULIA Inc., was used to do the study. The beginning of the cracking was determined using the contour integral approach, which was also required as an output parameter to compute the J-integral at the crack tip.

The theoretical concept of the J-integral was developed, independently, in 1967 by Cherepanov and in 1968 by Rice [14].

When the variation in elastic energy is greater than the variation in surface energy, there is propagation of the crack, which corresponds to a reduction in the free energy of the system. In an infinitely dimensioned plate of ideally elastic material containing a crack, it propagates when:

$$\sigma = \sigma_r = \sqrt{\frac{2E\gamma_s}{\pi a}} \quad (\text{Eq 3})$$

With E = Young's modulus, σ_r = breaking stress and a = crack length.

$2E\gamma_s$ Corresponds to the energy required to create a cracking unit surface. It is in fact a critical energy that we note: G_{Ic} in J m^{-2} .

We can therefore rewrite (3) as follows:

$$\sigma = \sigma_r = \sqrt{\frac{EG_{Ic}}{\pi a}} \quad (\text{Eq 4})$$

The two criteria K_{Ic} and G_{Ic} characterize the sudden propagation of a crack. These two criteria are linked by the relationships:

$$K_{Ic} = \sqrt{\frac{EG_{Ic}}{1 - \nu}} \quad (\text{Eq 5})$$

With ν = Poisson's ratio

For isotropic materials exhibiting a marked ductile/brittle transition, the J-integral can be directly related to the ductile failure mode. In the case of a plane deformation under loading conditions corresponding to mode I, the relationship is given by:

$$J_{Ic} = G_{Ic} = K_{Ic}^2 \left(\frac{1 - \nu}{E} \right) \quad (\text{Eq 6})$$

With G_{Ic} : the relaxation factor of the critical strain energy, K_{Ic} : the stress intensity factor in ductile failure under loading in mode I, ν : the Poisson ratio and E the Young's modulus of the material.

The energy release associated with the crack growth can be quantified using this integral when considering a path Γ that encloses the crack tip and has starting and ending points that rest on the two fractured faces. It has the following definition:

$$J = \int_{\Gamma} r \left\{ w_e n_1 - \sigma_{ij} n_j \frac{\partial u_i}{\partial x_1} \right\} ds \quad (\text{Eq 7})$$

where w_e , an open contour around the tip of the crack, represents the elastic deformation energy density. Along the crack's axis, this is supposed to be rectilinear.

It notice by: $\vec{n}$ The normal outside the contour, $\sigma_{ij} n_j$: The contour's stress and the corresponding displacement: u_i (Fig. 5).

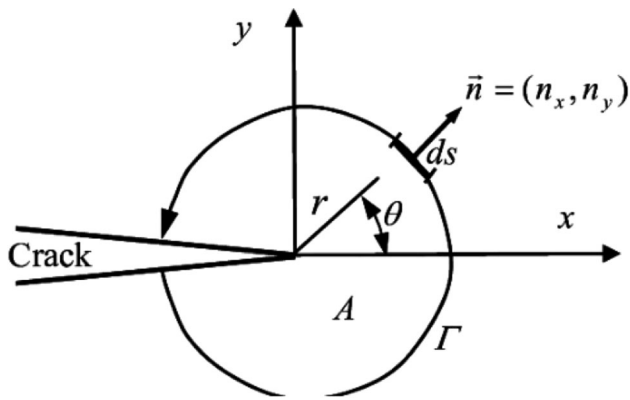


Fig. 5 Contour integration path around crack tip

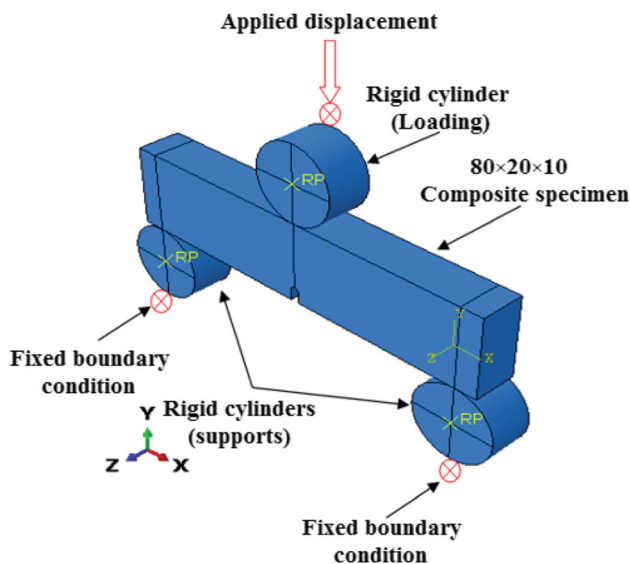


Fig. 6 Loading and boundary conditions applied to the model

Materials Properties and Loading Conditions

According to the mechanical testing, finite element models of SENB specimens were made to investigate the fracture and stress–strain curves of the polyester resin reinforced with different mass rates of two sizes of alfa fibers (coarse and fine). The geometry of bend mechanism is shown in Fig. 6, the analysis was performed using ABAQUS/explicit program. It was anticipated that all materials would behave in a nonlinear, homogenous, elastic/plastic manner (Tables 1 and 2). The boundary conditions have been defined in such a manner as to reproduce as well as possible the real conditions of the test. In fact, there are three boundary conditions applied to the model, the contact lines of the sample with the supports are blocked in translation along x , y and z ($U_x = U_y = U_z = 0$). The displacements of the punch are locked along y and z ($U_y = U_z = 0$), in shear, all models were loaded to failure using 0.001 mm displacement increments.

Finite Element Model

Numerous materials include the physical discontinuities that are commonly referred to as notches. When a material with a notch experiences load, tiny fissures may start to show up. These fissures may then combine with other small ones to produce a larger macroscopic crack, which spreads until the material completely ruptures. Determining the criteria of rupture for various stress conditions is necessary for estimating the performances of composites. By computing the J-integral at the crack front, the fracture behavior of the SENB specimens was investigated (Fig. 7). Based on a mesh convergence analysis, the generated model is meshing with 8-node linear brick (Hex) elements of type (C3D8R). ABAQUS, however, suggests that elements for the combination for three-dimensional contact analysis be (C3D8R). A study on mesh convergence was carried out, processing a number of runs with constant boundary conditions. There are 29546 nodes and 25790 elements in the entire finite element model. The mesh around the crack front has been sufficiently refined to predict the plastic zone and calculating the opening mode of J-integral with good precision (Fig. 8).

Results and Discussion

Experimental Results

Toughness presents a material property which states the material's capability to resist crack propagation. It is characterized by the mechanical field of the fracture process (considers the presence of defects) and appears when it recalls that the fracture is a failure mode governed by cracking (created, initiated and propagated). For all cases, it can be seen that the mode I fracture path was stable and grows proportionally traversing the notch plane in a straight line. Load–displacement curves under mode I fracture test of unaged and aged polyester/alfa composite with different reinforcement mass rates (0, 10, 20, 30 wt.%) are shown in Figs. 9 and 10, respectively (Fig. 11). During the manufacture of the samples, porosities are created. These porosities lead to a dispersion of results and affect the mechanical and fracture properties of composites (Fig. 12). The results obtained for the mechanical and fracture proprieties of the different composites are summarized in Tables 1 and 2.

Effect of Fiber Size

The effect of fiber size on the mechanical properties investigated in this study is highly significant as shown in Tables 1 and 2. In general, increasing fiber size improves

Table 1 Summary of the mechanical properties of alfa/ polyester composite reinforced with fine fibers

	Unaged composite			Aged composite		
	σ_{ultime} (MPa)	E (MPa)	K_{IC} (MPa m ^{0.5})	σ_{ultime} (MPa)	E (MPa)	K_{IC} (MPa m ^{0.5})
Neat matrix	25.24 ± 2	1202 ± 51	0.52 ± 0.02	18.40 ± 1.8	1001 ± 30	0.40 ± 0.01
10 wt.% fibers	27.77 ± 2.7	2003 ± 64	0.57 ± 0.03	20 ± 1.83	1265 ± 57	0.45 ± 0.01
20 wt.% fibers	40.34 ± 2.9	2200 ± 87	0.75 ± 0.05	20.3 ± 1.9	2024 ± 113	0.54 ± 0.02
30 wt.% fibers	42.2 ± 3.2	4000 ± 180	0.88 ± 0.07	31 ± 2.2	2850 ± 157	0.68 ± 0.04

Table 2 Summary of the specific mechanical performance of alfa/polyester composite reinforced with coarse fibers

	Unaged composite			Aged composite		
	σ_{ultime} (MPa)	E (MPa)	K_{IC} (MPa m ^{0.5})	σ_{ultime} (MPa)	E (MPa)	K_{IC} (MPa m ^{0.5})
Neat matrix	25.24 ± 2	1202 ± 51	0.52 ± 0.02	18.40 ± 1.8	1001 ± 30	0.40 ± 0.01
10 wt.% fibers	30.25 ± 2.9	2220 ± 70	0.62 ± 0.03	22.63 ± 1.9	1710 ± 57	0.51 ± 0.02
20 wt.% fibers	40.72 ± 3.1	3200 ± 101	0.79 ± 0.05	29.8 ± 2.1	2310 ± 91	0.62 ± 0.03
30 wt.% fibers	53.43 ± 3.6	6010 ± 203	0.97 ± 0.08	39.13 ± 2.8	3480 ± 121	0.74 ± 0.05

the modulus of elasticity and maximum strength in flexural tests. This result is consistent with previous reports on wood-fiber thermoplastic composites [15–17]. However, flexural Young modulus shows a steady increase with increase in fibers size at 30 wt.% content (Tables 1 and 2). It rises from 4 GPa at fine fiber sizes to 6.01 GPa at coarse fiber sizes.

