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FACULTY OF TECHNOLOGY
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Youcef Gheid

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Cyclic Fatigue Behavior in Tensile and Bending of a Glass/Epoxy Laminated Composite Material Loaded with Pure Iron Powder

Thesis was defended on 13/01/2025 before the jury composed of:

N°	Name and First name	Grade	Establishment	Quality
01	LAOUAR Lakhdar	Prof.	Badji Mokhtar -Annaba University	President
02	CHEMAMI Abdennacer	MCA	Badji Mokhtar -Annaba University	Rapporteur
03	LAGRED Ahmed	Prof.	Badji Mokhtar -Annaba University	Examiner
04	GUEDRI Abdelmoumene	Prof.	Mohamed Cherif Messaadia University	Examiner
05	KHELIFA Nouredine	MCA	The National Higher School of Technology and Engineering -Annaba	Examiner

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Dedication

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Abstract

This thesis explores the effects of incorporating pure iron powder as a filler in glass epoxy composite laminates, specifically focusing on its impact on flexural strength, tensile strength, and hardness. The study involved creating laminate plates with varying iron content (15%w, 20%w, and 25%w) and grain sizes (92 μm , 64 μm , and 32 μm) while maintaining a consistent 30%w fiberglass content. To evaluate the mechanical properties of the composite, hardness measurements were conducted on plates with identical iron powder content but different grain sizes, also bending tests were conducted following ISO 14125 standards, using Taguchi's orthogonal array L9 (3×3) with iron powder content, grain size, and test speed as factors.

The findings indicate that variations in iron powder content and grain size have a direct impact on the mechanical characteristics of the composite material. Specific percentages of filler and reduced grain sizes were found to significantly enhance hardness and flexural strength. Comparative analyses were conducted with a glass-epoxy laminate with 45%w of fiber, further highlighting the performance of the materials. The study also examined the tensile properties of various specimens, offering a nuanced understanding of how varying concentrations of iron powder shape the mechanical behavior of glass epoxy laminates under axial loading conditions. The results showed a progressive increase in tensile strength up to 20% of iron powder loading, beyond which diminishing returns or potential negative effects were observed. This understanding can inform the optimization of composite formulations and guide the design of materials with optimal mechanical performance.

Additionally, the study explores the impact of seawater and acidity on glass epoxy composites filled with pure iron powder. Plates with varying iron content underwent a three-point bending examination following ISO14125 standards, revealing significant declines in flexural strength under harsh environmental conditions. The section dedicated to exploring the cyclic fatigue behavior of materials under flexural loading analyzed the results of static bending tests conducted in challenging conditions. These tests revealed a significant decrease, up to 40%, in the mechanical properties of the materials following static bending. Consequently, a decision was made to forego additional fatigue tests.

Résumé

Cette thèse explore les effets de l'incorporation de poudre de fer pur comme charge dans les composites stratifiés verre-époxy, en se concentrant spécifiquement sur son impact sur la résistance à la flexion, la résistance à la traction et la dureté. L'étude impliquait la création de plaques stratifiées avec une teneur en fer variable (15 % en poids, 20 % en poids et 25 % en poids) et des tailles de grains (92 μm , 64 μm et 32 μm), tout en maintenant une teneur constante de 30 % en fibre de verre. Pour évaluer les propriétés mécaniques du composite, des mesures de dureté ont été effectuées sur des plaques avec une teneur en poudre de fer identique mais des tailles de grains différentes. Des essais de flexion ont également été effectués conformément aux normes ISO 14125, en utilisant le réseau orthogonal L9 (3 \times 3) de Taguchi en choisissant comme facteurs la teneur en poudre de fer, la taille des grains et la vitesse de l'essai.

Les résultats indiquent que les variations de la teneur en poudre de fer et de la taille des grains ont un impact direct sur les caractéristiques mécaniques du matériau composite. Il a été constaté que des pourcentages spécifiques de charge et des tailles de grains réduites améliorent considérablement la dureté et la résistance à la flexion. Des analyses comparatives ont été menées avec un stratifié verre-époxy contenant 45 % en poids de fibres, soulignant davantage les performances des matériaux. L'étude a également examiné les propriétés de traction de divers spécimens, offrant une compréhension nuancée de la manière dont les différentes concentrations de poudre de fer façonnent le comportement mécanique des stratifiés de verre époxy dans des conditions de chargement axial. Les résultats ont montré une augmentation progressive de la résistance à la traction jusqu'à 20 % de la charge de poudre de fer, au-delà de laquelle des rendements décroissants ou des effets négatifs potentiels ont été observés. Cette compréhension peut éclairer l'optimisation des formulations composites et guider la conception de matériaux offrant des performances mécaniques optimales.

De plus, l'étude explore l'impact de l'eau de mer et de l'acidité sur les composites de verre époxy remplis de poudre de fer pure. Les plaques avec différents teneurs en fer ont subi un essai de flexion en trois points conformément aux normes ISO14125, révélant une baisse significative de la résistance à la flexion dans des conditions environnementales difficiles. La section dédiée à l'exploration du comportement en fatigue cyclique des matériaux soumis à des charges de flexion a analysé les résultats d'essais de flexion statique menés dans des conditions difficiles. Ces tests ont révélé une diminution significative, jusqu'à 40 %, des propriétés mécaniques des matériaux suite à une flexion statique. Par conséquent, la décision a été prise de renoncer à des essais de fatigue supplémentaires.

خلاصة

تستكشف هذه الأطروحة تأثيرات دمج مسحوق الحديد النقي كمادة حشو في شرائح مركبة من الزجاج والإيبوكسي، مع التركيز بشكل خاص على تأثيره على قوة الانثناء، وقوة الشد، والصلابة. تضمنت الدراسة إنشاء ألواح مصقولة بمحتوى حديدي متفاوت (15% وزن، 20% وزن، 25% وزن) وأحجام حبيبات (92 ميكرومتر، 64 ميكرومتر، و32 ميكرومتر)، مع الحفاظ على محتوى ثابت من الألياف الزجاجية بنسبة 30%. لتقييم الخواص الميكانيكية للمركب، تم إجراء قياسات الصلابة على ألواح ذات محتوى مسحوق حديد متطابق ولكن بأحجام حبيبات مختلفة، كما تم إجراء اختبارات الانحناء وفقاً لمعايير ISO 14125، باستخدام مصفوفة تاجوتشي المتعامدة L9 (3 × 3) مع محتوى مسحوق الحديد، حجم الحبوب وسرعة الاختبار كعوامل.

تشير النتائج إلى أن الاختلافات في محتوى مسحوق الحديد وحجم الحبوب لها تأثير مباشر على الخصائص الميكانيكية للمادة المركبة. تم العثور على نسب محددة من الحشو وأحجام الحبيبات المخفضة لتعزيز الصلابة وقوة الانثناء بشكل ملحوظ. تم إجراء تحليلات مقارنة باستخدام صفائح زجاجية إيبوكسي تحتوي على 45% من الألياف، مما يسلط الضوء بشكل أكبر على أداء المواد. درست الدراسة أيضاً خصائص الشد للعينات المختلفة، مما يوفر فهماً دقيقاً لكيفية تشكيل التركيزات المتفاوتة لمسحوق الحديد للسلوك الميكانيكي لشرائح الإيبوكسي الزجاجية في ظل ظروف التحميل المحوري. وأظهرت النتائج زيادة تدريجية في قوة الشد تصل إلى 20% من تحميل مسحوق الحديد، وبعد ذلك لوحظ تناقص العوائد أو الآثار السلبية المحتملة. يمكن لهذا الفهم أن يفيد في تحسين التركيبات المركبة وتوجيه تصميم المواد ذات الأداء الميكانيكي الأمثل.

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Nomenclature

Abbreviation	Definition
2θ	The angle used in X-ray diffraction measurements.
AFM	Atomic Force Microscopy.
ANOVA	Analysis of Variance.
ASTM	American Society for Testing and Materials.
BHN	Brinell Hardness Number.
BN	Boron Nitride.
CDM	Continuum Damage Mechanics.
CFRP	Carbon Fiber Reinforced Polymer: A composite of carbon fibers and a polymer matrix.
CLT	Classic Lamination Theory.
CM	Composite Materials: Materials consisting of two main elements, the matrix and reinforcement.
CM	Corrosion Mitigation: Techniques to prevent or reduce corrosion in composites.
CMCs	Ceramic Matrix Composites: Composites where the matrix is made of ceramic materials.
CS	Control Sample.
CSM	Chopped Strand Mat.
D	Diameter of Indenter: The size of the indenter used in hardness testing.
DCB	Double Cantilever Beam.
DIN	Deutsches Institut für Normung.
DRX	X-ray Diffraction.
E	Flexural Modulus: A measure of a material's stiffness in bending.
E-tensile	Tensile Modulus: The material's resistance to elongation under tensile stress.
E-glass	A type of glass fiber commonly used in composites.
EE	Environmental Exposure: The interaction of materials with various environmental conditions.
F	Applied Force: The force applied to a specimen during testing.
FB	Fatigue Behavior: The response of materials to cyclic loading, leading to material degradation.
FDG	Fatigue Delamination Growth.
FEA	Finite Element Analysis.
FMLs	Fiber Metal Laminates.
FTIR	Fourier Transform Infrared Spectroscopy.
GFRP	Glass Fiber Reinforced Polymer: A composite made from glass fibers and a polymer matrix.
GSM	Grams per Square Meter.
H	Nanoindentation Hardness: A method of measuring hardness at the nanoscale.
HB	Brinell Hardness: A hardness test using a steel ball indenter.
HG181	Type of glass fabric used in composite materials.
HR	Hardness Rating.
HV	Vickers Hardness: A method to measure hardness using a diamond indenter.
IP	Insulation Properties: The ability to resist the flow of heat, important for energy and electronics.
IRAffinity-1S	Specific FTIR instrument model used for measurements.
ISO	International Organization for Standardization.
KIC	Fracture Toughness: The ability of a material to resist crack propagation.
Kα	X-ray wavelength emitted by copper anticathode in X-ray diffraction.
LB	Loss Function in Taguchi's method.
LCU250NS	Type of unidirectional carbon fiber composite.
M	Matrix: The material that provides cohesion and structure in a composite, surrounding the reinforcement.
MB	Mechanical Behavior: The response of a material under different forces, influencing its structural integrity.
MMCs	Metal Matrix Composites: Composites where the matrix is made of metal.
MP	Magnetic Properties: The characteristics of a material that define its response to magnetic fields.
N	Number of Cycles to Failure.

NFCs	Natural Fiber Composites: Composites made from natural fibers like flax, jute, and hemp.
P	Applied Load: The force applied during nanoindentation testing.
Pa	Pascal (unit of pressure or stress).
R	Reinforcement: The material used to strengthen a composite, typically fibers or particles.
R-ratio	Load Ratio (tension-compression ratio).
REA	Renewable Energy Applications: The use of composites in wind and solar energy industries.
REF	Reference.
RH	Relative Humidity.
RTM	Resin Transfer Molding: A composite manufacturing process involving the injection of resin into fibers.
S-N curve	Stress-Number of Cycles Curve.
S/N Ratio	Signal-to-Noise Ratio.
SEM	Scanning Electron Microscope.
SERR	Strain Energy Release Rate.
SWR	Strength-to-Weight Ratio: A measure of strength compared to weight, important in aerospace.
SiO ₂	Silicon Dioxide.
T700S	Type of carbon fiber used in composites.
TEM	Transmission Electron Microscopy.
TS	Tensile Strength: The maximum stress a material can withstand before breaking.
Tsai-Hill	A failure criterion is used for analyzing composite materials under stress.
UD	Uniform Dispersion: The even distribution of particles or reinforcements throughout the matrix.
URASM	Iron and Steel Applied Research Unit.
URMM	Mining and Metallurgy Research Unit.
UV	Ultraviolet.
V	Volume.
VARI	Vacuum-Assisted Resin Infiltration.
VHN	Vickers Hardness Number: A scale for hardness derived from the Vickers test.
cm ⁻¹	Centimeters inverse (unit for wavenumber in FTIR spectra).
pm ³	Cubic Picometer.
μm	Micrometer.
σ	Flexural Strength: The ability of a material to resist bending.
σ _a	Stress Amplitude.
σ _m	Mean Stress.

General Introduction

1. Introduction

Composite materials represent a revolutionary frontier in material science, combining the strengths of different components to create materials with enhanced properties. These materials consist of two main elements: the matrix, providing cohesion, and the reinforcement, imparting strength. The synergy between these components results in materials with superior mechanical, thermal, or electrical characteristics compared to their parts.

In the realm of composites, various types exist, including Polymer Matrix Composites (PMCs), Metal Matrix Composites (MMCs), and Ceramic Matrix Composites (CMCs). PMCs, for example, combine reinforcing elements with a polymer matrix, offering versatility and widespread use in everyday applications.

One of the key advantages of composite materials is their exceptional strength-to-weight ratio, making them crucial in industries like aerospace, automotive, and construction. Engineers can tailor the properties of composites to specific needs, providing flexibility in design and functionality.

Despite their numerous advantages, challenges persist in ensuring strong adhesion between the matrix and reinforcement while addressing potential issues like delamination. Nonetheless, the widespread applications of composites underscore their transformative impact across diverse sectors, ranging from cutting-edge aerospace technologies to everyday construction materials. As technological advancements continue, the realm of composite materials promises even more innovative solutions for future engineering challenges.

2. Metals and Alloys: Foundations of Material Science

In the vast realm of material science, **metals and alloys** stand as foundational components, embodying a rich interplay of properties that have fueled human innovation for centuries. Metals, characterized by their metallic luster, conductivity, and malleability, serve as the elemental building blocks for a myriad of applications.

Alloys, a strategic fusion of different metals, further broaden the spectrum of material possibilities. The deliberate combination of metals in alloys imparts tailored characteristics, addressing specific needs in various industries. From the strength of steel to the corrosion resistance of stainless steel, alloys epitomize the versatility inherent in manipulating material composition.

The study of metals and alloys extends beyond their mechanical properties to encompass thermal, electrical, and magnetic behaviors. This multidimensional understanding is paramount for crafting materials that meet the exacting demands of modern engineering.

As we embark on the exploration of composite materials, the incorporation of metals and alloys introduces a dynamic element, blending tradition with innovation. This synergy forms the bedrock upon which advancements like iron powder-infused composites can push the boundaries of material science, opening doors to unprecedented applications and capabilities.

3. Iron Powder in composite materials

Within the realm of composite materials, the integration of iron powder introduces a unique dimension, amplifying the material's properties and expanding its potential applications. Iron powder, a finely divided form of elemental iron, acts as a reinforcing element within composite matrices.

The addition of iron powder contributes to the overall strength and magnetic properties of the composite material. This inclusion can significantly enhance the material's performance in applications where magnetic characteristics or increased tensile strength are desirable. The versatility of iron powder allows for tailoring the composite to specific requirements, making it a valuable component in various engineering contexts.

In the context of fatigue studies, such as cyclic loading in tension and flexion, the behavior of composites incorporating iron powder becomes a focal point. Understanding how iron powder interacts with the matrix and influences fatigue properties is crucial for optimizing the performance of these advanced materials.

As researchers delve into the intricacies of composite materials enriched with iron powder, they open avenues for innovations across industries. From magnetic components in electronics to

robust structural elements, the strategic use of iron powder within composites exemplifies the continuous evolution and diversification of materials engineering. Exploring this dynamic interplay promises to unlock new frontiers in material science, ushering in advancements that transcend traditional boundaries.