Flexural strength development also demonstrates that fiber size has greater influence at higher fiber load (30 wt.%), with approximately 26.61% higher strength when fiber size increases from fine to coarse. On the other hand, the incorporation of alfa fibers in polyester matrix steadily increases toughness by 19.23, 51.92 and 86.54% at 10, 20 and 30 wt.% coarse fiber content, respectively, and by 9.61, 44.23 and 69.23% at 10, 20 and 30 wt.% fine fiber content, respectively. The phenomenon is more marked as fiber size increases. Because cracks travel around the alfa fibers, the fracture surface area increases with increase in fiber size.

Effect of Fiber Content

As shown in Tables 1 and 2, the effect of alfa fiber content is highly significant for all tested properties of unaged composites. In general, high mass ratio of fibers yields materials with high Young's modulus, flexural strength and toughness.

The results, illustrated in Table 1, show that the Young's modulus, flexural strength and toughness of the unreinforced polyester were initially 1202 MPa, 25.24 MPa and 0.40 MPa m^{0.5}, respectively. The addition of fine alfa fiber reinforcement to polyester matrix improved the Young's modulus, the flexural strength and the toughness. The values recorded for Young's modulus, the flexural strength

and toughness at 30 wt.% were 4000 MPa, 42.2 MPa and 0.88 MPa m^{0.5}, respectively, which represent increases of 232.78, 67.19 and 69.23%, respectively.

The results, illustrated in Table 2, show that the highest elastic modulus, flexural strength and toughness (6010 MPa, 53.43 MPa and 0.97 MPa m^{0.5}, respectively) were obtained using samples reinforced with 30 wt.% coarse alfa fibers, which represent increases of 5 times, 2.12 times and 1.86 times, respectively, compared with those of pure polyester.

On the other hand, the incorporation of short alfa fibers in polyester matrix steadily increases toughness by 19.23, 51.92 and 86.54% at 10, 20 and 30 wt.% coarse fiber content, respectively, and by 9.61, 44.23 and 69.23% at 10, 20 and 30 wt.% fine fiber content, respectively. These results are in good agreement with previously reported data [18–20].

Effect of Environmental Aging

The test results showed that the highest degradation for Young modulus was recorded at 30% for coarse and fine fibers, respectively, of 29.82, 38.52 and 72.41% for Young modulus at 10, 20 and 30 wt.% coarse fiber content, respectively. 58.34, 8.7 and 40.3% at the same fine fiber contents.

However, for the flexural strength a degradation of 36.54% was recorded at 30 wt.% of coarse fibers against 36.13% at 30 wt.% of fine fibers content. According to the results showing in Figs. 10 and 13, a similar trend was observed for the toughness according to the results showing in Figs. 10 and 13, the toughness properties of the composites alfa/polyester were affected by environmental

Fig. 7 3D model with the direction of the fracture

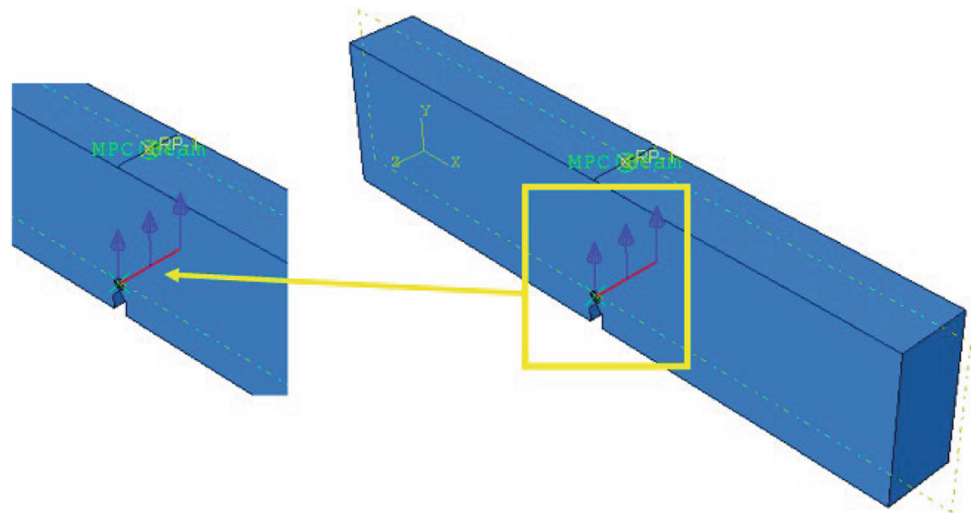
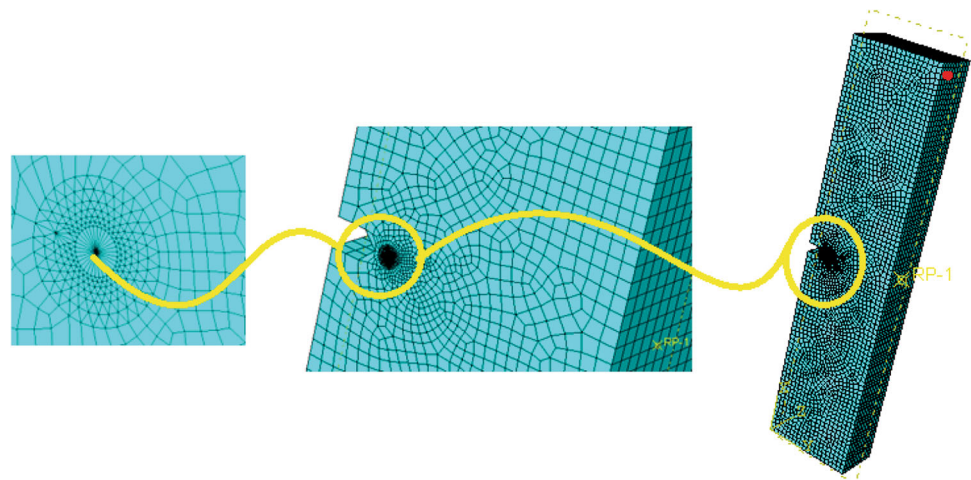


Fig. 8 The finite element mesh model: The SENB specimen, mesh around the crack emanating the notch and mesh around the crack tip (right to left)



aging. For all categories of composites, the cracking resistance of the specimens was considerably decreased for three months of aging in a particular environment. A degradation of 31 and 29% was recorded at 30 wt.% of coarse and fine fibers, respectively. These results are in good agreement with previously reported data [21–25]. This decrease in properties can be explained by the biodegradability of alfa fibers, one of the constituents of the composite and the sensibility of polyester resin to UV rays (Fig. 14).

Pattanaik et al. [21] found that the mechanical behavior of polymer composite degraded with respect to aging duration. Nicholas et al. [22] reported that the mechanical and physical properties of several polymer composite materials are significantly influenced by aging conditions, such temperature, pressure, humidity and curing conditions. As a result, it is considered that humidity and heat aging are the main reasons why composites exposed to the environment degrade over time. However, Hammiche et al.

[23] showed that several consequences of atmospheric aging, namely the degradation of the mechanical properties of the matrix, the damage at the matrix/reinforcement interface and the differential swelling associated with concentration gradients. The subsequent effects of atmospheric aging contribute to the loss of mechanical properties of the composite polyester/alfa; the properties degradation can be explained by the sensitivity of the polyester resin to ultraviolet (UV) rays, the biodegradability of alfa fibers.