4. Industrial Applications

Composites, a culmination of various materials working in tandem, represent a revolutionary paradigm in industrial applications. Their distinctive advantage lies in the ability to combine different elements, harnessing synergies that transcend the individual strengths of each component.

In industries ranging from aerospace to automotive and construction, composites have emerged as key players, offering a spectrum of benefits. One of their hallmark characteristics is a high-strength-to-weight ratio, providing structural integrity without unnecessary bulk. This trait is particularly crucial in aviation, where lightweight yet robust materials are instrumental in enhancing fuel efficiency.

Moreover, composites exhibit exceptional resistance to corrosion, a quality highly sought after in maritime and offshore structures. Their adaptability extends to insulation properties, making them valuable in sectors where temperature control is paramount, such as the energy and electronics industries.

As the demand for sustainable solutions grows, composites contribute by offering materials with enhanced durability and reduced environmental impact. This is evident in renewable energy applications, where composites play a pivotal role in wind turbine blades and solar panel structures.

In the realm of material science, the versatile nature of composites continues to drive innovation. From enhancing performance in sports equipment to revolutionizing medical devices, composites stand as a testament to the boundless possibilities when disparate materials converge in pursuit of progress.

5. Challenges and Opportunities

Iron powder stands out as a distinctive component in composite materials, imparting enhanced strength, conductivity, and magnetic properties. Its integration reinforces structures, adds conductivity for electronics, and introduces magnetic responsiveness. While offering diverse applications across industries, challenges like uniform dispersion and corrosion mitigation persist. This dynamic interplay of traditional metallurgy and modern material science opens doors to innovative solutions. The comprehensive understanding of mechanical behavior in composite materials, both before and after exposure to various environments, stands as a pivotal

aspect in evaluating their performance, endurance, and dependability. This intricate examination encompasses crucial properties such as strength, stiffness, and toughness, directly influencing the material's structural integrity. Exposure to diverse environments, be it natural or aggressive, has the potential to induce alterations in these properties, posing risks to structural stability, degradation, or eventual failure. A meticulous exploration of the material's mechanical behavior, both pre-and post-exposure, offers invaluable insights into its resilience, facilitating the optimization of applications across diverse conditions and ensuring sustained functionality over the long term.

Chapter I

State of the Art

This chapter reviews previous research on composite materials, covering their constituents and types based on matrices and reinforcing materials. It discusses the advantages of composites, elaboration methods, and damage modes in laminated composites. Mechanical characteristics are explored through hardness theory, Three-Point Bending analysis, and Tensile Behavior assessment. Environmental durability and fatigue behavior are also addressed, guiding future research objectives for advancing composite material technology.

I.1. Introduction

The study of composite materials is a crucial aspect of understanding and utilizing these materials effectively across various applications. This chapter initiates a comprehensive exploration, commencing with an Introduction to composite materials and systematically progressing through the components that define their unique properties. It delves into the various types of composite materials based on matrices, highlighting the pivotal role of the matrix in providing structural support and transferring loads. Each part is carefully looked at, from the complex materials like fibers that make composites stronger to the importance of fillers and additives in shaping how composites work overall.

Furthermore, the chapter discusses the advantages of these materials and provides insights into their elaboration methods and mechanical behaviors. This detailed examination establishes a solid foundation for understanding composite materials. As the exploration progresses, the chapter emphasizes the environmental durability and fatigue aspects of composite materials, showcasing their real-world applicability. This introductory overview sets the stage for a comprehensive literature review that not only encapsulates the current state of knowledge but also identifies key gaps and limitations, paving the way for our research objectives aimed at advancing and refining composite material technology.

Composite materials, a result of materials engineering, blend different components to create a single, advanced material. These materials are carefully composed of two or more components with very different physical or chemical properties. This combination results in a remarkable synergy, leading to improved characteristics. By arranging materials with distinct strengths, weaknesses, and capabilities, a material is created that surpasses the limitations of its individual components. The fundamental characteristic of composite materials lies in their unique composition, where the individuality of each component is preserved and strategically utilized. This delicate balance results in enhanced performance attributes, including increased strength, durability, flexibility, and resilience against adverse environmental conditions.

Composite materials are widely used across several industries due to their versatility. In aerospace, they provide lightweight yet durable materials crucial for enhancing aircraft efficiency. The automotive industry uses composites to improve fuel efficiency and produce high-performance vehicles. Similarly, the building industry benefits from the durability and structural properties of these materials, while sports equipment relies on their strength and flexibility. Laminated composites have thus far found applications in technological sectors such as the aerospace and automotive industries [1], where most of the components are static, due to their excellent mechanical properties. Furthermore, composite materials are characterized by their

dynamic nature, as evidenced by continual progress in material science and engineering. Researchers continually explore innovative combinations, production methods, and applications, pushing the boundaries of what composites can achieve.

The utilization of fiber-reinforced composite materials in engineering has witnessed substantial growth in recent years. This surge can be attributed to advancements in manufacturing processes, rendering these materials increasingly adaptable for a wide range of technical applications. [2]. Sectors such as the aerospace and automotive industries have a notable interest in lightweight yet robust materials, which has resulted in an increased demand for composite materials. [3].

Glass epoxy composites, reinforced with various substances including different metals, have emerged as a significant material type in various industries due to their exceptional strength [4]. Researchers have investigated the impact of incorporating various metals, such as iron powder, on the strength of these materials. A study explored metal-PMCs utilizing HDPE, PP, and PS as polymer matrices, incorporating differing volumes of Fe powder. The findings highlighted variations in the reduction of impact strength and the increase in hardness among the composites. PS-Fe composites demonstrated superior strength and modulus but lower elongation. Moreover, HDPE- and PP-based composites exhibited ductile fractures, while PS-Fe composites displayed brittle fractures. [5] [6].

Researchers conducted a study on the tribological behavior of glass epoxy polymer composites, incorporating SiC and graphite particles as secondary fillers. The investigation was carried out under dry sliding conditions using a pin-on-disc wear rig. The study aimed to analyze the influence of wear parameters such as applied load, sliding speed, sliding distance, and the percentage of secondary fillers on the wear rate. Employing Taguchi techniques, a systematic plan of experiments was executed. The findings revealed that the addition of SiC and graphite substantially improves the wear resistance of glass epoxy composites [7]. A study investigated the reinforcement of unsaturated polyester resin with metal powders, specifically copper (15.598 μm) and aluminum (21.533 μm), at various volume fractions (0%, 5%, 10%, 15%, 20%, and 25%). The mechanical properties under scrutiny included tensile properties (strength at fracture point, elastic modulus, and elongation at break), flexural strength and modulus, impact properties, and hardness. The findings revealed a notable increase in tensile strength and modulus with the rise in volume fraction, peaking at a 15% volume ratio for both types of metal powders. Flexural properties and fracture toughness reached their zenith at a 10% volume ratio. However, elongation at break decreased as the volume fraction of metal powder increased for both types of composites [8]. Prior studies have investigated the influence of nanoparticles on the micro-mechanical and surface properties of poly (urea-formaldehyde) composite microcapsules [9].

The advancement of fibers of diverse compositions substitutes the need for costly materials traditionally required for the fabrication of industrial components [10], this indicates a broader trend in materials science and engineering where researchers or industries are exploring alternative and cost-effective materials, likely with enhanced or specific properties, to meet the requirements of industrial applications.

I.2. Constituents of composite materials

Composite materials, renowned for their tailored properties, derive their strength and versatility from a careful combination of distinct constituents, each playing a pivotal role in shaping the material's characteristics. Understanding these constituents provides a profound insight into the intricate world of composite materials.

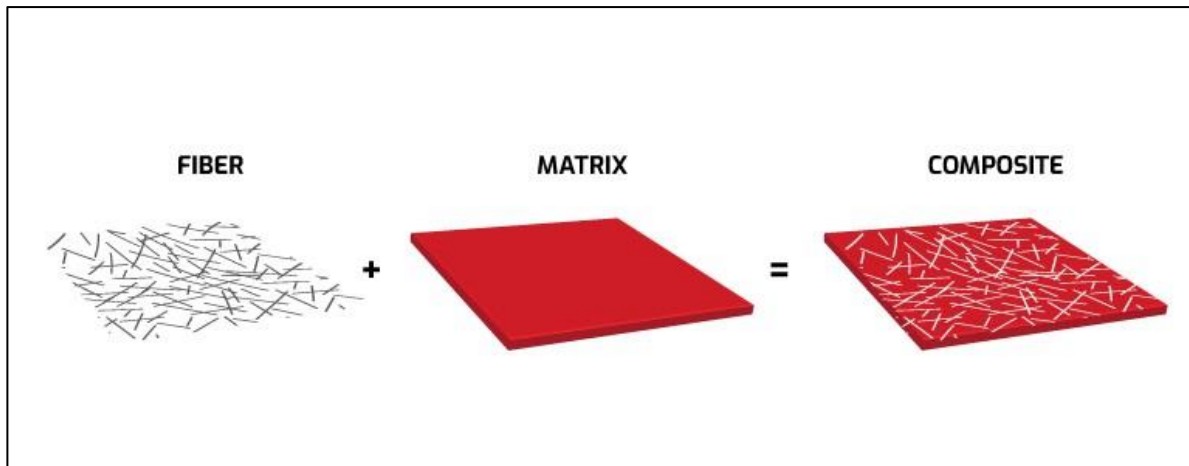


Figure I.1. Constituents of composite materials [11]

I.3. Types of Composite Materials Based on Its Reinforcement Form

According to the reinforcement form, composites can be classified into several categories, including continuous fiber composites, discontinuous fiber composites, sandwiches, and laminates.

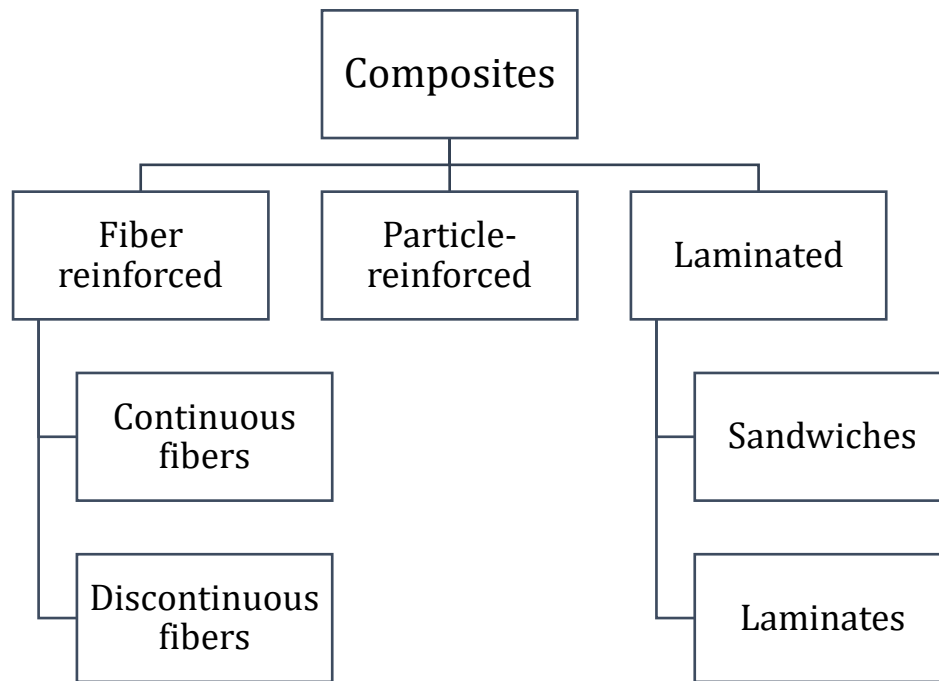


Figure I.2. Types of composites

Continuous Fibers: In continuous fiber composites, long and continuous fibers are used as reinforcement. These fibers can be wound, woven, or placed in a specific direction, providing significant strength along that particular direction. This type of composite is often used when high directional properties are required.

Discontinuous Fibers: Discontinuous fiber composites use short or chopped fibers as reinforcement. These fibers are typically randomly dispersed in the matrix. While these composites can offer improved mechanical properties, they may not have the same directional strength as continuous fiber composites.

Sandwiches: Sandwich structures are composed of outer layers, often rigid, called skins, and a lightweight central layer, usually made of foam or honeycomb. This type of composite offers an optimal combination of strength and lightness, making them ideal for applications such as aerospace and marine construction.

Laminates: Laminated composites are formed by stacking multiple layers of materials, usually fibers and a matrix, in a specific arrangement. Each layer can have different properties, allowing for customized mechanical and thermal characteristics. These composites are widely used in various applications, from sports equipment to structural components.

I.4. Types of Composite Materials Based on Its Matrix

Matrix Material

The matrix in composite materials acts as the binding agent, encapsulating and supporting the reinforcing elements. Depending on the application, matrices can be polymers, metals, or ceramics. Polymers, known as PMCs, are popular for their versatility, while MMCs and CMCs excel in demanding environments. The matrix provides cohesion, transfers loads, and protects reinforcements from external factors.

I.4.1. Polymer Matrix Composites (PMCs)

Within the vast landscape of composite materials, PMCs stand out as a versatile and widely adopted class. PMCs are engineered materials where a polymer resin matrix binds together reinforcing fibers or particles. This matrix, often made of epoxy, polyester, or other high-performance polymers, works in tandem with reinforcing materials to create a composite that boasts an impressive array of properties.

The reinforcing phase in PMCs can take various forms, such as glass, carbon, or aramid fibers, each contributing unique characteristics to the final composite. Carbon fibers, for instance, bring high-strength and stiffness, making them particularly valuable in applications where lightweight and robust materials are paramount. Glass fibers, on the other hand, provide cost-effective reinforcement, finding applications in a wide range of industries.

PMCs find extensive use in aerospace, automotive, marine, and sporting goods due to their advantageous properties. Their lightweight nature, corrosion resistance, and tailored mechanical properties make them ideal for components subjected to diverse and demanding conditions. The ability to precisely tune the composition of PMCs allows engineers to customize materials for specific applications, striking a balance between weight, strength, and durability.

Manufacturing PMCs involves processes like hand lay-up, resin transfer molding (RTM), and filament winding. These techniques enable the creation of intricate shapes and structures, expanding the application scope of PMCs. As research in materials science advances, innovations in polymer chemistry and composite manufacturing techniques continue to enhance the performance and broaden the utility of PMCs, ensuring their pivotal role in the evolution of modern materials engineering.

I.4.2. Metal Matrix Composites (MMCs)

In the realm of advanced materials, MMCs emerge as a fascinating class, seamlessly blending the strength of metals with the tailored properties of other reinforcing materials. These engineered

materials consist of a metal matrix hosting dispersed particles or fibers, resulting in composites that exhibit enhanced mechanical and thermal characteristics.

The metal matrix, often aluminum, magnesium, or titanium, forms the backbone of MMCs. Reinforcing elements, such as silicon carbide, alumina, or carbon fibers, are strategically integrated to impart specific attributes to the composite. This strategic combination capitalizes on the strength and ductility of metals while mitigating their limitations, such as weight or thermal conductivity.