Finite Element Evaluation of J-Integral with Crack Propagation on Alfa Composite

Effect of the Fiber Proportion, Fiber Size and the Crack Length

The change in the value of the J-integral throughout a crack's length for an unaged composite alfa polyester with

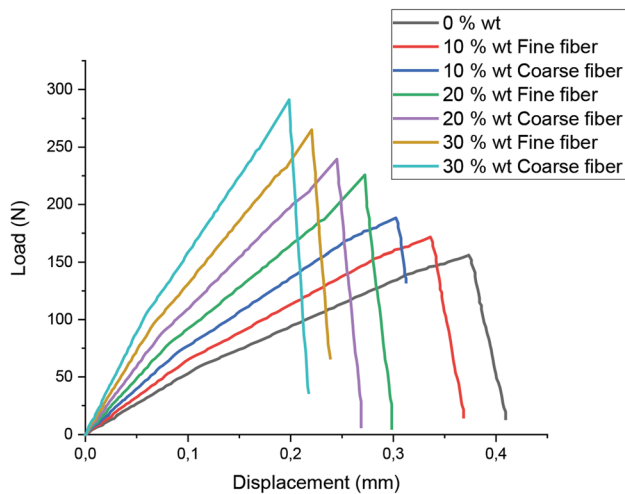


Fig. 9 Load–displacement unaged polyester/alfa composites curve with different fractions of reinforcement (0, 10, 20, 30 wt.%) for both sizes of alfa fibers (fine and coarse)

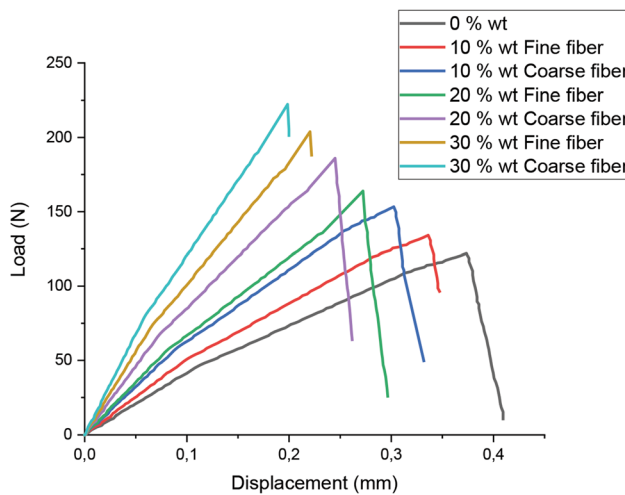


Fig. 10 Load–displacement aged polyester/alfa composites curve with different fractions of reinforcement (0, 10, 20, 30 wt.%) for both sizes of alfa fibers (fine and coarse)

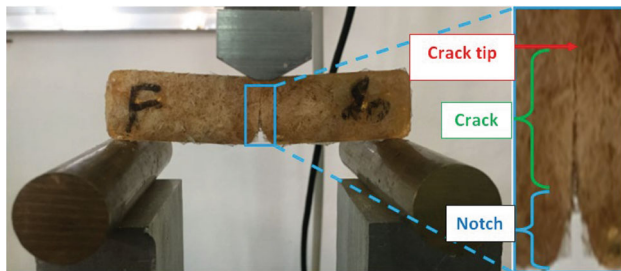


Fig. 11 Crack propagation under mode I fracture

fine and coarse fibers under mode I loading is shown in Fig. 15. One of the principal geometrical characteristics of the fracture is the length of the crack. The J-integral was

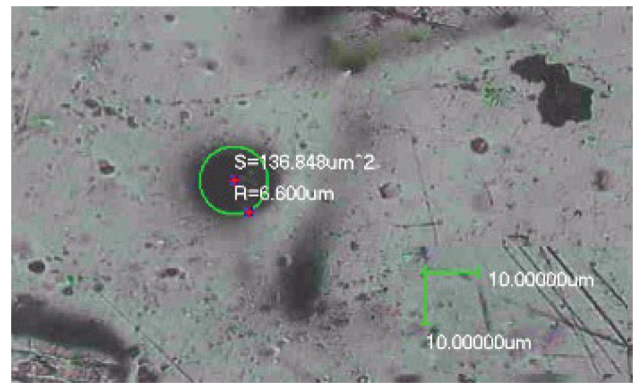


Fig. 12 Optical microscope observation of alfa/polyester composite (porosities observed in material)

computed for different crack lengths, preliminary at $a = 1$ and increasing to 6 mm. In all categories of composites, it has been observed that the value of J-integral rises as the crack length grows. The results further indicated that the fiber content has a considerable impact on the J-integral. This figure demonstrated that as the percentage of alfa fibers in the polyester/alfa composite increased, the J-integral decreased. This implies that the energy at the crack tip is significantly reduced, and the fatigue life of the structure can be enhanced. Shear forces cause the load to be transferred into the matrix through the reinforcement. The augmentation of the reinforcement in the composite decreases the crack tip's shear stress. Consequently, decreasing the plastic zone around the crack tip (Fig. 14), which is consistent with a decrease in the J-integral. As the fiber's ratio in composite increases, the crack propagation decreases. Thus, it can be concluded that adding more alfa fibers to a composite will greatly enhance its reinforcing properties and increase its resistance to crack propagation. On other hand, the fiber size is expected to have important consequences on the values of J-integral of the composites. The coarse fiber size in the composites is able to provide a highest resistance to the fracture and crack opening than the fine fibers.

Effect of Natural Aging

Figure 16 illustrates how the J-integral varies based on the size of the crack in the polyester/alfa composite affected by atmospheric (1–6 mm of crack length with increment of 1 mm). For all cases of composite, the J-integral was considerably increased for three months of aging in a particular environment. The lowest values are noted for the case where the composite is reinforced with 30 wt.% of coarse and fine alfa fiber when a length of crack $a = 1$ mm. It has been observed that the J-integral of the composite reinforced with 30 wt.% of coarse and fine alfa fiber is about 3.20 and 3.88 J/m², respectively. These values were

Fig. 13 Variation of fracture toughness (K_{IC}) of different polyester/alfa composites (coarse and fine size)

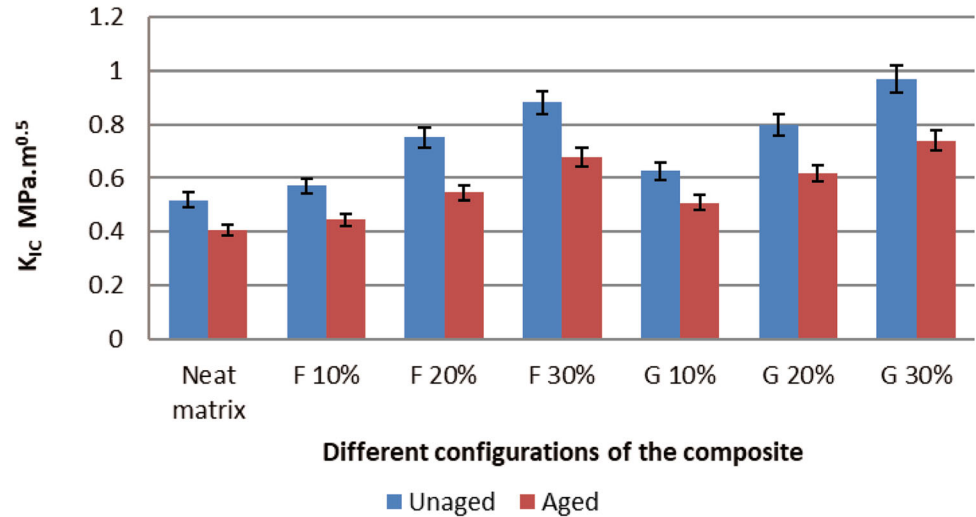


Fig. 14 A three-dimensional representation of the fractured composite

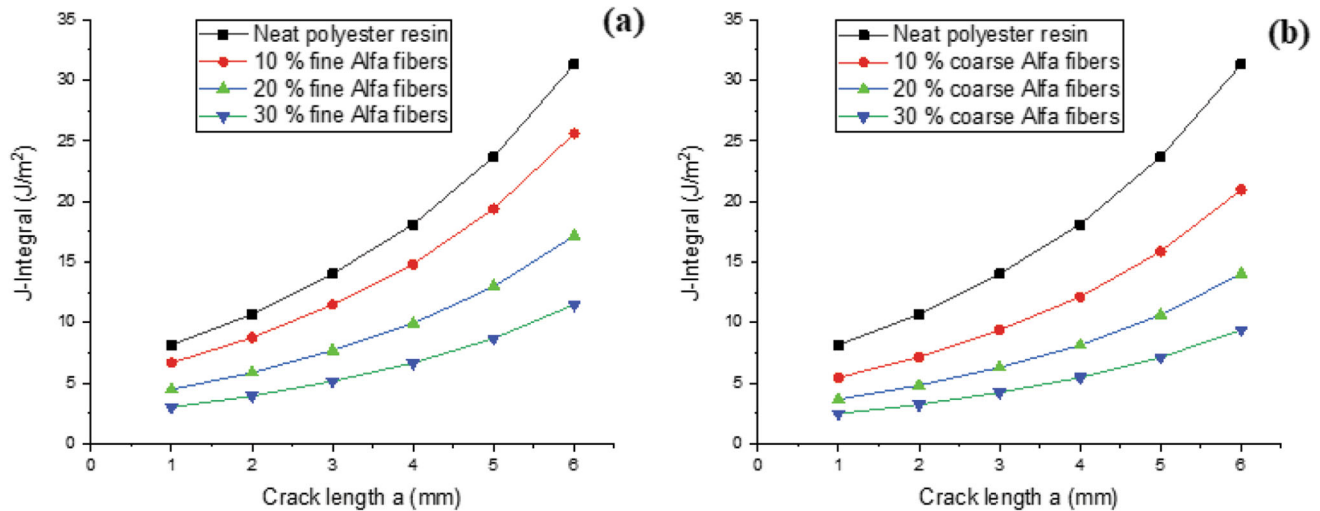
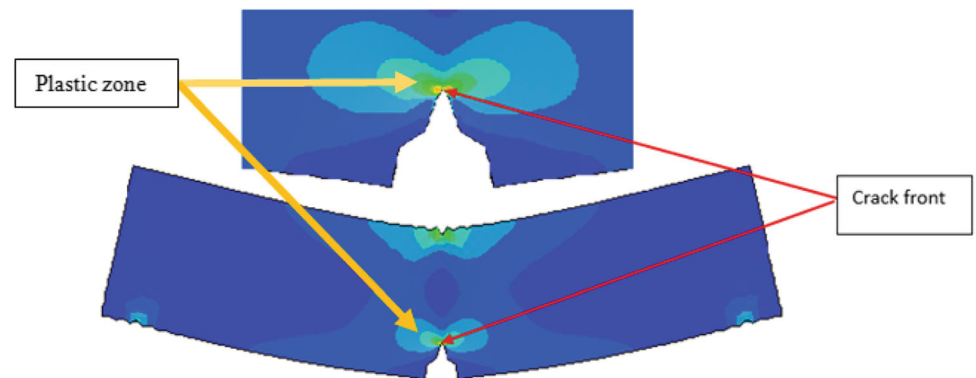


Fig. 15 J-integral versus crack length for young composite alfa polyester with fine (a) and coarse fibers (b)

increased to 12.31 and 14.93 J/m² for a crack length $a = 6$ mm. The values of J-integral at the crack tip were about 25% higher than those of the unaged polyester/alfa

composite. These results confirm that the atmospheric aging degrades the mechanical properties of composites and in particular the resistance of fracture.

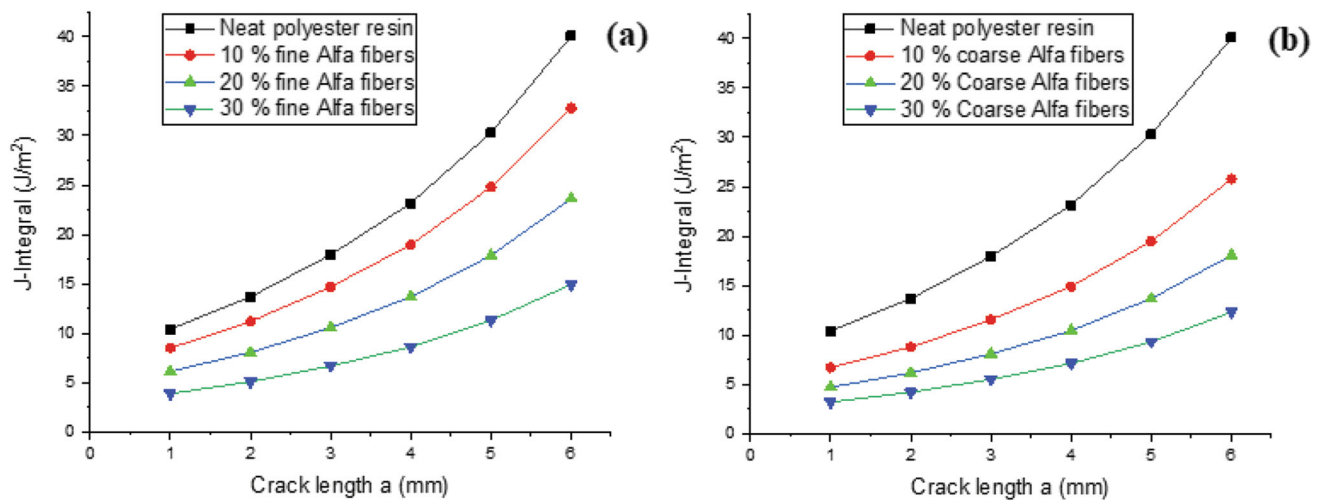


Fig. 16 J-Integral versus crack length for aged composite alfa polyester with fine (a) and coarse fibers (b)

Conclusion

In this study, three-month periods of aging settings were applied to polyester/alfa composites. In the investigation, it was found that environmental aging and filler percentage had a significant impact on composite materials properties. Therefore, the current research allows for the deduction of the following findings:

- Alfa fibers have the potential to be used as polymer matrix reinforcement.
- The resistance of the composite reinforced with coarse esparto fibers is more effective than that of fine fibers.
- The increase in the percentage of alpha fibers was beneficial for improving the mechanical properties of the composites.
- The J-integral in the composite structure increased noticeably as the crack's size increased; however, the values of the J-integral in the polyester/alfa composite reinforced with 30% of alfa fibers are significantly lower.

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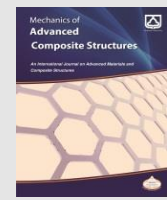
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Annex 2



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Research Article

Experimental and Numerical Analyses of Alfa/Polyester Composite Under Three-Point Bending

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ABSTRACT

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Keywords:

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This study investigated the effects of atmospheric aging through three summer months on the mechanical properties of polyester reinforced with different mass rates of alfa fibers (Stipa Tenacissima). For this purpose, three-point bending tests were performed on pure polyester and polyester /alfa fiber composite specimens. A finite element model of flexural testing was developed to analyze the mechanical behavior of the Alfa/polyester composite. The test results showed that the alfa coarse fibers with 30 wt % were capable of enhancing the mechanical properties of the polyester/alfa composite.

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1. Introduction

Polyester resins are now widely employed in a variety of materials engineering disciplines, including aerospace composite material matrices, adhesives, sealants, and protective coatings. Epoxy resins have become a good material for engineering applications due to their high chemical and corrosion resistance, good mechanical and thermal qualities, flexibility, and good electrical properties. In comparison to other resins, such as urethanes and epoxies, it is also reasonably priced, and in many cases, it provides better performance. It

also has amazing physical and chemical qualities [1].

Vegetable fibers have seen a sharp increase in application as reinforcement for plastic composite materials during the past few years across all industrial sectors, including the aerospace and automobile industries [2-5]. Alfa grass is a kind of tussock grass that grows in semi-arid and dry parts of southwestern Europe and North Africa [6]. Alfa is a Mediterranean plant with a heterogeneous structure consisting mainly of cellulose (40–50%), lignin (between 17.71 and 24%), hemicellulose (from 22.15 to 28%), and 5% wax [7]. Alfa fibers have captivated researchers and engineers in various

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fields such as packaging, construction, biomedical, and automotive industries. Moreover, Alfa fiber-based composites are important to the development of high-performance engineering materials because of their easy availability, recyclability, and environmental friendliness. Alfa fibers have recently been employed in a few research areas to strengthen thermoplastic and thermosetting polymers [8 - 11].

Analyzing these resources' mechanical behavior is crucial for efficient usage and exploitation. The study of the bending behavior of composites derived from biological sources has garnered a great deal of interest in the last several years. The goal of Bahloul et al, research was to determine the impact of Alfa fibers on cement mortar's mechanical and thermal resistance [12]. The flexural properties of starch-based composites reinforced with alfa grass are examined by Espinach et al. [13]. Khaldi et al investigated the mechanical characteristics of anisotropic Alfa vegetable fibers and epoxy resin, as well as the viscoelastic properties of the interphase between the matrix and Alfa fibers, using an experimental approach and a numerical model [14]. Composites made of Alfa fibers and polyethylene with a high density also showed a significant improvement in tensile strength [15]. Ajouguim et al. investigate the viability of adding raw Alfa fibers to cement mortar in a mildly prepared form. They look at how the mortar's mechanical and physical characteristics have changed as Alfa fibers have been added [16].

This research aimed to examine the mechanical behavior of polyester resin reinforced with different mass rates of alfa fibers (10, 20, and 30 wt.%) at different sizes (fine and coarse) of short Alfa fibers under atmospheric aging conditions. Based on the mechanical tests, compared to pure polyester. The coarse and fine fibers were used to realize the composites polyester/alfa. A finite element model of bending test specimens was created to analyze the fracture behavior of the polyester/alfa composites.

2. Materials and Methods

All the specimens were composed of Alfa fibers and polymer (polyester resin) matrix composite. Alfa fibers were harvested from the region of Bougtob El Bayadh (South-West area of Algeria) in October 2020. The matrix used is Unsaturated Polyester Resin (UPR) supplied by Concordal SPA (Algiers, Algeria), and obtained by a mixture of primary resin with a Methyl Ethyl Ketone Peroxide (MEKP) polyester catalyst, it is a chemical compound used in the polymerization of unsaturated polyester resins.

MEKP acts as a powerful catalyst, promoting the polymerization reaction and hardening the composite material. With a weight ratio of 100/2 (For 100 g of resin 2 g of hardener is added).

2.1. Preparation of Alfa Fibers

Based on reference [17], the fibers were first cleaned under tap water to remove surface contaminants. Then, first, it was sun-dried for four days. This environmental process will remove most of the moisture in the Alfa fibers, making them more straightforward to crush. After that, they were dried in a vacuum oven for 4 h at 75°C. Next, Alfa stems were divided into tiny pieces of 3-5 cm length before being crushed with a blade crusher. Figure 1(a).