One prominent example of MMC application is in the aerospace industry, where the demand for lightweight yet robust materials is critical. Aluminum matrix composites, reinforced with particles like boron carbide or alumina, find applications in components like aircraft structures and engine parts. The enhanced strength and stiffness of MMCs make them ideal for scenarios where traditional metals might fall short.

The manufacturing of MMCs involves methods like powder metallurgy, stir casting, or infiltration techniques. These processes enable the homogeneous distribution of reinforcing elements within the metal matrix, ensuring optimal performance in the final composite. MMCs continue to push the boundaries of material science, offering solutions for industries seeking materials with tailored properties, improved performance, and a higher degree of versatility.

I.4.3. Ceramic Matrix Composites

Within the intricate landscape of advanced materials, CMCs stand as sculpted marvels, embodying the fusion of ceramics with reinforcing elements to forge materials with unparalleled properties. CMCs are engineered to overcome the inherent brittleness of traditional ceramics by introducing a matrix fortified with fibers or particles, resulting in materials that excel in both strength and toughness.

At the heart of CMC lies a ceramic matrix, often composed of materials like silicon carbide (SiC), alumina (Al_2O_3), or zirconia (ZrO_2). These matrices, renowned for their high-temperature stability and corrosion resistance, are interwoven with reinforcing elements such as silicon carbide fibers or whiskers. This strategic combination grants CMCs exceptional mechanical properties, making them adept at withstanding extreme conditions.

One of the defining features of CMCs is their ability to retain strength at elevated temperatures, a quality coveted in industries such as aerospace and energy. Components like turbine blades, heat shields, and exhaust components benefit from the superior thermal and mechanical performance of CMCs. Their lightweight nature further positions them as game-changers, contributing to fuel efficiency in aviation and improving overall system performance.

The manufacturing processes for CMCs encompass techniques like chemical vapor infiltration and liquid silicon infiltration. These methods ensure a uniform distribution of reinforcing elements within the ceramic matrix, optimizing the material's properties for diverse applications.

As technology continues to push the boundaries of what's possible, CMCs emerge as stalwart contenders, offering solutions that redefine the standards of high-performance materials across various industries.

I.5. Natural Fiber Composites (NFCs)

In the pursuit of sustainable and eco-friendly materials, NFCs have emerged as a compelling choice, harnessing the inherent strength of natural fibers to create robust and environmentally conscious composites.

NFCs are composed of a polymer matrix, often derived from renewable resources, and reinforced with natural fibers like jute, flax, hemp, or sisal. The amalgamation of these components results in materials that exhibit impressive mechanical properties while offering the advantages of being biodegradable and renewable.

One of the key attractions of NFCs lies in their low environmental impact. The cultivation and processing of natural fibers typically involve fewer energy-intensive processes compared to traditional synthetic fibers. Additionally, the biodegradability of these materials contributes to reduced environmental pollution, aligning with the principles of sustainability.

The natural fibers embedded in the composite matrix contribute to its strength, stiffness, and impact resistance. NFCs find applications in various industries, including automotive, construction, and packaging. Automotive components like door panels and interior parts benefit from the lightweight and durable nature of NFCs, leading to improved fuel efficiency.

The manufacturing processes for NFCs are diverse, ranging from compression molding to injection molding, allowing for versatility in shaping components for different applications. As technology and research in the field progress, NFCs continue to evolve, presenting an exciting avenue for sustainable materials that marry strength with ecological responsibility.

In a world increasingly conscious of its environmental footprint, NFCs emerge as a testament to the potential of marrying strength, functionality, and sustainability in the realm of advanced materials.

I.6. Hybrid Composites

In certain applications, hybrid composites leverage multiple matrix materials. For instance, combining a polymer matrix with metal or ceramic reinforcements can provide a balance of

lightweight design and enhanced mechanical properties. This approach is common in high-performance sporting goods and advanced structural components.

I.7. Role of the Matrix: Structural Support and Load Transfer

Beyond encapsulation, the matrix plays a crucial role in load transfer and distribution. It ensures that external forces are effectively transmitted to the reinforcing fibers, optimizing the composite's mechanical properties. The matrix also protects against environmental factors, preventing moisture, chemicals, and other elements from compromising the integrity of the composite.

In essence, the matrix material acts as the backbone of composite materials, dictating their mechanical and environmental performance. The choice of matrix type is a critical decision in the design of composites, as it profoundly influences the material's overall behavior and suitability for specific applications. The constant evolution of matrix materials contributes to the continual advancement of composite technology across diverse industries.

I.8. Reinforcing Materials (Fibers)

Reinforcing materials, often fibers like glass, carbon, or aramid, play a crucial role in enhancing the mechanical properties of composites. These fibers contribute to tensile strength, stiffness, and impact resistance. Their arrangement and orientation within the matrix significantly influence the overall performance of the composite. Fibers form the backbone of composites, reinforcing them for various applications.



Figure I.3. Principle types of fibers [12]

I.8.1. Glass Fibers

Glass fibers are a ubiquitous choice in composite reinforcement. Their high tensile strength, low-cost, and versatility make them suitable for diverse applications, ranging from automotive components to construction materials. Additionally, glass fibers exhibit good chemical resistance, enhancing the durability of composite structures.

Versatile Foundations

Glass fibers stand as stalwart pillars in the realm of composite materials, offering versatility and reliability. Composed primarily of silica, glass fibers provide a strong and lightweight foundation for a myriad of applications across industries.

Manufacturing Process

The production of glass fibers involves melting silica-based raw materials at high temperatures, drawing the molten material into fine threads, and then solidifying them rapidly. This process yields fibers with various diameters, allowing customization based on specific application requirements.

Strength and Stiffness

One of the key attributes of glass fibers is their impressive tensile strength and stiffness. This inherent strength is a result of the molecular alignment within the fibers. Glass fibers contribute significantly to enhancing the mechanical properties of composite materials, making them resilient under various loads.

Corrosion Resistance

An inherent quality of glass fibers is their resistance to corrosion, making them particularly suitable for applications where exposure to harsh environmental conditions is expected. This resistance ensures the longevity and durability of composite structures in challenging settings.

Cost-Effectiveness

Beyond their mechanical properties, glass fibers are known for their cost-effectiveness. The abundance of raw materials and the efficiency of manufacturing processes contribute to the affordability of glass fiber-reinforced composites. This economic advantage has played a pivotal role in the widespread adoption of glass fibers across industries.

Applications Across Industries

Glass fiber-reinforced composites find applications in diverse sectors, including automotive, construction, aerospace, and marine. They are integral to the production of lightweight yet robust components such as car bodies, boat hulls, and structural elements in buildings.

Innovations and Beyond

Continuous research and technological advancements in glass fiber manufacturing have led to innovations such as enhanced surface treatments and tailored fiber compositions. These

innovations aim to further optimize the performance of glass fibers in specific applications, opening doors to new possibilities.

There are several types of glass fibers used in composite materials, each with distinct properties and applications. The major types include:

Table I.1. Types and properties of glass fibers used in composite materials

Glass fiber	E-Glass (Electrical Glass)	S-Glass (High-Strength Glass)	C-Glass (Chemical-Resistant Glass)	A-Glass (Alkali Glass)	AR-Glass (Alkali-Resistant Glass)	D-Glass (Dielectric Glass)
Composition	Alumino-borosilicate glass	Alumino-silicate glass	Calcium-alumino-silicate glass	Alkali-lime glass	Alumino-lime silicate glass	High-silica glass
Properties	High electrical resistance, good strength, and stiffness	Higher tensile strength, stiffness, and temperature resistance compared to E-glass	Excellent chemical resistance, used in corrosive environments	Good resistance to alkalis, moderate strength	Specifically designed for resistance to alkali attack	High dielectric strength, low dielectric constant
Application	Construction, automotive, electrical insulation	Aerospace, military, high-performance structural components	Chemical industry, oil and gas, marine	Insulation, textiles, low-cost composites	Concrete reinforcement, where alkali resistance is crucial	Electrical insulators, electronic components

These types are distinguished by their chemical composition and are chosen based on the specific requirements of the application. The selection depends on factors such as strength, resistance to environmental conditions, and cost considerations.

In essence, glass fibers are foundational elements that have revolutionized the landscape of composite materials. Their enduring strength, corrosion resistance, and cost-effectiveness continue to position them as a material of choice for engineers and designers seeking reliable solutions across a spectrum of industries.

I.8.2. Carbon Fibers

Renowned for their exceptional strength-to-weight ratio, carbon fibers are pivotal in high-performance composites. Used extensively in aerospace, automotive, and sports industries,

carbon fibers provide outstanding stiffness and tensile strength. Their resistance to corrosion and heat makes them ideal for demanding applications.

I.8.2. Aramid Fibers

Kevlar, a well-known aramid fiber, offers impressive impact resistance and strength. Commonly used in ballistic applications such as bulletproof vests, aramid fibers find their way into composite materials for protective gear, sporting equipment, and structural components where impact resistance is critical.

I.8.3. Natural Fibers

Growing emphasis on sustainability has led to the integration of natural fibers like flax, jute, and hemp in composite materials. These fibers, derived from plants, offer a balance between strength and eco-friendliness. NFCs are employed in automotive interiors, furniture, and packaging materials.

I.8.4. Basalt Fibers

Basalt fibers, derived from volcanic rock, exhibit excellent heat resistance and durability. Composites reinforced with basalt fibers find applications in infrastructure, marine, and automotive industries. The fibers' resistance to corrosion and high-temperature environments enhances the performance of the composites.

I.8.5. Hybrid Fiber Composites

Hybrid composites leverage the strengths of multiple fiber types. For instance, combining carbon and glass fibers in a composite can achieve a balance between cost-effectiveness and high-performance. Hybridization allows engineers to tailor composites to meet specific requirements, providing a versatile solution.

The selection of reinforcing fibers depends on the desired properties of the composite. Engineers meticulously choose fibers based on strength, weight, cost, and environmental considerations to create composites that excel in diverse applications. As technological advancements continue, new and innovative reinforcing materials are continually emerging, broadening the horizons of composite engineering.

I.9. Fillers and Additives

Fillers and additives are incorporated to modify specific properties of the composite. Nanoparticles, flame retardants, and pigments are common examples. Fillers enhance certain characteristics, such as conductivity or flame resistance, while additives provide additional

functionalities. Careful selection of fillers and additives allows customization to meet the specific requirements of diverse applications.

Fillers and additives play crucial roles in modifying the properties of composite materials, contributing to their enhanced performance and functionality.

I.9.1. Fillers

Fillers are materials added to the composite matrix to improve specific characteristics such as strength, stiffness, and thermal conductivity. They can be particulate or fibrous and are typically smaller in size compared to the reinforcement fibers. Fillers are dispersed within the matrix, influencing the overall material behavior. Common filler materials include silica, alumina, carbon black, and various nanoparticles.

Incorporating fillers in powder form into composites significantly impacts the resulting material. The source of the powder, its structural properties, as well as the geometry and dimensions of its grains, all play a crucial role in determining the mechanical characteristics of the composite. A Research conducted is noteworthy for its focus on the development of environmentally friendly bio-composites by incorporating bio-waste horn powder (HP) into an epoxy resin matrix. The utilization of the Taguchi L9 (3×3) orthogonal array facilitated a systematic approach to composite fabrication. Through meticulous optimization using grey relational analysis and ANOVA, key parameters were identified to achieve optimal results. The study underscored the significance of factors such as NaOH concentration, HP size, and volume percentage in enhancing the properties of the bio-composites. Notably, the HP volume percentage emerged as the most influential factor, contributing significantly (90.87%) to the overall improvement of the material's characteristics. The success of the optimized specimen, demonstrating superior properties compared to untreated HP specimens, highlights the potential of these bio-composites for diverse applications across various industries [13]. Research conducted delves into the intricate relationship between various types of fillers (micro and nano) and the mechanical properties of epoxy matrix composites. By incorporating micro-fillers such as Al_2O_3 , TiO_2 , and fly ash at varying weight percentages, alongside nano-fillers like Al_2O_3 , TiO_2 , and clay, the study offers a comprehensive investigation. The observed trends suggest a trade-off, wherein increasing filler concentration leads to a reduction in tensile strength, flexural strength, and elongation percentage at fracture. However, the study identifies a significant increase in both tensile modulus and flexural modulus with higher filler content. This research sheds light on the nuanced interplay between filler types, concentrations, and the resulting mechanical properties of epoxy composites [14]. A study conducted focus on an often-overlooked aspect of particulate-polymer nanocomposites, specifically those incorporating fillers with small aspect ratios. In contrast to extensively studied high aspect ratio fillers such as layered silicate and carbon nanotubes, this

paper systematically investigates the influence of particle size, interface adhesion, and loading on the mechanical properties of these composites. The research aims to elucidate the mechanisms of stiffening, strengthening, and toughening unique to this category of materials. The authors offer a critical analysis of experimental findings, while also comparing them with theoretical models. The emphasis lies in fostering a comprehensive understanding of these mechanisms to facilitate the development of high-performance particulate composites [15]. Researchers conducted an investigation into the utilization of Cancun natural sand as a lightweight filler in epoxy composites, aiming to enhance properties without resorting to complex chemical treatments. Epoxy composites containing up to 5 wt % sand particles were assessed for thermal and mechanical properties. The findings revealed the highest flexural strength in the composite with 1 wt % sand particles. The study suggests that Cancun sand holds promise as a cost-effective filler for composites with improved mechanical and thermal properties, thereby positioning it as a viable natural filler for industrial applications [16].

Research investigates the influence of incorporating various volume fractions of iron powder on the mechanical properties of high-density polyethylene (HDPE). Through systematic adjustments of iron particle concentration, the study uncovers subtle effects on the mechanical behavior of HDPE. This investigation yields vital insights into customizing the properties of HDPE for specific applications through controlled modifications, thereby paving the way for enhanced material performance across diverse industrial settings [6]. The utilization of waste corundum particles in epoxy composites offers a sustainable approach to enhance material properties, specifically abrasive wear resistance and hardness. By incorporating Al_2O_3 waste as a filler in the composite matrix, an improvement in the wear resistance was demonstrated, indicating the material's ability to withstand abrasive forces. Additionally, the observed increase in hardness suggests that the composite exhibits greater resistance to deformation and penetration. This research underscores the potential of repurposing waste materials to create composite materials with enhanced mechanical performance, aligning with sustainable and eco-friendly practices in material development [17].

Furthermore, various studies have investigated the thermo-mechanical behavior of epoxy composites, incorporating diverse fillers such as E-glass fibers and iron oxide particles. Additionally, research has focused on the mechanical performance of glass/epoxy composites enhanced with both micro- and nano-sized aluminum particles. [18] [19]. Other studies have delved into composite materials that include iron powder and mixed glass fiber-reinforced polymers, with the aim of comprehending their properties and performance. Besides, researchers have examined the use of waste iron filings to enhance glass fiber-reinforced epoxy (GFRE) composites, analyzing the influence of factors such as particle size, interface adhesion, and loading on particulate-polymer composites [20] [21].

While previous research has significantly contributed to our understanding of the behavior of glass epoxy composites under challenging conditions, there remain important questions regarding the behavior of these laminates, particularly when loaded with iron powder in such conditions. This study aims to address these knowledge gaps by investigating how the amount of iron powder, in conjunction with environmental conditions, impacts the flexural strength of glass epoxy laminated composites. We will utilize statistical analysis (ANOVA) to assess their relative effects and potential interactions. The inclusion of iron powder is particularly relevant due to its abundance in the Annaba region, located in the eastern part of Algeria, owing to the presence of the sedimentary iron complex. Beyond addressing material availability, this choice aligns with sustainable practices, emphasizing the reuse of waste materials.