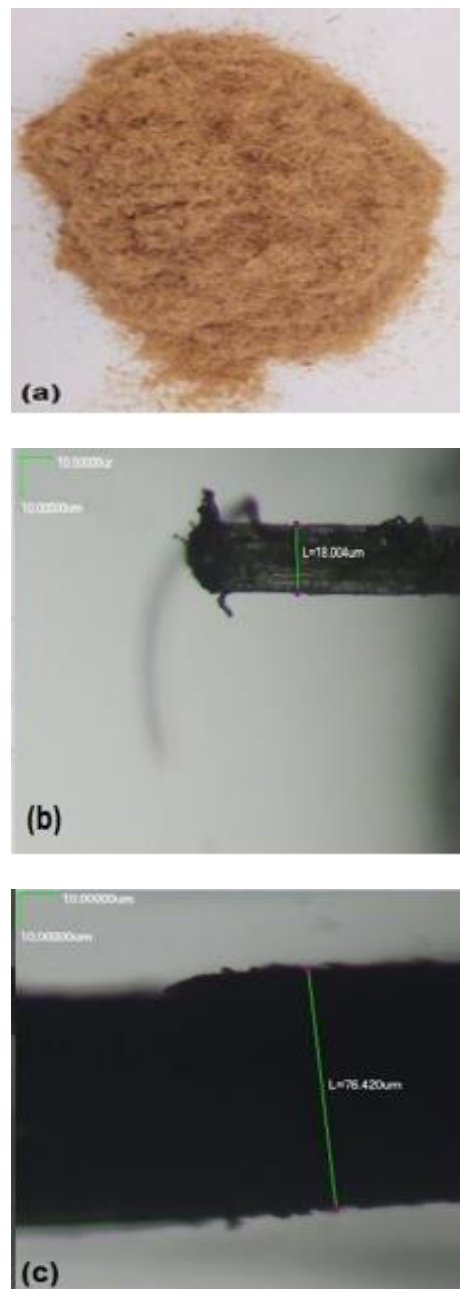


Fig. 1. Alfa fibers: (a) Crushed Alfa fibers, (b) Fine fibers, (c) Coarse fibers.

A blade crusher was used for the mechanical treatment to enhance the fibers' individualization. Secondly, the fibers are sieved with a filter to obtain a fine size lies between 15 and 20 μm in diameter, see Figure 1 (b), and coarse size lies between 70 and 80 μm in diameter, see Figure 1 (c). Optical micrographs of coarse and fine Alfa fibers were photographed on the optical microscope, OPTIKA, Model: HDMI Easy-4083. 13E. Finally, Sodium hydroxide was used in an alkaline treatment to increase fiber compatibility. For 48 hours, the crushed fibers were immersed in a 5wt% concentration solution of NaOH. After immersion, before being used, the fibers were dried at room temperature for 48 hours after being washed with fresh water to remove any remaining NaOH.

2.2. Preparation of Polyester/Alfa Composites

As reinforcing fillers, two varieties of 5% alkali-treated Alfa fibers (coarse and fine sizes) were employed. Alfa fibers were vacuum-dried for four hours at 75 °C to eliminate any absorbed moisture and stop the creation of voids before the extrusion procedure. The initial step in creating complex composite specimens is to combine Alfa fibers with polyester resin (UPR) for three distinct fiber weight ratios (10, 20, and 30 wt%). Then, the mixture of polyester resin/Alfa fibers was injected into a mold using injection-compression molding under a pressure of 3 bars for 24 hours at room temperature. The length, width, and thickness of bending specimens are 140 x 20 x 4.7 mm³, respectively (Figure 2).



Fig. 2. Polyester/Alfa composite specimens for bending test.

2.3. Natural Aging

The environments of Mediterranean weather were considered. As, sunlight, air and relative humidity (RH) are the key parameters involved in Alfa/polyester composite aging processes in the atmosphere.

The experiment was started on July 23rd 2022 for an exposure duration of 3 months in Mascara City (North of Algeria). Temperatures range from 15 °C in the morning to 45 °C at 1 p.m. from July 23rd to October 23rd and the relative humidity fluctuated between 3% and 65%.

2.4. Bending Tests

Three-point bending tests were performed on a BED 100 tensile testing machine with a capacity of 1KN equipped with a test speed control system (Figure 3).



Fig. 3. Bending testing machine.

All the tests were performed at the speed of 6 mm/min. Flexural test specimens of the non-reinforced polyester and polyester reinforced with different proportions of Alfa fibers (10, 20, and 30 wt %) were tested under two different conditions: the first under normal conditions and the second after natural aging for 3 months. For each proportion of Alfa/polyester composite, two sizes of Alfa fibers (Coarse and fine) were used.

The flexural tests were performed according to the ASTM D 790 standard. The dimensions of the bending specimens were 140 mm in length, 20 mm in width, and 4 mm in thickness, and the distance between supports was 110 mm (Figure 4).

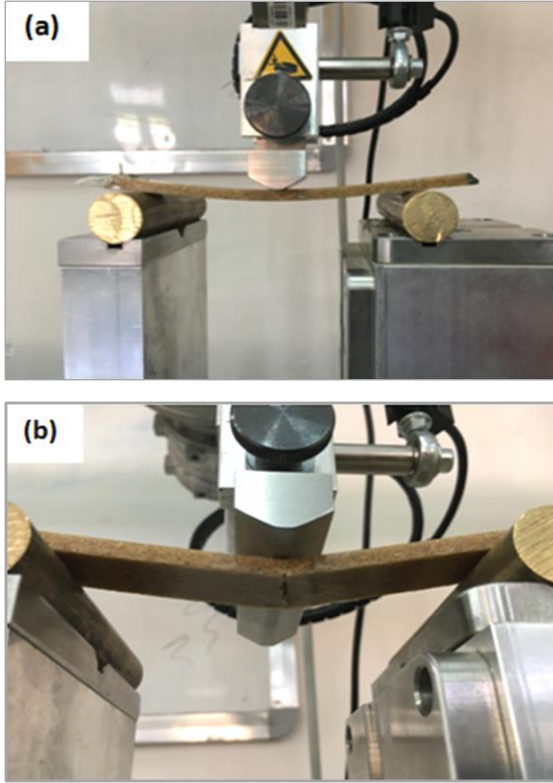


Fig. 4. Sequence of the experimental 3-point bending test until failure: (a) Initial state, (b) Final state.

The resulting stress and strain for a rectangular sample under a load in a three-point bending setup is given by the formulas (equations 1 and 2) below:

$$\sigma = \frac{3FL}{2bd^2} \quad (1)$$

$$\varepsilon = \frac{6Dd}{L^2} \quad (2)$$

where F is the load (N), L is the length of the support span, b is the width d is the thickness of the bending specimen and D is the maximum deflection of the center of the beam.

The Timoshenko beam theory (equation 3) provides the value of the deflection w :

$$w = \frac{Fl^3}{4Ebh^3} = \frac{Fl^3}{48EI} \quad (3)$$

Equation 4 provides the resultant Young's modulus:

$$E = \frac{dF}{dw} = \frac{l^3}{48I} \quad (4)$$

2.5. Finite Element Analysis

Considering the mechanical testing, finite element models of bend specimens were developed to analyze the fracture and stresses-strain curves of the polyester resin reinforced with different volumes of two sizes of alfa fibers (coarse and fine), and commercial benchmark. The geometry of the bending mechanism is shown in Figure 5, the analysis was performed using the ABAQUS/explicit program [18].

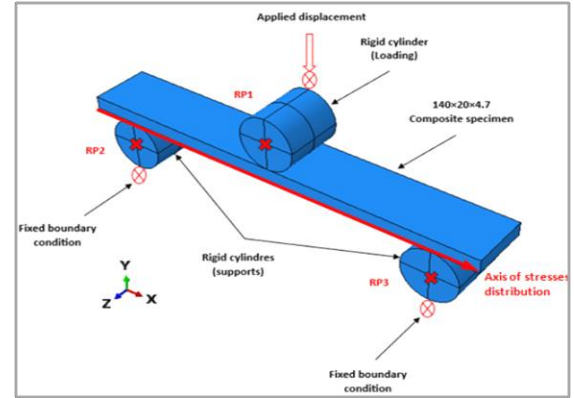


Fig. 5. Loading and boundary conditions applied to the model.

2.5.1. Validation of Numerical Bending Model

Numerical modeling is considered a validation of the experiment findings. Numerical computes are carried out in three dimensions, using the ABAQUS software, based on finite element development. Among the variety of elements available in the ABAQUS documentation, The boundary conditions have been defined in such a manner as to reproduce as well as possible the real conditions of the test. In fact, there are three boundary conditions applied to the model, the contact lines of the sample with the supports are blocked in translation along x , y , and z ($U_x=U_y=U_z=0$). The displacements of the punch are locked along x and z ($U_x=U_z=0$), with the application of a displacement in the y direction. All models were loaded to failure using 0.001 mm displacement increments in shear.

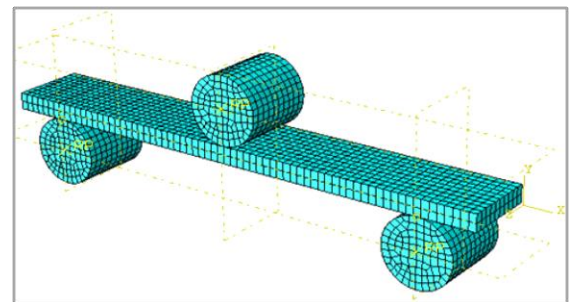


Fig. 6. The finite element mesh model (sample, punch, and supports).

For all cases under bend loading conditions, the finite element analysis established the distribution of von Mises stresses and shear stresses vs strain in the composite (Figure. 6).

2.5.2. X-FEM and Fracture Criteria

To pretend the crack nucleation and growth numerically we applied the X-FEM in the polyester/alfa composites. The criterion of the maximum principal stress (σ_1) was chosen for the damage initiation as specified in Eq. (5) [19]. However, when the maximum principal stress becomes bigger than the tensile strength of the composite material, the crack presence can be seen, and the factor f^e refers to the stress ratio in an element (e):

$$f^e = \frac{\sigma_1^e}{\langle \sigma_{max}^* \rangle} \quad (5)$$

Fracture energy G_c shows the criterion of crack propagation. In addition, supplementary numerical parameters are necessary to ensure the computation convergences which are 0.01 for Damage Initiation Tolerance and $10e-05$ for the viscosity coefficient and the dissipated energy fraction. This value allowed the calculation in a reasonable time without influencing the final result (the viscous dissipation energy remains much lower than strain energy during the whole calculation). Hence, only 1400 elements of the specimen mesh have been enriched, the head being subjected to a concentrated force which enrichment would cause inadequate cracking.

2.5.3. Finite Element Model

A numerical simulation of the bending test was performed in order to replicate laboratory test results. Numerical analysis was performed using the finite element method (FEM) with Abaqus explicit. Three-dimensional finite element models of specimens made of alfa/polyester composite were created based on mechanical testing in order to assess the stress fracture in polyester reinforced with varying volumes of alfa fibers for benchmarking purposes. Flexural simulations were conducted on Alfa composite materials using finite element analysis (FEA) software ABAQUS 2022. This software was utilized to generate the model and refine the finite element specifics, including contact properties, boundary conditions, and meshing the details of numerical modeling. The dimensions used in these simulations were identical to those employed in the experiments conducted in the current study. The geometrical model is composed of a plate composite, of the following size: length, $L = 140$ mm, width, $w = 20$ mm, thickness, $e = 4.7$ mm. Three pins 19 mm in

diameter were in contact with the specimen to apply the three-point bending loading. The supports and puncher were modeled using discrete rigid elements. Figure 5 displays the finite element model. The mechanical properties of the composite were derived from the experimental results obtained and are shown in Table 1&2. The Maxps damage model is used to simulate material damage when the material properties are simplified to linear elasticity. The puncher and the two supports are designed to be rigid without regard to the setting of its material properties. For a reasonable computational time, the stable time increment was $1E-9$. The creation of operation points RP-1, RP-2, and RP-3 allowed for the more reasonable setting of "load" and "boundary conditions." Three special points are defined and bound to three rigid bodies in the model. The puncher was linked to the point RP-1 and configured as a "rigid body." The left and the right supports were similarly configured as a "rigid body" and bound to the points RP-2 and RP-3 respectively. A reference point (RP-1) was established in the middle of the FE model's top surface, as shown in Figure 5. The direct cyclic was the step process. The boundary condition of "Displacement/ Rotation" was assigned to the composite model's bottom surface. The interaction between the loading puncher and the specimen is assumed to have general contact, as is the interaction between the supports and the specimen. The puncher was allowed to move freely along the z-axis, which is perpendicular to the face of the composite plate. An imposed displacement "U" is applied longitudinally to the specimen. In fact, there are three boundary conditions applied to the model, the contact lines of the sample with the supports are blocked in translation along x, y, and z ($U_x=U_y=U_z=0$). The displacements of the punch are locked along x and z ($U_x=U_z=0$), with the application of a displacement of 10 mm in the y direction ($U_y=10$ mm), in order to simulate the loading. The composite component was loaded with displacement increments of 0.01 mm in shear until it failed. Tables 1 and 2 show the material attributes that were used, which were expressed in terms of yield strength, yield strain, Young's modulus, and Poisson's ratio for all related components. All materials were assumed to exhibit homogeneous materials. Based on a mesh convergence study, hexahedral elements of type C3D8R are used to mesh the generated model. For two-dimensional contact analysis, however, ABAQUS advises the use of C3D8R hexahedral elements for the combination. The element type of each component is set to C3D8R. The Alfa composite specimen's "approximate global size" was set to 1 mm, while the puncher

and supports were set to 2 mm. A study on mesh convergence was carried out, processing a number of runs with constant boundary conditions. There are 1400 elements and 2343 nodes in the entire finite element model. The numerical analysis determined the bending stresses versus strain and von Mises stresses distribution in the composite for all cases under pure bend loading conditions.

3. Results and Discussion

In this investigation, the mechanical behavior failure of polyester reinforced with different mass rates of alfa fibers was analyzed. The stress-strain curves exhibit the bend loading response of the non-reinforced polyester and polyester reinforced with different fractions of two sizes (coarse and fine) of natural fibers (0, 10, 20, and 30 wt%). The three-point bend specimen of the pure polyester and polyester/alfa composite was tested under two different conditions: the first under normal conditions and the second after natural aging for 3 months. Using experimental data, the finite element method was used to analyze the stress distributions in validated numerical models.

3.1. Experimental Results

An analysis of the results revealed that the mechanical behavior of alfa/polyester composites occurred in two phases: elastic linear behavior followed by a nonlinear part (plastic deformation and damage) until the fracture of the material. From the bending test, the stress-strain curves of polyester/alfa composite reinforced with different fractions of reinforcement for both alfa fiber sizes (fine and coarse) were plotted. The mechanical properties of the composite such as Young's modulus, ultimate stress, and ultimate strain were deduced from these curves.

3.1.1. Effect of Fiber Content

Three alfa contents (10% w/w, 20% w/w, and 30% w/w) were studied in comparison to neat Polyester. The behavior was measured thanks to bending tests. Figure 7 illustrates the stress-strain curves of the bend loading response of polyester resin reinforced with different fractions of alfa fibers (10, 20, 30wt %). The curve of the pure polyester resin was taken as a reference for the comparison. As found in this figure, the flexural strengths and Young modulus of the composites at all reinforcement ratios were greater than polyester resin. Moreover, the mechanical properties of the composites increase with increasing fiber content, reaching the highest value at 30%.

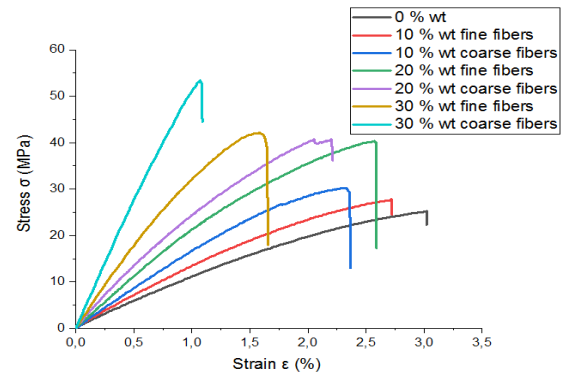


Fig. 7. Stress-strain polyester/Alfa composite with different fractions of reinforcement (0, 10, 20, 30 wt%) for both sizes of Alfa fibers (fine and coarse).

It can be seen also that the effect of alfa coarse fibers is more significant than fine fibers on the strength of the polyester/alfa composite. The ultimate stress of the non-reinforced polyester is about 25 MPa, whereas the presence of 30 wt% coarse alfa fibers increases this resistance to 110 % and 68% for the fine alfa fibers. This improvement in bending strength confirms that the coarse fibers act as the best reinforcement. These results show that the flexural modulus and maximum flexural strength increase with increasing fiber content.

3.1.2. Effect of Particle Size

The effect of particle size on the mechanical properties investigated in this study is highly significant as shown in table 1 and table 2. In general, increasing fiber size improves the modulus of elasticity and maximum strength in flexural tests. This result is consistent with previous reports on wood-particle thermoplastic composites [20-22]. As flexural Young modulus shows a steady increase with increasing fiber size at 30 wt% content (table 1, 2). It rises from 4 GPa at fine particle sizes to 6.01 GPa at coarse particle sizes. Flexural strength development also demonstrates that particle size has greater influence at higher fiber load (30 wt%), with approximately 26.61 % higher strength when fiber size increases from fine to coarse. The poor performance of fine fibers can be attributed to weak interfacial bonding and poor transfer of loads from the fibers to the matrix. However, the reinforcement reduces the ultimate strain of the polyester/alfa composite but the rate of this reduction depends on the alfa fiber content and size. The ultimate strain of the polyester is 3.2%, hence this value is reduced to 1.6% for the composite reinforced with 30 wt% of alfa fine fibers, and 1% for the polyester reinforced with 30 wt% of coarse fibers. This reduction of ultimate strain by the alfa is mainly due to the high stiffness of the alfa fibers. Consequently, the presence of alfa fibers in the polyester decreases its plasticity.

Table 1. Mechanical properties of polyester/alfa composite reinforced with fine fibers.