I.9.2. Additives

Additives are substances incorporated into composites to impart specific properties or enhance processing. They can influence aspects like curing, viscosity, flame resistance, and UV stability. Additives are diverse and may include plasticizers, stabilizers, UV absorbers, and flame retardants. The selection of additives depends on the intended application and the desired performance characteristics of the composite.

I.9.3. Benefits of Fillers and Additives

Enhanced Properties: Fillers contribute to increased strength, stiffness, and thermal conductivity, while additives can improve flame resistance and UV stability.

Cost Efficiency: The use of fillers is often a cost-effective way to enhance certain properties without significantly increasing material costs.

Processability: Additives can facilitate the processing of composite materials, improving aspects such as viscosity, curing time, and flow behavior during manufacturing.

Tailored Performance: By carefully selecting fillers and additives, engineers can tailor the composite's properties to meet specific application requirements.

Considerations

- **Compatibility:** It's crucial to ensure compatibility between fillers, additives, and the matrix material to prevent adverse reactions that could affect the composite's overall performance.
- **Loading Levels:** The amount or loading level of fillers and additives must be optimized to achieve the desired properties without negatively impacting other aspects of the material.

Processing Conditions

The incorporation of additives should consider processing conditions to avoid degradation or unwanted reactions.

In summary, the synergy between the matrix, reinforcing fibers, fillers, and additives defines the performance and versatility of composite materials. The matrix provides a structural framework, reinforcing fibers impart strength, and fillers/additives fine-tune properties for specific applications. The careful integration of these elements showcases the ingenuity behind composite materials, making them indispensable across industries.

I.10. Advantages of composite materials

High-Strength-to-Weight Ratio: Composites can be significantly stronger than traditional materials, such as metals while being lighter.

Tailored Properties: The combination of different materials allows for tailoring properties like strength, stiffness, and thermal conductivity to meet specific requirements.

Corrosion Resistance: Unlike metals, many composites do not corrode, making them suitable for harsh environments.

Design Flexibility: Composites offer designers greater flexibility in shaping components for optimal performance.

Fatigue Resistance: Composite materials often exhibit better fatigue resistance than traditional materials.

Applications:

- **Aerospace:** Composites are widely used in aircraft components to reduce weight and improve fuel efficiency.
- **Automotive:** Car parts like body panels, bumpers, and interior components benefit from the lightweight and durable nature of composites.
- **Construction:** Composites find applications in infrastructure, providing high-strength materials for bridges, buildings, and pipelines.

The elaboration methods for composites can vary depending on the type of composite (polymer matrix, metal matrix, ceramic matrix, etc.) and the desired application. Here, let's delve into the elaboration methods for PMCs, which are among the most common types.

I.11. Elaboration Methods for PMCs

I.11.1. Hand Lay-Up

This is a simple and manual process where layers of reinforcement (usually fibers) are manually placed in a mold.

The polymer matrix, often in the form of resin, is then poured or brushed onto the reinforcement layers.

The layers are compacted to remove air and ensure good impregnation.

I.11.2. Spray-Up

Spray-up involves simultaneously spraying chopped fibers and resin onto a mold. This method is efficient for quickly building up layers and is often used for large and relatively simple structures. However, achieving precise fiber orientation can be more challenging compared to other methods.

I.11.3. Filament Winding

Filament winding utilizes continuous fibers, such as rovings or tows, that are wound onto a rotating mandrel. This method allows for controlled winding patterns, offering flexibility in creating structures with desired strength and stiffness. Filament winding is suitable for producing cylindrical or tubular shapes.

I.11.4. Pultrusion

Pultrusion involves pulling continuous fibers, typically rovings or mats, through a resin bath. The impregnated fibers then pass through a heated die, where the polymer matrix cures and solidifies. Pultrusion is well-suited for creating continuous profiles with a constant cross-section, offering high-strength and durability.

I.11.5. Resin Transfer Molding

RTM entails injecting liquid resin into a closed mold containing reinforcing fibers. The mold is filled under pressure to ensure proper impregnation of the fibers. RTM is suitable for producing complex shapes with a high degree of detail and is commonly used in automotive and aerospace applications.

I.11.6. Prepregs

Prepregs are sheets of reinforcing fibers pre-impregnated with resin, often partially cured to a B-stage. These sheets are laid up in the desired orientation and then cured under heat and pressure. Prepregs offer precise control over fiber content and orientation, making them ideal for high-performance applications.

I.11.6. Vacuum Bagging

Vacuum bagging involves laying up reinforcement layers and resin in a mold, and then using a vacuum bag to remove air and compact the layers. This method is effective for producing high-quality composites with good consolidation. Vacuum bagging is often used in combination with other methods to enhance composite properties.

I.11.7. Extrusion

Extrusion involves forcing a composite material, typically in the form of pellets or granules, through a shaped die to produce continuous profiles or specific shapes. In the case of composite materials, this process is often used for thermoplastic matrix composites. Continuous fibers or

fillers may be incorporated during the extrusion process to enhance specific properties. Extrusion is advantageous for its efficiency in producing continuous lengths of composite with consistent cross-sectional shapes.

These methods offer flexibility and can be tailored to the specific requirements of the composite and the intended application. The choice of method depends on factors such as geometry, production volume, and the desired mechanical properties.

I.12. Damage Modes in Laminated Composites

Damage modes in laminated composites represent critical aspects that influence the structural integrity and performance of composite materials. This chapter provides an in-depth exploration of the various modes of damage that laminated composites may undergo during their lifecycle, delving into their mechanisms, detection, and implications for structural applications.

I.12.1. Modes of Damage

Laminated composites can experience different modes of damage, each with its distinct characteristics and implications. The primary modes include:

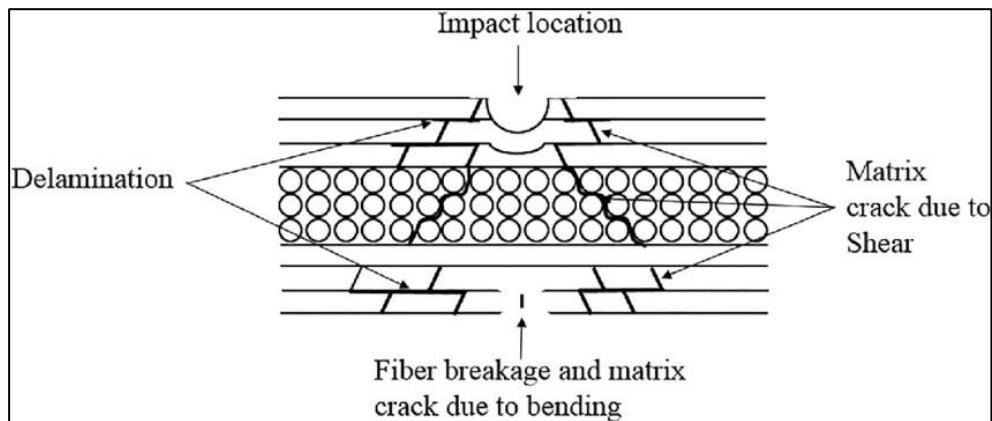


Figure I.4. Schematic representation of impact damage mode of composite laminate [22]

Table I.2. Damage Modes in Laminated Composites

Delamination
Delamination is a common and critical mode of damage in laminated composites, characterized by the separation of layers along the laminate thickness. It can result from various factors, including impact, fatigue, or manufacturing defects. Delamination significantly compromises the structural integrity of the composite and is a focal point in damage assessment.
Matrix Cracking
Matrix cracking involves the development of fractures within the resin matrix of the composite. These cracks may propagate due to external loads, thermal cycling, or other environmental

factors. Matrix cracking can affect the load-carrying capacity of the composite and is often considered in the context of structural health monitoring.

Fiber Breakage

Fiber breakage occurs when the reinforcing fibers within the composite fracture. This mode of damage is typically associated with overloading or localized stress concentrations. Fiber breakage can lead to a reduction in the composite's strength and stiffness, affecting its overall mechanical performance.

Fiber-Matrix Debonding

Fiber-matrix debonding refers to the separation between the reinforcing fibers and the surrounding matrix. This phenomenon can arise from incompatible material properties, thermal stresses, or environmental effects. Debonding can compromise the transfer of loads between fibers and matrix, impacting the load-carrying capacity of the composite.

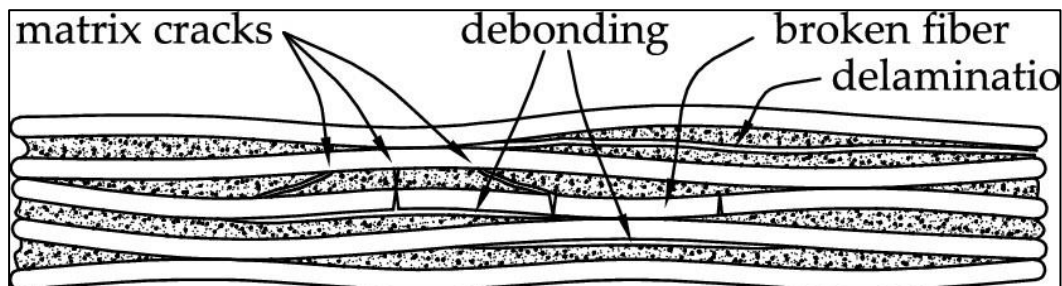


Figure I.5. Matrix cracks, broken fibers, debonding, and delamination [23]

I.13. Implications for Structural Applications

Understanding and mitigating damage modes in laminated composites are crucial for enhancing their structural reliability and longevity. Advanced modeling techniques, predictive maintenance strategies, and material advancements aim to minimize the impact of damage on composite structures in various applications, including aerospace, automotive, and civil engineering.

I.14. Hardness Theory in composite materials

I.14.1. Introduction

Hardness is a primary mechanical feature that is equally as important to examine in composites as other mechanical properties since it is critical in determining how resistant composite materials are to deformation, wear, and penetration. Researchers investigated the hardness properties of a hybrid composite comprising Carbon Fiber Reinforced Polymer (CFRP) and Glass Fiber Reinforced Polymer (GFRP) which are commonly used in car manufacturing. The aim is to enhance mechanical properties and reduce costs. Micro-hardness testing was conducted,

revealing that the hybrid composite's hardness falls between CFRP and GFRP values [24]. In a study of the NFCs, using areca fiber as a reinforcement and epoxy resin as the matrix, were evaluated for their mechanical properties. Areca fibers, extracted from areca husk, underwent chemical treatment to enhance the fiber-matrix interfacial bonding. The composites, prepared with varying fiber-to-matrix ratios, were subjected to impact and hardness tests. The results indicate that an increase in fiber volume fraction and composite post-curing time leads to improved mechanical properties [25]. An investigation on the surface micro-hardness (VHN) and fracture toughness (KIC) of resin-composites was made, with and without incorporated short fibers, after solvent storage, the results indicated a decrease in VHN with storage time, particularly in 75% ethanol/water. The inclusion of short fibers enhanced the fracture toughness of ever XTM. However, no significant benefits were observed for nano-fiber containing composites in terms of fracture toughness or hardness compared to control composites. Extended water storage for 7 days did not significantly alter KIC values for all resin-composites relative to 1 day storage [26].

The present work emphasizes the effect of composite filler for different contents added in weight percent (15% 20% and 25%) of pure iron powder on the mechanical characteristics of an epoxy resin matrix laminate, reinforced with E-glass fiber mat. Furthermore, the influence of iron grains' dimensions on the mechanical characteristics are also studied.

The hardness test was carried out in this work, as well as three points bending test, on a number of specimens that have been manufactured according to L9 (3 × 3) Taguchi's orthogonal array.

This chapter delves into the theoretical aspects of hardness in composite materials, exploring the underlying principles, measurement techniques, and factors influencing hardness.

I.14.2. Theoretical Foundations of Hardness

Hardness, a fundamental measure of a material's resistance to localized deformation or indentation, is a critical property significantly influenced by various factors within composite materials. Composites, characterized by their combination of reinforcing fibers and a matrix, derive their overall hardness from the collaborative contribution of both these elements.

Firstly, the hardness of the matrix, typically composed of polymers or metals, stands as a primary determinant in the material's ability to resist deformation when subjected to external forces. The type of matrix material and its inherent hardness profoundly influence the composite's overall resistance to localized stress and wear.

Secondly, the hardness of the reinforcing elements, such as carbon, glass, or aramid fibers, plays an equally crucial role in defining the composite's hardness profile. The inherent hardness of these fibers contributes to the overall structural integrity and durability of the composite material.

Moreover, the strength of the interfacial bonding between the matrix and reinforcing elements is a pivotal aspect influencing hardness. A robust interface ensures effective load transfer between the matrix and fibers, preventing premature failure and enhancing the composite's ability to withstand external pressures.

In summary, the intricate interplay of matrix hardness, reinforcing element hardness, and interfacial bonding strength collectively determine the hardness characteristics of composite materials. This multifaceted approach to hardness assessment is essential for understanding and optimizing the performance of composites in various applications.

I.14.3. Measurement Techniques

Several methods are employed to measure hardness in composite materials:

Micro-hardness Testing

Involves applying a known load to a small indenter and measuring the resulting indentation size. Common methods include Vickers and Knoop hardness tests, providing localized hardness values.

Macrohardness Testing

Often performed using methods like Rockwell or Brinell Hardness tests, applies larger loads to assess overall material hardness. These tests are valuable for bulk hardness characterization.

Nanoindentation

Involves applying extremely small loads to measure hardness at the nanoscale. This technique is valuable for assessing localized hardness variations and understanding material behavior at the micro- and nanolevels.

I.14.4. Factors Influencing Hardness in Composites

Various factors impact the hardness of composite materials, including:

Reinforcement Type: Different reinforcing materials exhibit varying hardness, influencing the overall composite hardness.

Loading Conditions: Hardness can be load-dependent, with materials responding differently under various applied loads.

Temperature and Environmental Effects: Changes in temperature and exposure to environmental conditions can affect hardness, especially in PMCs.

I.14.5. Equations and Models

In Hardness Theory, specific equations and models can be applied to predict or measure hardness values. Some commonly used equations include:

Vickers Hardness (HV):

$$HV = \frac{1.854 \times F}{d^2}$$

Brinell Hardness (HB):

$$HB = \frac{2 \times F}{\pi \times D \times (D - \sqrt{D^2 - d^2})}$$

Nanoindentation Hardness (H):

$$H = \frac{2 \times P}{\pi \times d \times (2 \times \sqrt{r})}$$

Where:

F (N) is the applied force,

d (m) is the diagonal length of the indentation,

D (m) is the diameter of the indenter,

P (N) is the applied load during nanoindentation,

r (m) is the indentation radius.

Understanding Hardness Theory in composite materials is essential for optimizing material selection, manufacturing processes, and predicting performance in real-world applications. Tailoring composites for specific hardness requirements ensures their effectiveness in diverse industries, including aerospace, automotive, and construction.

I.15. Three-Point Bending in Composite Materials

I.15.1. Introduction

Three-point bending is a critical mechanical test employed to evaluate the flexural properties of composite materials. This chapter delves into the theoretical foundations, testing methodologies, and influencing factors in three-point bending tests, providing comprehensive insights into the behavior of composite materials under bending loads.