	Unaged composite				Aged composite			
	$\sigma_{ultime}(MPa)$	ϵ (%)	E(MPa)	ν	$\sigma_{ultime}(MPa)$	ϵ (%)	E(MPa)	ν
Neat matrix	25.24	3.02	1202	0.3	18.40	2.57	1001	0.3
10 wt % fibers	27.77	2,72	2003	0.3	20	2,32	1265	0.3
20 wt % fibers	40.34	2,57	2200	0.3	20.3	1,55	2024	0.3
30 wt % fibers	42.2	1,58	4000	0.3	31	1,72	2850	0.3

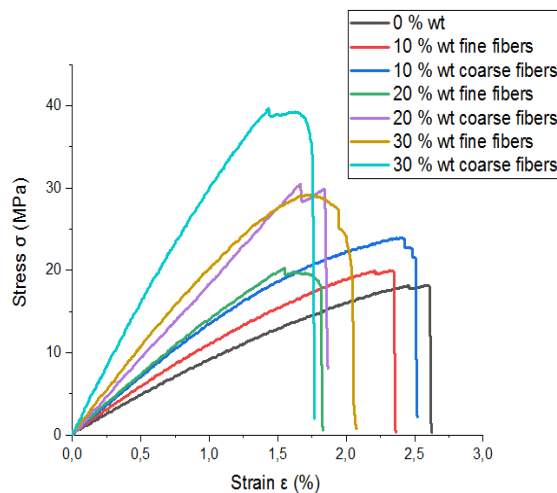
Table 2. Mechanical properties of polyester/alfa composite strengthened with coarse fibers.

	Unaged composite				Aged composite			
	$\sigma_{ultime}(MPa)$	ϵ (%)	E (MPa)	ν	$\sigma_{ultime}(MPa)$	ϵ (%)	E (MPa)	ν
Neat matrix	25.24	3.02	1202	0.3	18.40	2.57	1001	0.3
10 wt % fibers	30.25	2,33	2220	0.3	22.63	2,08	1710	0.3
20 wt % fibers	40.72	2,20	3200	0.3	29.8	1,62	2310	0.3
30 wt % fibers	53.43	1,07	6010	0.3	39.13	1,58	3480	0.3

3.1.3. Effect of Environmental Aging

After exposure of the samples during the three summer months, the materials underwent different types of attacks, first and foremost the combined action of solar UV light and oxygen. There was the absorption of UV rays from sunlight by the polymer matrix causes photodegradation or Photo-oxidation characterized by the degradation of the surface of a polymer and the scission of the molecular chains which initiates the cracks causing the degradation of the mechanical properties. This process is the most important factor in polymer weathering.

The stress-strain curves for different polyester/Alfa composites and pure polyester affected by atmospheric aging are shown in Figure 8.

**Fig. 8.** Effect of three months of atmospheric aging on the mechanical behavior of the different categories of the polyester/Alfa composite.

For the comparison of ultimate stress and strain of the different polyester/Alfa composite categories, before and after atmospheric aging conditions. Relative to non-affected composite by atmospheric aging, the reduction of the ultimate stress lies between 25% and 45%. We note that the ultimate stress of the composite reinforced with 30 wt% of coarse and fine Alfa fiber are about 54 MPa and 42 MPa, respectively. Therefore, the values were reduced to 41 MPa and 29 MPa after atmospheric aging as illustrated in Figure 9. This decrease can be attributed to the appearance of surface anomalies (microcracks), which amplified the local stresses thereby reducing the mechanical resistance of the material. When the samples were exposed to UV radiation, all properties of both biocomposites were degraded due to surface oxidation, changes in matrix crystallinity, and interfacial degradation [23]. The mechanical properties of both biocomposites were degraded, with a significant decrease. Subsequently, thermal and humidity aging are considered the major causes of long-term failure of composites exposed to the atmosphere. There are several consequences of atmospheric aging, namely the degradation of the mechanical properties of the matrix, the differential swelling linked to the concentration gradients, and the damage at the matrix/reinforcement interface [24].

When the composites are exposed to different environmental conditions and especially to moisture from the environment, the cellulosic fibers will swell. Consequently, the shear stresses will rise at the fiber/matrix interface. Thereby, the debonding risk of the interface augments. Joseph et al found that the

composites after water absorption induced an increase in the mechanical proprieties' degradation of natural fibers [25]. In addition, the diminution of thermal stability can lead to the degradation of polymer, fibers, and polymer/fiber interface loosening [26].

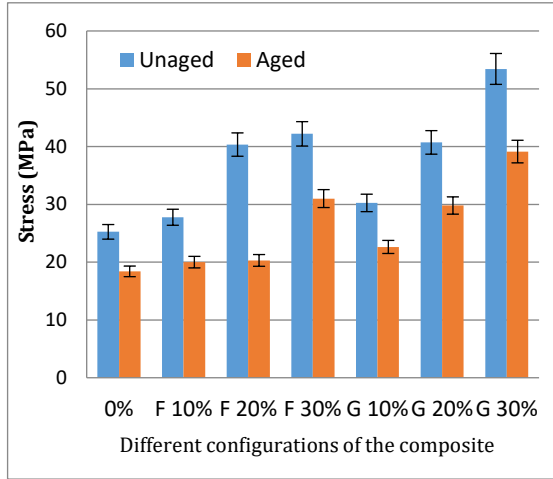


Fig. 9. Effect of three months of atmospheric aging on the bending strength of the polyester/Alfa composite with different fractions of reinforcement (0, 10, 20, 30 wt%) for both sizes of Alfa fibers (fine and coarse).

Figure 10 illustrates the evolution of Young's modulus of unaged and aged composites with respect to fiber content. As shown in this figure, an important Young's modulus increase with fiber addition was observed for all composite types.

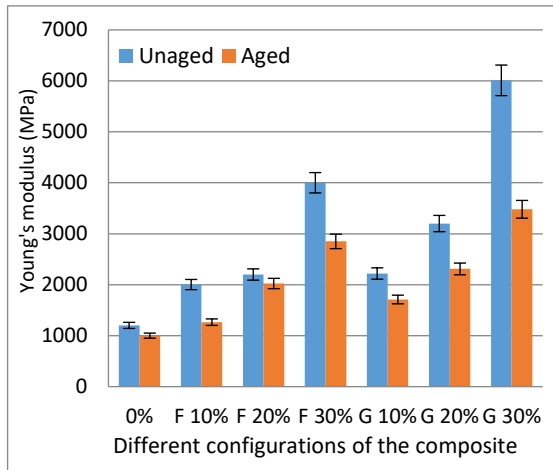


Fig. 10. Young's modulus variation of different Polyester/Alfa composites (Coarse and fine size).

This significant increase can be attributed to lower porosity and an indication of a strong interphase (Figure 11). Cavities whose genesis is connected to structural faults begin in the amorphous phase when the material is mechanically loaded [27]. The stress concentration can be located in the defects and leads to the creation, initiation, and propagation of the crack in the material [28].

Furthermore, the composite with coarse fibers exhibits a much higher Young's modulus than the one with fine fibers and neat polyester resin. These findings demonstrate that variations in Young's modulus are caused by atmospheric aging. Indeed, atmospheric aging induces a decrease in Young's modulus for all aged composites and this variation was amplified by Alfa fiber content. Moreover, the reduction of Young's modulus is less than 1% for the pure polyester caused by natural aging and reached 40% for the composite reinforced with 30 wt% of coarse Alfa fibers. Numerous studies have detailed the linear evolution of the longitudinal Young's modulus with the volume filling ratio.[29–31], where relationships between the filling percent and the composites' mechanical properties were shown.

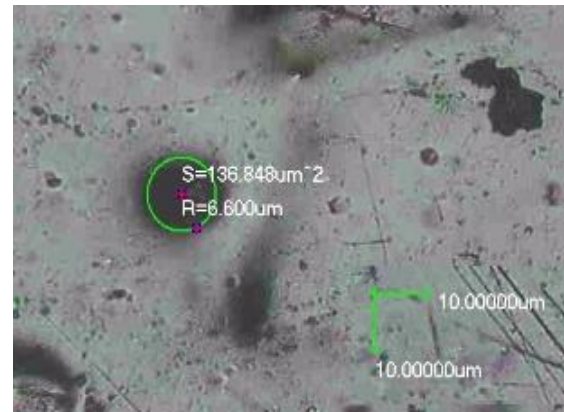


Fig. 11. Microvoids observed experimentally in the polyester/Alfa composite.

3.2. Numerical Analysis and Validation

To examine the specifics of the composite Alfa polyester's bending resistance ability, its deformation mode during the bending process is shown in Figure 12, where the general case's experimental and numerical deformation states were compared. Clear fracture in Zone (a) (marked with red lines) can be seen both in the experiment and simulation. They align rather well with one another. These details of composite Alfa polyester further demonstrate the numerical model's applicability and validity.

The comparison between the FE and experimental findings (stress-strain curves) of both alfa/polyester composite cases (fine and coarse alfa fibers) reinforced with different fractions of fibers (0, 10, 20, and 30 wt%) is illustrated in Figure 13. The experiment findings and FE results are in good agreement, as seen in the figure, with an estimated average relative inaccuracy of less than 15% in the perceived strength. The continuum damage mechanics-based model appears to be sufficient for simulating the failure of the alfa/polyester composite.