Therefore, a number of recent scientific publications have focused on the analysis of the mechanical behavior of composites. Accurately determining the bending properties of laminates is crucial since bending loads is taken into consideration in many laminate composite applications.

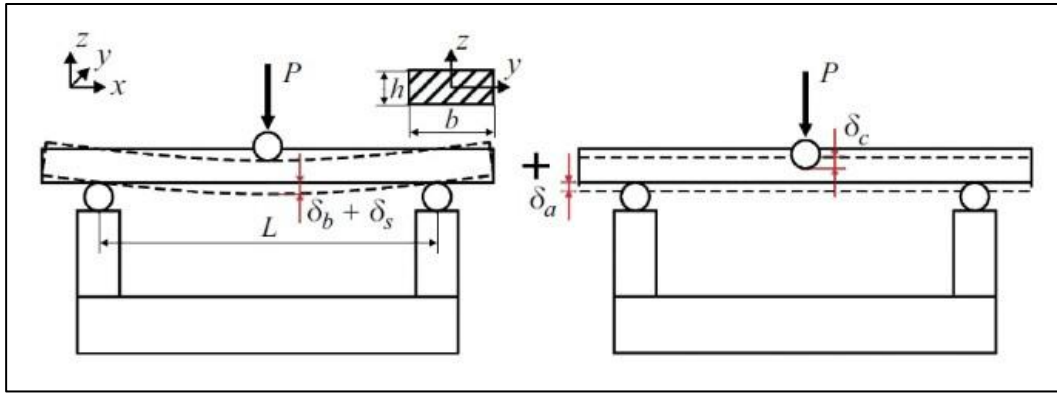


Figure I.6. Three points bending test [27]

The failure of unidirectional carbon fiber (LCU250NS) and glass fabric (HG181) laminated composites subjected to bending loads was examined [28]. The flexural characteristics of bidirectional hybrid epoxy composites with T700S carbon fibers and E-glass in inter-ply arrangements were investigated. Test specimens are made by hand lay-up, and put through a three-point bend test, and their flexural characteristics are evaluated using analytical Classic Lamination Theory (CLT) and numerical finite element analysis (FEA). The results indicate that the model predictions and the experimental data accord well. Although there are no discernible hybrid effects on flexural strength in the studies, simulations suggest that hybridization may be able to increase flexural strength. [29]. Research employed a theoretical approach to calculate the flexural strength of a composite material comprising Araldite resin CY223 and varying weight percentages (20%, 40%, 60%) of woven roving carbon fibers (0° - 45°). Utilizing the Ansys program version 11, the study assessed the impact of different reinforcement configurations on flexural strength under varied loading conditions. The theoretical results highlight an increased flexural strength for Araldite resin CY223 when reinforced with carbon fibers, showing an upward trend with higher fiber percentages [30]. other studied the mechanical behavior in 3-point bending fatigue on two types of sandwich materials [31].

I.15.2. Theoretical Background

In three-point bending tests, a load is applied to a specimen, inducing bending stresses and strains. The distribution of bending stress across the specimen's cross-section is a critical factor, with outer fibers experiencing tensile stresses and inner fibers undergoing compressive stresses, creating a neutral axis with minimal stress. The composite flexural modulus, or bending modulus, which signifies the material's stiffness in bending, is influenced by both the matrix and reinforcing elements. Understanding failure modes is crucial, as fiber rupture, matrix cracking, and delamination are common occurrences that impact the overall bending behavior of the material.

I.15.3. Testing Methodologies

Three-point bending tests follow standardized procedures, including specimen preparation, loading configuration, and data analysis:

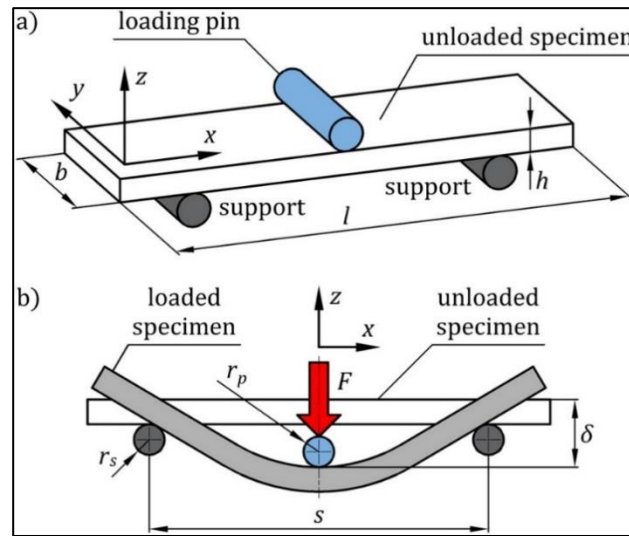


Figure I.7. Shape and dimensions of typical flexure specimen and three-point flexure loading configuration [32]

Table I.3. Flexure Testing Methodologies

Specimen Preparation	Loading Configuration	Data Analysis
Specimens are typically rectangular beams with specified dimensions. Careful preparation ensures accurate test results, considering factors like fiber orientation and volume fraction.	A test apparatus applies a load at the center of the specimen, creating a bending moment. The span length and loading rate are controlled to adhere to testing standards, ensuring reproducibility.	Key parameters, such as flexural strength and flexural modulus, are derived from load-deflection curves. Analysis involves understanding the specimen's response to bending and identifying failure points.

Table I.4. Factors Influencing Three-Point Bending Behavior.

Fiber Orientation	Matrix Properties	Loading Rate
The alignment of reinforcing fibers significantly influences bending properties.	The matrix's flexibility and resistance to deformation contribute to the overall bending behavior.	The rate at which the load is applied affects the material's viscoelastic response.

I.15.4. Equations and Models

Flexural Strength σ (N/m²):

$$\sigma = \frac{3 \times F \times L}{2 \times b \times h^2}$$

Flexural Modulus E (N/m²):

$$E = \frac{L^3 \times F}{4 \times b \times h^3 \times d}$$

Where:

F (N) is the applied force,

L (m) is the span length,

b (m) is the width of the specimen,

h (m) is the thickness of the specimen,

d (m) is the deflection at the center.

I.16. Tensile Behavior of composite materials

I.16.1. Introduction

This chapter delves into the intricate world of tensile behavior in composite materials, a critical aspect that defines their performance under axial loads. Understanding how composites respond to tensile stress is paramount for engineers aiming to optimize their use in diverse applications.

The tensile strengths of several carbon fabric-reinforced epoxy composites were compared. The laminates were created by hand lay-up after the proper autoclave curing cycle. Tensile tests were performed on the samples in accordance with ASTM D3039 [33]. The methods for assessment of the interlaminar tensile strength of composite materials were studied in order to develop efficient and accurate methods to capture the interlaminar tensile strength of composites. The study challenges ASTM Standard D 6415's effectiveness in assessing interlaminar tensile strength, proposing an efficient short-beam method with digital image correlation to address manufacturing challenges and discrepancies observed in curved-beam tests for composite structural designs [34]. A research studied the influence of specimen configuration and size on the transverse tensile strength of two glass/epoxy materials, the study examined how specimen configuration and size affect transverse tensile strength in glass/epoxy and carbon/epoxy materials, revealing lower strength with longer span lengths and highlighting limitations in the Weibull scaling law for predicting transverse tensile strength in fiber-reinforced polymer composites [35]. The results of a study on fiberglass-reinforced polymer composite materials in

which each test examining variations in fiberglass lamination (CSM 300, CSM 450, and WR 600) and weight percentages (ranging from 99.5%-0.5% to 97.5%-2.5%) highlight an increase in tensile strength with more laminates, while higher hardener weight percentages lead to a decrease in tensile strength. This research underscores the significance of tensile testing for optimizing the mechanical properties of fiber boats [36].

Research explores the use of gourd sacks as a substitute for glass fiber in rowing, assessing its strength through pull tests to determine its material properties [37]. A study delved into epoxy resin-composites reinforced with carbon fiber, examining the impact of fiber orientations, resin types, and laminates on mechanical properties in terms of tensile, flexural and impact strengths. Results highlighted the significant influence of fiber orientations and the number of laminates, with EM500 epoxy resin showing slightly superior properties. The hierarchy of influencing factors was identified as fiber orientation > number of laminates > resin type, emphasizing the importance of $\pm 35^\circ$ fiber orientations for superior tensile and flexural properties [38]. An investigation made, explore the tensile strength of unidirectional carbon fiber-reinforced plastics at high strain rates using the tension-type split Hopkinson bar technique. Findings revealed that tensile modulus and strength in the longitudinal direction were independent of strain rate, while transverse and shear properties increased with strain rate. The strain-rate dependence of shear strength was notably stronger than that of transverse strength. The tensile strength of off-axis specimens was effectively characterized using the Tsai-Hill failure criterion under dynamic loading conditions [39]. A study investigates jute fiber composites made with vacuum-assisted resin infiltration (VARI) techniques and different stacking sequences. The composites, containing 25% jute fiber, undergo tensile and three-point bend tests, with results compared to theoretical values. Fracture surfaces are examined using high-resolution FEG SEM. In the 0/0/0/0 and 0/+45°/-45°/0 laminate, longitudinal tensile strength surpasses the transverse direction, while the 0/90°/90°/0 laminate shows comparable strengths in both directions. The study highlights the strong influence of jute fiber tensile strength and its sensitivity to defects on composite properties, along with insights into mechanical behaviors from observed fracture morphologies under SEM [40]. A study investigates the tensile behavior of Fiber Metal Laminates (FMLs), combining aluminum alloy with glass and Kevlar fiber-reinforced plastic. Analyzing the influence of fiber orientation on mechanical properties, the study uses modified CLT and finite element modeling (FEM) for predictive accuracy. Findings reveal enhanced properties with zero-oriented fiber sheets. Statistical analysis highlights Kevlar fiber orientation as a crucial factor, offering insights into variable significance and interactions in FML tensile behavior [41].

I.16.2. Tensile Stress-Strain Relationship

The relationship between tensile stress and strain (ϵ) in composites is crucial for assessing their mechanical response. The general tensile stress-strain curve can be represented as:

$$\sigma_{tensile}(N/m^2) = \frac{F}{A_0}$$

where

F (N) is the applied force and A_0 (m^2) is the original cross-sectional area.

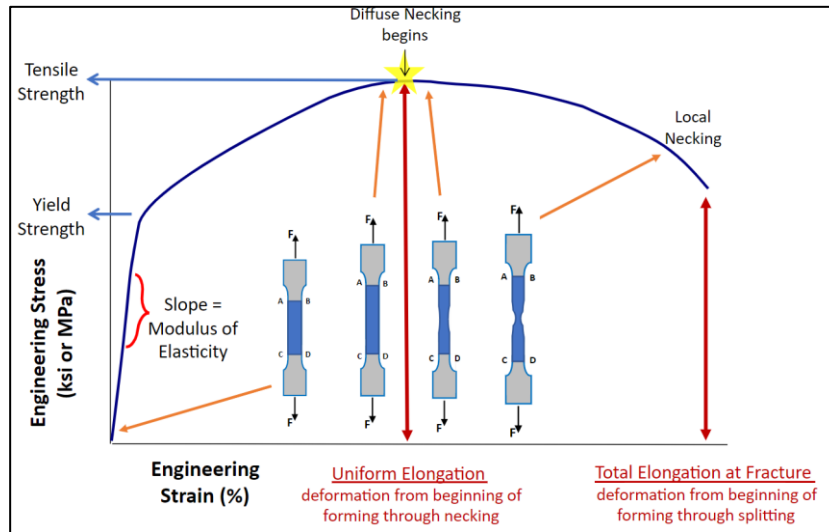


Figure I.8. The engineering stress-strain curve serves as the foundation for deriving mechanical properties [42]

I.16.3. Factors Influencing Tensile Behavior

Table I.5. Factors Influencing Tensile Behavior.

Fiber Orientation	Matrix Properties	Loading Rate
The alignment of fibers significantly affects the tensile strength and modulus of the composite.	The matrix material's characteristics, such as ductility and elasticity, play a role in determining tensile behavior.	The rate at which tensile loads are applied can influence the material's response.

Predicting the tensile modulus (E tensile) in composite materials involves a comprehensive consideration of the elastic properties of both the reinforcing fibers and the matrix. Models such as the Halpin-Tsai equations play a crucial role in estimating the tensile modulus. Concurrently, understanding when and how composites fail under tensile stress is imperative for design and structural integrity. Failure criteria, exemplified by the Tsai-Hill criterion, provide valuable

insights into potential failure modes. Experimental techniques, including tensile testing, acoustic emission analysis, and microscopy, serve as essential tools for researchers and engineers to characterize the tensile behavior of composites. This knowledge, in turn, informs applications and design considerations, guiding the selection of composite materials for specific uses to ensure optimal performance and structural reliability.

I.17. Environmental Durability of Composite Materials

I.17.1. Introduction

The burgeoning use of composite materials across diverse industries necessitates a nuanced exploration of their response to harsh environmental conditions. This chapter delves into the intricate interplay between composite materials and challenging environments, emphasizing the significance of understanding their behavior under various stressors.

While glass epoxy composites boast exceptional strength, they encounter challenges when exposed to harsh conditions. Prolonged contact with corrosive substances, such as acids or seawater, poses a significant threat to the integrity of these materials, particularly to their flexural strength [43], the vulnerability of glass epoxy composites to corrosion underscores the importance of considering environmental factors in their application. Engineers and designers must implement strategies to mitigate the impact of corrosive environments, such as incorporating protective coatings or exploring alternative composite formulations that offer enhanced resistance to chemical degradation. Balancing the impressive strength of glass epoxy composites with strategies to address specific environmental vulnerabilities is crucial for optimizing their performance in diverse applications. Epoxy resins are used to hold these materials together and protect them from harsh conditions like water and salt [44]. However, epoxy can absorb moisture over time, which can harm its physical properties and the components made with it [44]. Researchers studied the effects of seawater and temperature on glass/epoxy and glass/polyurethane composites. After a year of immersion, glass/epoxy composites absorbed 2.5% of their weight in water at ambient temperature and 5% at 65°C. Tensile strength was reduced by 0.8% at room temperature and 6% at 65°C, indicating accelerated water absorption and reduced strength at higher temperatures [45]. Researchers explored the degradation of GFRP laminates through 2500 hours of immersion in a saline solution. Tensile strength retention was 84% at 35°C, 70% at 50°C, and 61% at 65°C, revealing swelling and plasticization in the GFRP composites, leading to microcracking and fractures [46]. A study focused on SiO₂-epoxy polymer nanocomposites and presented intriguing findings regarding their mechanical resilience in seawater. Unlike some traditional materials that experience significant strength reduction in corrosive environments, this research suggests that SiO₂-epoxy nanocomposites demonstrate

minimal strength reduction over a six-month exposure period in seawater. The optimal performance observed at a 3% SiO₂-epoxy ratio indicates the potential for tailored formulations to enhance resistance to seawater-induced degradation. This finding is noteworthy for applications where materials are exposed to marine environments, such as maritime structures or equipment. The study's implications may extend beyond mechanical properties, potentially influencing the design and selection of materials for various marine applications. Further exploration into the long-term durability, stability, and other relevant properties of SiO₂-epoxy polymer nanocomposites in seawater could offer valuable insights for engineers and material scientists working in marine engineering and related fields [47]. Other researchers evaluated the impact of corrosive conditions on epoxy nanocomposites with BN nanoplatelets, enhancing mechanical and tribological properties. And found that, despite the corrosive influence on mechanical performance, tribological behavior improves with exposure, acting as a lubricant and forming a protective layer [48].