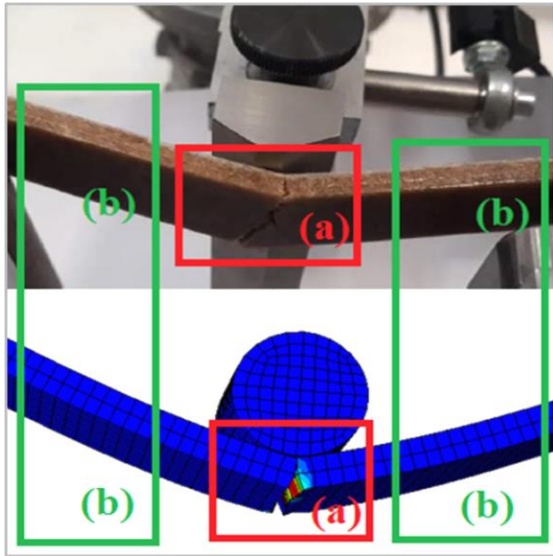


Fig. 12. Central area failure: experimental and numerical

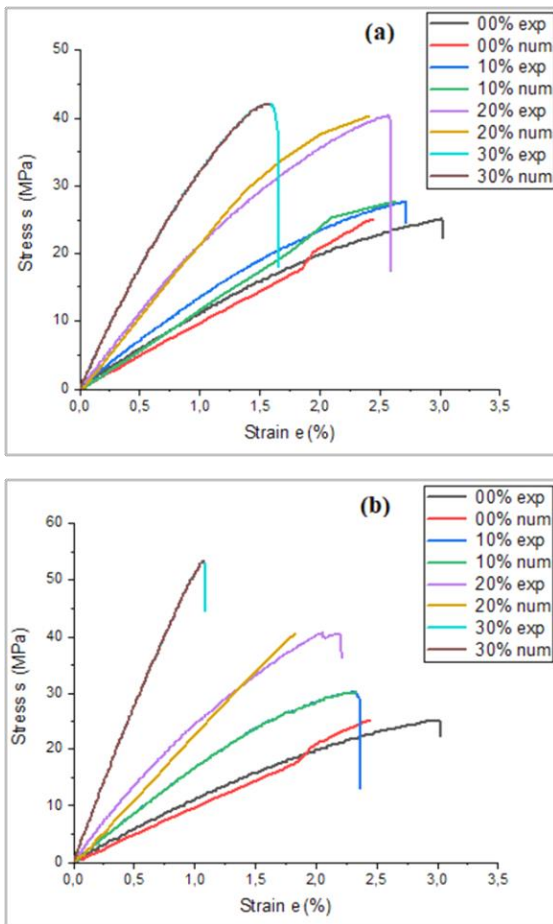


Fig. 13. The experimental and numerical stress-strain curves of unaged Alfa/polyester composite with different fractions of reinforcement (0, 10, 20, 30 wt%) for both sizes of alfa fibers: (a) fine fibers and (b) coarse fibers.

Figure 14 shows a comparison of the predicted and the experimental stress versus strain curves of all cases of aged alfa/polyester composite for bending loading conditions. The overall mechanical reactions found in the studies

appear to be accurately captured by the numerical data, and the average relative error in the apparent stress is estimated as <15%. These composites' experimental and numerical curves show the same shape with a tolerable error. The existence of voids, discontinuities, and porosity in the fiber and matrix might cause this error.

Higher strength and fracture energy are typically associated with mostly shear loading due to bend loading conditions, which tends to cause numerous damages such as fracture of the polyester and alfa fiber as well as decohesion of the polyester/alfa fiber interface.

Data from the flexural analysis can be used to forecast the failure behavior of composite materials, suggesting that the constitutive principles chosen for damage initiation and evolution appear to be plausible.

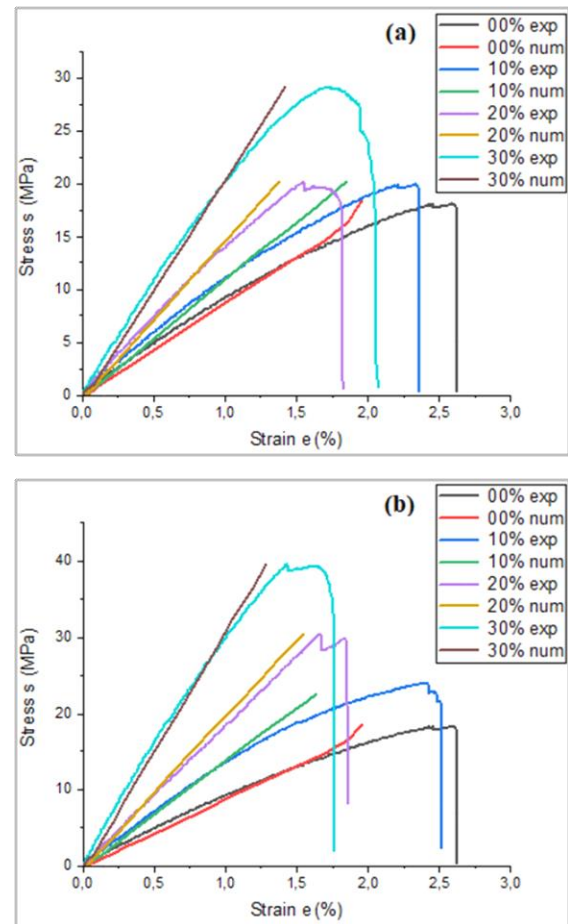


Fig. 14. The experimental and numerical stress-strain curves of aged Alfa/polyester composite with different fractions of reinforcement (0, 10, 20, 30 wt%) for both sizes of alfa fibers: (a) fine and (b) coarse.

3.3. Van Mises Stress Distributions

Figure 15 illustrates the von Mises stress distributions along the longitudinal axis of the composite specimen. According to this figure, for all cases of composites, it has been seen that the

highest von Mises stress is noted in the middle of the specimen. It seems that the von Mises stress values found in the composite reinforced with coarse fibers are greater than the stress values in the composites reinforced with fine fiber. The analysis of the von Mises stress distribution at different weight fractions (0%, 10 %, 20 %, 30 %) can be seen in this figure, revealing that the curve of the von Mises stress at 30% is different from those at the other fractions. Without reinforcement, the polyester has a maximum stress of around 23 MPa., in polyester reinforced with 30% fine and coarse alfa fiber, this value increases to 38 MPa and 50 MPa. The association between the effects of the percentage of alfa fiber and its size increases the strength of the alfa/polyester composite.

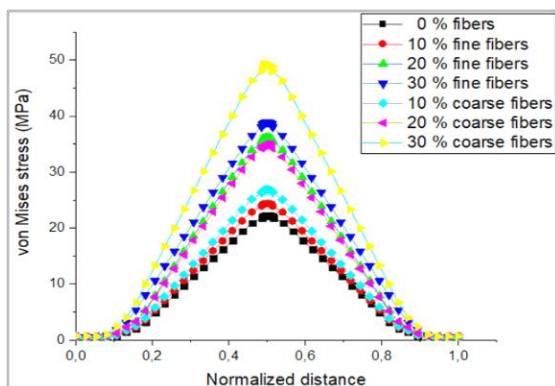


Fig. 15. Von Mises stress distributions: Young polyester/Alfa composite with different fractions of reinforcement (0, 10, 20, 30 wt%) for both sizes of Alfa fibers (fine and coarse).

By comparing the values of the maximum von Mises stresses in the unaged and aged tests, it can be observed that natural aging has a significant effect on the degradation of the mechanical properties of alfa/polyester composite. For pure polyester (without reinforcement), three months of aging decreased the stress concentration by more than 30%; this reduction reaches 28%, in the case of the alfa/polyester composite reinforced with 30% of coarse alfa fibers (Figure 16).

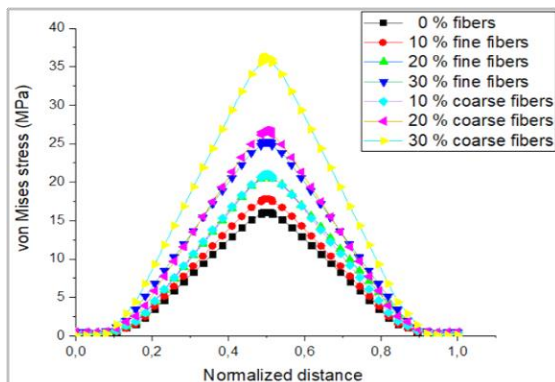


Fig. 16. Von Mises stress distributions: Aged polyester/Alfa composite with different fractions of reinforcement (0, 10, 20, 30 wt%) for both sizes of Alfa fibers (fine and coarse).

4. Conclusions

In this study, three-month periods of aging settings were applied to polyester/alfa composites. In the investigation, it was found that environmental aging and filler percentage had a significant impact on composite materials properties. Therefore, the current research allows for the deduction of the following findings:

- Alfa fibers can strengthen polymer matrices.
- The strength of the composite reinforced with the coarse fibers of alfa is more effective than the fine fibers one.
- The increase of alfa fibers percentage was beneficial for the improvement of composite mechanical properties.
- The three months of atmospheric aging for a specific environment contributes to the deterioration significantly the composite mechanical behavior.
- The simulation results showed strong agreement with the experimental results.
- The addition of UV stabilizers is suggested to enhance the outdoor performance of natural fiber/polymer composite and to achieve a balance between strength and durability requirements for natural fiber composites.

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Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this article.

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