Researchers have conducted numerous studies on the impact of water and seawater on GFRP composites, assessing factors like flexural strength, tensile strength, compressive strength, and elastic modulus, the investigation was conducted on the water absorption and mechanical properties of an epoxy resin casting and glass fiber reinforced epoxy matrix composites (GFRP) immersed in artificial seawater. GFRP composites with a higher fiber volume fraction (V_f) exhibited increased water absorption due to more micropores and capillary paths. After 42 days of immersion, tensile strength, flexural strength, and ILSS decreased, with desorption partially restoring the original properties. Irreversible changes in the resin casting and fiber/matrix interface failures contributed to the observed mechanical property variations [49], the study explored the moisture absorption of untreated and alkali-treated water hyacinth (WH) fibers. Alkali treatment did not significantly reduce moisture absorption in individual WH fibers. Scanning electron microscopy revealed swollen cell walls in treated WH fibers. The wet composite samples showed lower tensile and flexural strength than dried ones, with a minimal enhancement in mechanical properties with increased WH fiber volume fraction in the unsaturated polyester (UPR) matrix [50]. Researchers examined the effects of concentrated HCl on aged fiber-glass polyester composites. After 21 days of acid immersion at 35°C, a significant destructive impact was observed, as acid infiltrated the composite, forming cavities and cracks, and reducing mechanical properties [51]. Additionally, the flexure strength and modulus of elasticity in a glass fiber/epoxy composite decreased over time when exposed to hydrochloric acid (HCl) and sodium hydroxide (NaOH), resulting in mechanical degradation [52]. Similar degradation was observed in Kevlar/epoxy and carbon/epoxy laminates subjected to low-velocity impact tests following immersion in HCl and NaOH in which the impact strength is significantly affected by the aggressive

solutions, with concentrations playing a crucial role. Temperature variations also impact the performance and residual bending strength, regardless of the aggressive solution used [53].

I.17.2. Corrosion in composite materials

Corrosion, a pervasive issue in material science, poses a unique challenge for composite materials. This section provides a detailed examination of the corrosion mechanisms affecting composites. The corrosive impact of substances like acids and seawater on different composite types is explored, highlighting the specific vulnerabilities and potential degradation pathways.

I.17.3. Flexural Strength under Harsh Conditions

Flexural strength, a key mechanical property, is critically evaluated in the context of prolonged exposure to corrosive environments. A thorough exploration of how composites respond to flexural loads under harsh conditions provides insights into their durability, load-bearing capabilities, and the complex interplay between mechanical properties and environmental stress.

I.17.4. Mitigation Strategies

Mitigating the impact of harsh environmental exposure is paramount for ensuring the longevity of composite structures. This section explores proactive strategies, including the application of protective coatings, meticulous material selection, and design modifications aimed at enhancing the resistance of composites to corrosive elements. Real-world examples and case studies illustrate the effectiveness of these strategies.

I.17.5. Weathering and UV Effects

Beyond corrosive substances, the chapter scrutinizes the impact of weathering and UV radiation on composite materials. Detailed discussions cover the mechanisms leading to degradation, discoloration, and alterations in mechanical properties. The exploration goes beyond surface-level effects, delving into the underlying processes influencing the long-term behavior of composites under various environmental stressors.

I.17.6. Composite Aging and Long-Term Performance

Aging is a critical aspect influencing the long-term performance of composite materials in harsh environments. This section navigates through factors contributing to composite aging, including temperature fluctuations, humidity, and chemical exposure. The chapter provides a holistic understanding of how these factors collectively influence the mechanical and structural characteristics of composites over time.

I.17.7. Testing and Evaluation Protocols

Robust testing protocols are essential for accurately assessing the environmental durability of composites. This section outlines standardized methodologies for evaluating corrosion resistance, weathering effects, and long-term performance. In-depth discussions on testing procedures equip researchers and engineers with a comprehensive toolkit for conducting precise and meaningful assessments.

I.18. Fatigue Behavior of composite materials

I.18.1. Introduction

This chapter delves into the intriguing realm of fatigue behavior in composite materials, exploring how repeated loading over time can influence their structural integrity. Understanding fatigue is pivotal for ensuring the reliability and longevity of composite structures in various applications.

Fatigue refers to the weakening of a material over time due to cyclic loading. In composites, which often serve in dynamic conditions, fatigue resistance is a critical consideration.

The study of fatigue performance in composite materials has been integral to their integration into various engineering applications since the 1950s. Over the past 70 years, research in this domain can be categorized into three distinct periods.

The initial period, spanning from 1950 to 1975, witnessed the discovery of these "new" materials and the exploration of their behavior under simple fatigue loading patterns. Researchers during this time focused on understanding the fundamental aspects of how composite materials responded to fatigue.

The mature period, from 1975 to 2000, marked a phase of deeper investigation, where more loading and material parameters were explored. This era established the basic theoretical foundations for understanding the fatigue behavior of fiber-reinforced polymer composite laminates.

In the new millennium, the field entered a later period characterized by more detailed experimental campaigns. This phase benefited from advancements in various engineering and scientific disciplines. Researchers began considering parameters that were previously overlooked, leading to a more comprehensive understanding of the fatigue performance of composite materials.

In summary, the history of fatigue investigations in fiber-reinforced polymer composite laminates has evolved through distinct periods, each contributing to a richer understanding of the material's behavior under varying conditions [54].

A study delves into the fatigue behavior and damage mechanisms of symmetrical thin/thick hybrid design laminate-titanium joints subjected to tension-tension loading. Four laminate configurations, including thick, thick-thin-thick, thin-thick-thin, and thin, were investigated. Fatigue tests at two load levels were conducted based on the quasi-static bearing limit of thick-ply laminate joints. The investigation, aided by scanning electron microscopy (SEM), revealed that incorporating thin plies, especially on the outermost layers, significantly enhances fatigue performance. Thin-ply laminates demonstrate approximately 50% less deformation around a hole and delayed damage, leading to superior fatigue resistance and reduced delamination compared to thick-ply laminates under the same number of cycles [55].

This study explores fatigue delamination growth (FDG) behavior in carbon-fiber-reinforced polymer composites under mode I loading, considering fiber bridging at various R-ratios and temperatures. Utilizing unidirectional double cantilever beam (DCB) specimens, the study introduces a fatigue model based on parameters like strain energy release rate (SERR). The model effectively collapses FDG data, revealing accelerated growth at higher temperatures and a decrease at sub-zero temperatures. Strong correlations between model parameters and temperature offer insights for accurate predictions under different thermal conditions. Fractographic examinations highlight temperature-dependent FDG damage mechanisms, enhancing our understanding of composite material responses [56].

A study focuses on modeling damage evolution in carbon/epoxy laminated composites under static and fatigue loading. A cumulative damage model is developed, integrating static and fatigue-induced damage during cyclic loading. The model employs continuum damage mechanics (CDM) coupled with micromechanics to predict fatigue behavior. A multicriterion approach predicts damage in longitudinal, transverse, and shear directions at the ply scale. Experimental results on T300/EPL1012 carbon/epoxy laminates validate the model, showing accurate predictions of damage evolution under both static and fatigue loading. Consideration of damage due to static loading enhances accuracy, especially in low-cycle fatigue conditions [57].

Research investigates the impact of aging on the fatigue performance of glass/epoxy quasi-isotropic laminates. Specimens underwent 180 days of aging in artificial seawater under various conditions. Static tensile and dynamic tension-tension fatigue tests were conducted on both pristine and aged specimens, revealing that fatigue damage growth depends on aging conditions and moisture content. The deterioration of fatigue properties is attributed to factors such as multiple cracks, interfiber debonding, and matrix decomposition from the fiber surface [58].

Numerical and experimental studies have explored the impact of Automated Fiber Placement (AFP) defects on the quasi-static and fatigue behavior of composite parts. This review covers

identified types of AFP defects and their influence on laminate response and failure characteristics during both static and fatigue loading. The document is structured to introduce AFP defects, discuss their impact on quasi-static behavior, delve into numerical investigations of fatigue behavior, explore combined experimental and numerical studies, and compare the time costs associated with these approaches [59].

I.18.2. Fatigue Loading Conditions

Composites experience various loading conditions, including tension-tension ($R = 0$), tension-compression ($R = -1$), and fully reversed tension-compression ($R = 1$). Each loading condition has a distinct impact on fatigue life.

I.18.3. Fatigue Life Prediction Models

Several models, such as the S-N (Stress-Number of cycles) curve, are employed to predict the fatigue life of composites. These models consider factors like stress amplitude (σ_a), mean stress (σ_m), and loading frequency (f).

The S-N curve equation can be expressed as:

$$\sigma_a \text{ (N/m}^2\text{)} = \sigma_m \times N^{\frac{-1}{m}}$$

Where:

N is the number of cycles to failure, and

σ_m is a material constant.

7.5 Factors Affecting Fatigue Behavior

Understanding the factors influencing fatigue behavior is crucial:

Table I.6. Factors Affecting Fatigue Behavior.

Material Properties	Loading Frequency	Environmental Effects
The matrix and fiber properties significantly impact how composites respond to fatigue.	The rate at which cyclic loading occurs affects fatigue life.	Factors like temperature and humidity can influence fatigue resistance.

I.18.4. Experimental Techniques and Design Considerations

Experimental techniques play a pivotal role in understanding the fatigue behavior of composites, with fatigue testing and microscopy being prominent methods. These approaches provide valuable insights into how composites respond to cyclic loading and potential failure modes over time. Designing composite structures with fatigue considerations requires a holistic approach. Factors such as load spectrum, stress concentrations, and material selection become crucial considerations to enhance durability. By incorporating insights gained from experimental techniques into the design process, engineers can create composite structures that effectively withstand cyclic loading, ensuring long-term reliability and performance.

I.19. Conclusion

In conclusion, the exploration of composite materials has been comprehensive, spanning from an Introduction to the Constituents of Composite Materials. The review has delved into the types of composite materials based on their matrices, emphasizing the crucial role of the matrix in providing structural support and facilitating load transfer. The discussion extended to the various reinforcing materials, such as fibers, and the significance of fillers and additives in enhancing composite properties.

The advantages of composite materials were elucidated, showcasing their appeal in numerous applications. The chapter also provided insights into the elaboration methods for Polymer Matrix Composites (PMCs) and examined damage modes in laminated composites. The incorporation of hardness theory, analysis of Three-Point Bending, and exploration of the tensile Behavior of composite materials offered a comprehensive understanding of their mechanical characteristics.

Moreover, attention was given to the environmental durability and fatigue behavior of composite materials, addressing crucial aspects for real-world applications. The literature review has successfully established the current state of knowledge in the field, shedding light on both the strengths and limitations of composite materials. As we move forward, the identified gaps and limitations underscore the rationale for the subsequent research objectives, aiming to contribute to the refinement and advancement of composite material technology.

Chapter II

Composites Production

Materials and Methods

This section delves deeply into the process of creating composite materials, covering various aspects from component selection to experimental procedures and mechanical testing. It begins by examining composite components, including epoxy resin as a matrix and various types of fiberglass as reinforcement materials. The chapter discusses the incorporation of additives like iron powder and details material processing methods. Experimental procedures for mechanical testing, statistical analysis, and investigating the impact of aggressive environments on composites are covered. Finally, a focused analysis of fatigue cyclic flexure concludes the chapter, providing a comprehensive understanding of composites production and their performance characteristics.

II.1. Introduction

This section of composite production represents a pivotal segment in our exploration of composite materials. This chapter unfolds the intricate journey of creating composite materials, meticulously navigating through the material and method employed in their production.

We commence with a thorough examination of composite components, dissecting matrix types with a special emphasis on the versatile epoxy resin. The discussion seamlessly transitions to reinforcement, exploring various fiber glass types that play a critical role in enhancing the composite's structural integrity. The inclusion of additives, particularly the nuanced analysis of iron powder, adds a layer of complexity to our understanding, scrutinizing granulometry through microscopic analysis, sieving, and purity assessments.

As we delve into material processing, the chapter unveils the diverse production methods employed in crafting these innovative materials. The experimental procedure section introduces us to the rigorous testing processes, including hardness tests, bending strength evaluations, and the application of statistical methods in mechanical testing, providing a robust foundation for material assessment.

Expanding our horizon, we investigate the impact of aggressive environments on our composite materials. The chapter unfolds experimental procedures for solution preparation, assessing submersion's influence on specimen weight, and delves into crucial aspects of ANOVA, microscopic, and FTIR analysis. Key considerations such as moisture absorption and flexure testing are explored, offering a comprehensive view of the material's behavior.

As we progress, the chapter concludes with a focused analysis of fatigue cyclic flexure, illuminating the material's endurance under prolonged stress. This chapter not only sets the stage for subsequent discussions but also establishes a solid framework for understanding the production intricacies, material characteristics, and performance assessments in diverse conditions. Join us on this journey into the heart of composite production, where innovation meets rigorous analysis.

II.2. Composite Components

II.2.1. Matrix Types

Before discussing the merits of specific resins, it's essential to understand the broader category of matrix materials, each offering unique characteristics and influencing the overall behavior of composites. Considering the broader matrix landscape, ceramic matrices, known for their excellent thermal and chemical resistance, find application in high-temperature environments. Silicon carbide and alumina matrices, for instance, are prevalent in aerospace and defense

applications where temperature resistance is critical. However, the inherent brittleness of ceramics necessitates careful consideration.

Metal matrices, including aluminum and titanium, offer advantages such as high thermal conductivity and excellent strength-to-weight ratios. Applied in industries like automotive and aerospace, MMCs balance structural integrity with thermal management. However, challenges arise in balancing these benefits with potential concerns related to weight and cost.

Polymer matrices, including both thermoplastics and thermosetting polymers, provide versatility in processing and application. Thermoplastics, characterized by reusability and ductility, find application in diverse fields, while thermosetting polymers contribute enhanced mechanical properties to composites requiring high-strength and dimensional stability.

II.2.2. Resin Options

Polyester Resin

Polyester resin, a widely-used thermosetting polymer, has gained popularity due to its affordability and ease of use. It is renowned for its versatility in applications such as boat construction, automotive parts, and general composites. However, its susceptibility to shrinkage during curing and relatively lower mechanical properties compared to other resins necessitate a careful evaluation of its suitability for specific composite formulations.

Vinylester Resin

Vinylester resin, an intermediate between polyester and epoxy resins, combines the cost-effectiveness of polyester with enhanced mechanical properties and chemical resistance. This resin type has found favor in applications requiring superior corrosion resistance, such as marine and chemical industries. However, consideration must be given to its cure time and potential issues related to storage stability.

Epoxy Resin

Epoxy resin, a standout within polymer matrices forms the foundational matrix for our glass/epoxy composite loaded with pure iron powder. Possessing a versatile chain-like molecular structure with reactive epoxide groups, epoxy resin undergoes crucial cross-linking during curing, resulting in a robust three-dimensional network. This imparts exceptional adhesion, bonding, and mechanical properties to the composite, including high tensile and flexural strength. The low shrinkage and dimensional stability of the cured epoxy contribute to the composite's structural integrity under diverse conditions. Additionally, epoxy resin exhibits notable thermal stability, making it suitable for applications requiring resistance across a range of temperatures. Its compatibility with both glass fiber reinforcement and iron powder filler ensures the homogeneity and durability of the composite structure. The review of existing literature underscores the

pivotal role of epoxy resin in composite materials, emphasizing its proven track record and comparative advantages. The chemical structure of epoxy resin is characterized by a chain-like backbone containing reactive epoxide groups, specifically oxirane rings. This structure features repeating units with a three-membered cyclic ring, where the epoxide groups serve as key reactive sites for cross-linking reactions during curing.

Epoxy resin was also chosen for its ubiquity and cost-effectiveness. Its low-density, typically an asset, poses intriguing challenges in this study. While low-density contributes to the overall lightweight nature of the composite, it prompts us to delve deeper into potential implications on structural integrity, an aspect unique to this investigation.

The resin epoxy used in this study is GENC VX100.00/VX101.00, an Ultra Transparent Glossy Casting Epoxy. This epoxy is an artisanal product available in the local market, chosen for its accessibility, cost-effectiveness, and notable properties such as excellent bonding strength, high chemical and physical resistance, ultra-transparency, and low-density varnish.

Features

- Excellent yellowing resistance
- High gloss level before and after polish application
- High scratch resistance on the surface may occur over time
- Minimum foam formation
- Easy sanding
- Excellent spreading and surface wetting
- Excellent adhesion strength after application
- High resistance to capillary cracking

Table II.1. Epoxy Specifications and Drying Time (45-65 % RH)

Specific Gravity (kg/l)	1,13 ± 0,03			
Solid Content (% by weight)	99-100			
Theoretical Coverage Area	1,00 - 1,10			
Polymerization Temperature	10-25 °C		25-40 °C	
Thickness	(2 cm)	(3 cm)	(2 cm)	(3 cm)
Stackable	6-7 day	6-7 day	5-6 day	5-6 day

Table II.2. Epoxy Hardener Mixing Ratio

Description	Code	by weight (%)	by volume (%)
Ultra-Transparent Glossy Casting Epoxy	VX101.00	100	100
Epoxy Hardener	HX100.00	33	40

II.2.3. Reinforcement

Fiber Glass Types

Before delving into the specifics of fiberglass mat E, it is imperative to recognize the diverse array of fibers available. The decision to utilize glass fibers is rooted in their widespread application and exceptional tensile strength. Different fiber types were scrutinized, and the choice of glass fibers aligns with the overarching goal of creating a composite with a balanced set of mechanical properties.

Fiber Diversity

The realm of fiber reinforcements is broad, encompassing aramid, carbon, and glass fibers, each with its unique characteristics. The selection of glass fibers, renowned for their cost-effectiveness and excellent tensile strength, was made with a holistic view of creating a composite that not only serves its purpose but does so efficiently and economically.

The fiberglass employed is EM 1002 type Chopped Strand Mat (CSM) manufactured by the renowned brand KROSGLOSS, boasting a surface weight of 300 g/m². This CSM is meticulously fabricated from E-glass fibers featuring a nominal filament diameter of 12 μm and a nominal length of 50 mm.

Features of the Glass Mat

- Good tensile strength,
- Easy handling,
- Easy impregnation,
- Low resin consumption,
- High mechanical properties of the laminate,
- Approved by:

Table II.3. Technical characteristics (nominal values).

	Test method	300
Surface weight(density) [g/m ²]	ISO 3374:2000	± 10%
Loss on ignition [%]	ISO 1887:1995	4,0÷5,5
Moisture content [%]	ISO 3344:2000	max.0,25
Wet-out time [min]	Internal instruction ZKJ-IO-11-009	max.6
Breaking force [N]	ISO 3342:2000	min.50

II.2.4. Additives

Beyond the foundational role of fiber reinforcements, the augmentation of composite materials is achieved through the incorporation of a diverse array of additives. These additives play a pivotal role in elevating the properties of composites, ushering in improvements in performance, durability, and overall functionality. Among this cohort of additives, powders emerge as particularly influential contributors, with metallic powders standing out for their distinct advantages in meticulously tailoring both the mechanical robustness and functional attributes of composite materials.

Impact Modifiers and Toughening Agents

These additives act as catalysts in enhancing resistance to impact and mitigating the propagation of cracks within composite structures.

Mechanical Enhancement: The inclusion of impact modifiers and toughening agents fortifies the composite against dynamic loading scenarios, endowing it with heightened resilience and durability, especially in applications where impact resistance is paramount.

UV Stabilizers and Inhibitors

Role: UV stabilizers and inhibitors are deployed to counteract the deleterious effects of ultraviolet radiation on composite materials.

Mechanical Enhancement: By safeguarding against UV-induced degradation, these additives extend the longevity of composites deployed in outdoor or exposed environments, ensuring sustained mechanical integrity over time.

Fire Retardants: Fire retardant additives are introduced to bolster the fire resistance of composite materials.

Mechanical Enhancement: Essential for applications prioritizing fire safety, these additives elevate the composite's capacity to withstand and repel combustion, thereby enhancing overall safety measures.

Powders as Additives in Composites:

The utilization of powder additives in composite matrices presents a versatile avenue for effecting functional enhancements. Notably, metallic powders emerge as a subset of powders offering distinctive advantages for tailoring both the mechanical and functional attributes of composite materials.

Metallic Powders in Composites:

Metallic powders, exemplified by iron powder, are strategically integrated to impart specific characteristics to composite materials.

Mechanical Enhancement: Iron powder, for instance, serves as a formidable reinforcing agent, contributing to amplified strength and hardness within the composite matrix. This versatile metallic additive can also introduce magnetic properties, broadening the range of applications in which the composite can be employed.

The infusion of metallic powders, such as iron powder, into composite matrices unfolds a realm of possibilities for engineers and materials scientists. This deliberate selection and incorporation of additives become instrumental in sculpting composite materials with tailored mechanical robustness, resilience to dynamic forces, and targeted functional characteristics. As a result, these fortified composites find application across diverse industries, ranging from automotive engineering to construction, where multifaceted performance requirements are met with precision and efficiency.

II.3. Iron Powder

Abundance in the Annaba Region

In this study, iron powder is not only included because of its natural properties, but also because it is carefully incorporated into the surrounding environment. The Annaba region has an abundance of iron powder because to a metallurgical complex. It is acquired by recycling underutilized materials. By carefully considering all options, this choice aligns with the goals of sustainable materials development and transforms a metal that was previously disregarded into a robust and useful composite material. By making use of easily accessible materials and the unexplored potential of iron powder, this work employs a sustainable approach to create strong and effective composite materials. This approach not only optimizes the utilization of nearby resources but also advances the broader notions of environmentally conscious and sustainable materials engineering.

II.3.1. Granulometry of Iron Powder

The granulometry of iron powder, denoting both the geometry and size distribution of its particles, plays a pivotal role in shaping the properties and behavior of composite materials. The geometry of iron powder particles can vary, exhibiting shapes such as spherical, irregular, or dendritic structures, a diversity influenced by the production methods employed. This morphological variability has significant implications for how the particles interact within the composite matrix. Simultaneously, the particle size distribution, categorized into fractions like fine, medium, and coarse, holds key insights into the reinforcement effect within the composite. The impact of granulometry extends to processing considerations, influencing mixing, dispersion, and, consequently, the uniform distribution of iron powder within the composite material.

In the regional context, particularly in the Annaba region, understanding the granulometry of locally sourced iron powder is paramount. The availability and characteristics of the powder are instrumental in its sustainable utilization, especially when repurposed from neglected resources within the metallurgical complex. This intentional choice aligns seamlessly with the principles of sustainable materials development, transforming an underutilized metal into a resilient and effective composite material. By harnessing the granulometry of iron powder, tailored adjustments can be made to influence the mechanical properties of the composite, including strength, stiffness, and resistance. This knowledge not only ensures effective processing and dispersion but also underscores the strategic and sustainable utilization of locally abundant resources, contributing to the development of resilient and efficient composite materials in the Annaba region.

II.3.2. Microscopic Analysis

Figure II.1 demonstrates how microscopic imaging offers useful insights into the granulometry of iron powder utilized in the composite. The photo displays a varied and random arrangement of iron powder grains, exhibiting a multitude of shapes such as irregular, spherical, and dendritic structures. The variation in geometry is a crucial factor in comprehending the interaction of iron powder within the composite matrix. In addition, the microscopic analysis reveals the presence of iron powder grains with different sizes, suggesting a non-uniform distribution of particle sizes. The heterogeneous distribution of grains enhances the overall intricacy of the composite material. The microscopic study provides visual documentation of the granulometry and offers essential insights for customizing the material's qualities depending on the specific characteristics seen in the iron powder grains. Microscopic imaging provides crucial insights into the detailed structure and behavior of composites at the microscale, which helps in making educated decisions for material design and engineering applications.

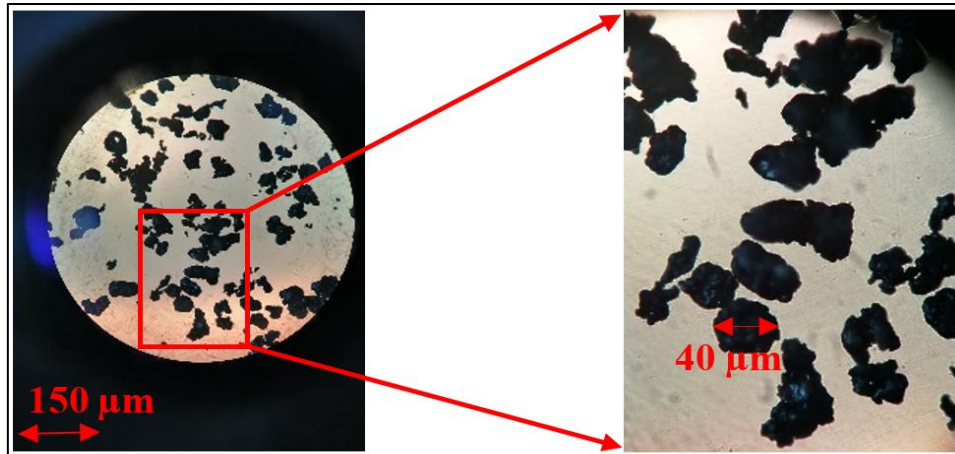


Figure II.1. Microscopic analysis of the pure iron

II.3.3. Sieving

When making iron powder for composite materials, sieving is an essential step, particularly if regulated granularity and well-defined size groups are the desired results. By passing iron powder through a sieve or mesh, this mechanical separation technique allows different-sized particles to be collected in different sections. Sieving is important because it can precisely categorize iron powder particles based on their sizes, resulting in a distribution that is well-defined and controlled. By arranging distinct groups of sizes, researchers and engineers may precisely manage a composite material's properties.

In the field of composite manufacturing, sieving allows for the intentional selection of particle sizes that can improve desired mechanical, thermal, or functional characteristics in the final product. This makes it possible to create a customized blend of iron powder fractions, increasing the composite material's overall effectiveness and strengthening effect. The importance of sieving is further highlighted by the need for consistency and homogeneity in the processing of materials. The application of a finely sifted iron powder ensures homogeneity within the composite matrix, minimizing distinctive variations and enhancing result consistency.

Furthermore, the process of sieving becomes especially important in situations when certain applications require a precise distribution of particle sizes. For example, in businesses that necessitate meticulous control over the mechanical characteristics of composite components, the process of sieving becomes an essential stage in preparing the materials. Sieving enables researchers to separate various size groups, allowing them to investigate a wide range of options and adjust the composition to match precise specifications.

The sieving procedure used at the Annaba research unit—more precisely, the "Mining and Metallurgy Research Unit (URMM)"—previously called "The Iron and Steel Applied Research Unit (URASM)"—is depicted in Figure II.2. Using sieves certified by DIN ISO 3310-1, the iron powder

was sorted by particle size, starting at 125 μm and going down to 64 μm , or the smallest possible size 32 μm , in steps of 92 μm .

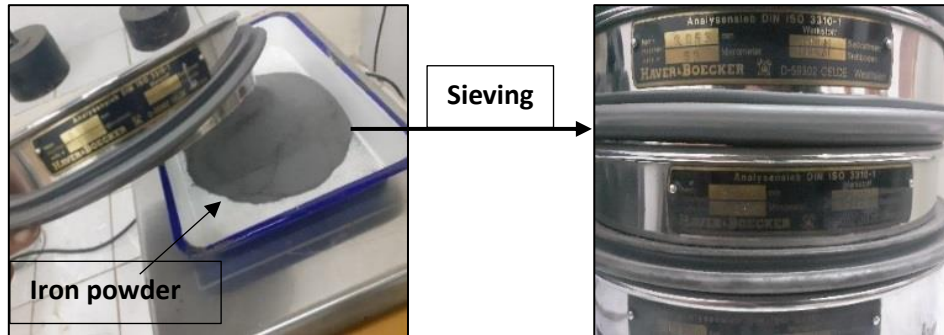


Figure II.2. Sieving operation at the URASM Annaba

II.3.4. Iron Powder Purity

Knowing how pure iron powder is, is essential to ensuring the dependability and effectiveness of composite materials. Because iron powder's purity directly affects its mechanical, thermal, and chemical properties, understanding it is essential for researchers and engineers. Impurities or compositional variations can significantly affect the final properties of the composite, which in turn affects the structural soundness and functional properties of the material.

DRX Analysis

X-ray diffraction (DRX) is a potent technique used to determine the purity of iron powder. This analytical technique is essential for determining and measuring the specific crystalline phases found in the powder, enabling a comprehensive evaluation of impurities and contaminants. Through the utilization of X-ray diffraction analysis, scientists may determine the crystalline arrangement of the iron powder, thereby detecting any supplementary phases or foreign components that may undermine its purity.

DRX (X-ray diffraction) offers exceptional precision in identifying the iron powder's composition by distinguishing various crystal forms and detecting even minute impurities. Accurate accuracy is especially important in industries that require strict quality control, such as aerospace or medical fields, where the purity of materials directly affects performance and safety.

The diffractogram for the iron powder was conducted using a Rigaku Ultima IV powder diffractometer (REF: Ultima IV), utilizing $K\alpha$ radiation sourced from a copper anticathode with a wavelength of 1.54060. The measurement involved an automatic point-to-point counting system, employing a step size of 0.0100 / $^\circ$ (2θ) and a duration of 10000 seconds, covering an angular range between 5 - 100 $^\circ$ (2θ) with generator parameters set at 0.

The X-ray diffraction pattern, depicted in Figure 3 and obtained at room temperature, reveals the purity of the iron powder (Fe) with impurities measuring less than 0.0013%. The material exhibits well-crystallized characteristics, and its indexing in the cubic system (space group Im3m) aligns with the reference: 00-006-0696.

The refinement of mesh parameters yields the following results: $a = b = c = 2.8664$; $\alpha = \beta = \gamma = 90^\circ$; $V = 23.55 \times 10^6 \text{ pm}^3$.

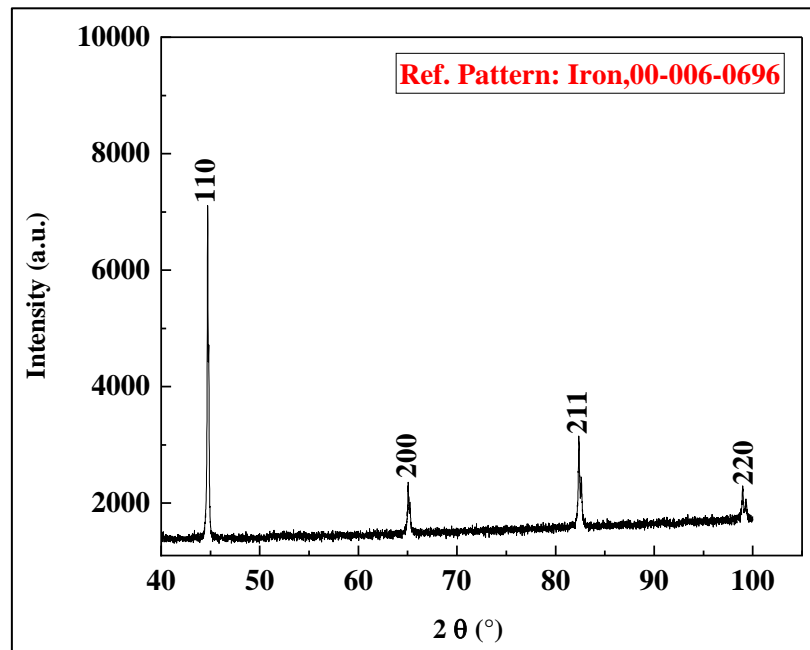


Figure II.3. X-ray diffraction of Iron (Fe) at room temperature (25 °C).

II.4. Material Processing

Laminated composites are made using a range of procedures to provide materials with unique properties suitable for a range of applications. Each technique has unique advantages that meet specific needs for costs, volume of production, and complexity. Because of its adaptability and compatibility with the study's objectives, the "moulage au contact" method, also referred to as contact molding—was selected from among these techniques for this investigation. With this process, reinforcing elements, like fibers or textiles, are inserted into a mold right away, and resin is put directly onto the mold surface. Because of its affordability and ease of use, contact molding is a very beneficial solution, particularly for large-scale production and the creation of complex part shapes.

While there are benefits to manual lay-up, spray-up, vacuum bagging, autoclave molding, and RTM, contact molding was selected due to its practicality and ability to adapt to the particular requirements of the research. Contact molding's flexibility is especially important when incorporating locally sourced iron powder into the composite matrix. The flexibility required to

incorporate iron powder, control its particle size, and achieve the desired distribution of particle sizes inside the laminated structure is provided by this production method. In addition, the contact molding method provides efficacy and practicality in attaining the targeted properties of composite materials in studies. It facilitates the smooth investigation of locally accessible resources, encouraging creativity and sustainability in combined solutions.

II.5. Production Method

The production of laminated composite plates required a careful combination of epoxy resin and eight layers of E-glass fiber mat, constituting 30% of the overall weight per plate. Concurrently, iron powder fillers of different sizes (92 μ m, 64 μ m, and 32 μ m) and concentrations (15%, 20%, and 25%) were added. During the initial manufacturing process, the epoxy resin matrix was prepared with precision, following a specified weight percent ratio of 100 parts resin to 33 parts hardener. The precise combination of ingredients was crucial in achieving a harmonious and efficient curing process.

The epoxy resin, hardener, and iron powder were completely mixed with an agitator to create a homogenous mixture after the resin matrix was prepared. The careful use of this phase aimed to produce a consistent distribution of parts, resulting in a unified and seamlessly combined framework. The principal objective was to furnish a robust and long-lasting foundation for subsequent stages in the production of composite plates.

The hand lay-up molding technique entails the sequential placement of individual layers of reinforcing glass fiber in an exposed mold, followed by the infusion of resin. In order to reduce porosity, an aluminum roller specifically designed for removing air was used, followed by a vacuum process to eradicate any remaining air bubbles. The polymerization process lasted for a duration of seven days, in accordance with the epoxy manufacturer's prescribed recommendations. Figure II.4 illustrates the successive procedures involved in producing these composite plates, highlighting the meticulous precision and careful attention to detail employed during the manufacturing process.

It was essential to maintain a consistent room temperature of 24 to 26 degrees Celsius during the whole manufacturing process of the laminated composite plates, starting from the preparation of epoxy resin and continuing through the seven-day polymerization period. Precise temperature control emphasizes the dedication to guaranteeing the excellence and dependability of the end composite product.

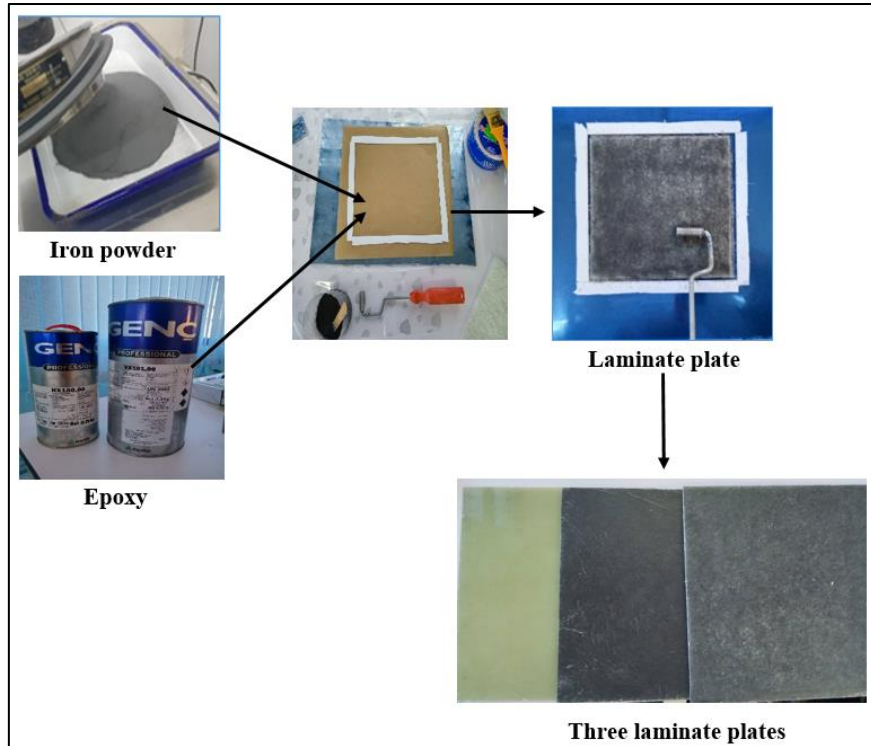


Figure II.4. Plate preparation by contact molding

II.6. Experimental Procedure in Normal Conditions

II.6.1. Hardness test

The hardness test is a common method used to assess the resistance of a material to localized deformation or indentation. It provides valuable information about a material's mechanical properties, especially its ability to withstand wear, abrasion, and deformation. There are various hardness testing methods, each suitable for specific applications. Three widely-used methods are Brinell Hardness, Vickers hardness, and Rockwell hardness.

Brinell Hardness Test

In the Brinell Hardness test, a hardened steel ball or tungsten carbide ball is pressed into the surface of the material being tested under a known load.

The resulting indentation diameter is measured, and the Brinell hardness number (BHN) is calculated using a standardized formula.

This method is often employed for materials with coarse microstructures or rough surfaces.

Vickers Hardness Test

The Vickers hardness test involves indenting the material with a square-based diamond pyramid under a specific load.

The length of the diagonals of the resulting indentation is measured, and the Vickers hardness number (VHN) is calculated.

This method is widely used for measuring the hardness of metals, ceramics, and some polymers.

Rockwell Hardness Test

Rockwell hardness testing measures the depth of penetration of an indenter into the material under varying loads.

The Rockwell hardness number is determined based on the difference in depth between the initial penetration and the final penetration under the total load.

Different scales (A, B, C) are used depending on the material's hardness characteristics.

II.6.1.1. Importance and Applications

Material Characterization: Hardness testing provides a quantitative measure of a material's resistance to deformation, helping to characterize its mechanical properties.

Quality Control: Industries use hardness testing as part of quality control processes to ensure that materials meet specified hardness requirements.

Material Selection: Engineers and designers use hardness data to select materials suitable for specific applications, such as choosing wear-resistant materials for cutting tools or high-strength materials for structural components.

Heat Treatment Monitoring: Hardness testing is often employed to monitor the effectiveness of heat treatment processes in altering the mechanical properties of materials.

The evaluation of a component's surface hardness, which expresses its resistance to deformation, becomes a critical factor influencing tribological properties and overall performance [60] [61]. Within the scope of this study, a comprehensive examination was conducted using the Brinell Hardness (HB) test. This method quantifies hardness by measuring the pressure applied to the pressmark per unit area, providing valuable insights into the material's resistance to deformation [62].

II.6.1.2. Application of Brinell Hardness Test in the Study

The mechanical properties of the composite materials were evaluated using the Brinell Hardness test in this study. The approach entailed methodically altering the iron powder composition and grain size, with the aim of comprehending the influence of these variables on the hardness of the composite material.

The selection of the Brinell Hardness test was driven by its appropriateness for heterogeneous materials, which corresponds to the intricate characteristics of composite constructions. The outcomes derived from this approach provide a significant understanding of the mechanical properties of the composite material under various circumstances, assisting in the analysis of its characteristics and the enhancement of its performance.

This approach is consistent with the overarching goal of enhancing our comprehension of composite materials and their suitability in various technical applications.

The Brinell Hardness assessment was conducted with great attention to detail, following the ISO 2039-1 standard specification. The assessment utilized the advanced "Innovatest HT16/17" apparatus, which is equipped with a powerful microscope and can apply loads ranging from 1 to 3000kg.

II.6.1.3. Experience methodology

The test specimens scrutinized were plates fabricated from a composite blend comprising 15% pure iron powder and fiberglass laminate, exhibiting an average thickness of 4mm. Noteworthy, the iron powder grains differed in size across the plates, measuring 92 μ m, 64 μ m, and 32 μ m, respectively.

For the examination, a 5 mm diameter steel ball was pressed into the surface of the plates under a substantial load of 25kN. Multiple measurements, a minimum of 5, were taken at various points across each plate, as illustrated in Figure II.5.

To provide a comprehensive comparative analysis, identical testing procedures were applied to assess the hardness of both an epoxy resin plate and a laminate epoxy reinforced with a 45% E-glass fiber mat. The ensuing analysis of the test data was meticulously carried out using the advanced "Impression" software.

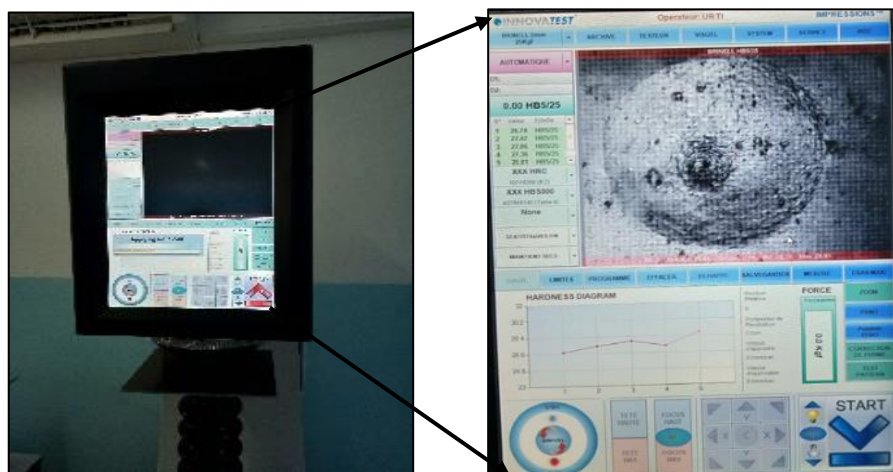


Figure II.5. Hardness measurement procedure using Innovatest HT16/17.

II.6.2. Bending strength

The ability of composites or materials to endure bending deflection when subjected to applied energy within the structure is known as flexural strength. [63].

The three-point bending test serves as a fundamental evaluation of the mechanical characteristics of composite materials, with a particular focus on flexure strength and Young's modulus. In the

comparison specimen, composed of 45% fiberglass E and 55% epoxy without any iron powder, the three-point bending test provides valuable insights into the material's intrinsic ability to resist bending forces and maintain structural integrity. A higher flexure strength signifies enhanced resistance to deformation and fracture, while Young's modulus reflects the material's stiffness and elastic behavior.

In contrast, specimens filled with pure iron powder, as per the specifications outlined in an L9 (3 × 3) Taguchi orthogonal array (which is explained below), undergo the same three-point bending test to assess the impact of iron powder inclusion on mechanical properties. Variations in flexure strength and Young's modulus in comparison to the reference specimen elucidate the reinforcing or modifying effects of iron particles. These results offer valuable insights into how the addition of iron powder influences the composite material's bending resistance, stiffness, and overall structural behavior.

The comparison between the two sets of specimens, one without iron powder and the other incorporating pure iron powder forms a critical analysis of the material's response to external forces. This comprehensive examination contributes to a nuanced understanding of the role played by iron particles in altering the mechanical properties of the composite. By leveraging the L9 Taguchi orthogonal array, this study not only provides quantitative data on flexure strength and Young's modulus but also sheds light on the intricate interplay between material composition and mechanical performance in the specific context of three-point bending.

II.6.3. Statistical Methods in Mechanical Testing

Statistical methods play a pivotal role in the analysis of data derived from mechanical tests, offering valuable insights into material behavior and performance. Two widely-used statistical techniques in this context are ANOVA and regression.

II.6.3.1. ANOVA

ANOVA is a powerful tool for comparing means among different groups. In mechanical testing, ANOVA can assess the impact of various factors, such as material type or testing conditions, on the observed mechanical properties. While its importance is acknowledged, a more detailed exploration of ANOVA will follow.

II.6.3.2. Regression

Regression analysis helps model the relationships between variables, allowing for a quantitative understanding of how changes in one variable influence another. In mechanical testing, regression can be employed to predict mechanical properties based on experimental conditions.

II.6.3.3. Taguchi Method

The Taguchi method, developed by Genichi Taguchi, is an approach to the Design of Experiments (DOE) that focuses on optimizing processes while considering variability. It is particularly relevant in mechanical testing, where the goal is to identify optimal test conditions for enhanced material performance.

Key Aspects of the Taguchi Method in Mechanical Testing:

Orthogonal Arrays: Taguchi employs orthogonal arrays to systematically vary input factors in a controlled manner, allowing for the study of their combined effects with a minimal number of experiments. This efficiency is crucial in mechanical testing where resource optimization is essential.

- The Taguchi method uses the S/N ratio to evaluate the impact of factors on the performance output. The objective is to maximize the S/N ratio, indicating robust performance in the presence of variability.
- Taguchi emphasizes parameter design to make processes less sensitive to variations and provides a systematic approach for tolerance analysis. This is particularly valuable in mechanical testing where material properties may vary.
- The Taguchi method facilitates robustness testing, helping identify optimal conditions that minimize the effects of external variations, ensuring consistent and reliable results in mechanical testing.

In summary, the Taguchi method offers a systematic and efficient approach to optimizing experimental conditions in mechanical testing, promoting robustness and reliability in the face of variability. This method will be further clarified in subsequent discussions.

Enhancing quality, reliability, and optimizing performance can be achieved through practical methods, and one such effective approach involves the application of statistical techniques known as DOE. Dr. Genichi Taguchi's standardized version of DOE stands out as the optimal choice for experimental application and result analysis, encompassing the exploration of all conceivable conditions involving multiple factors [64].

In this study, orthogonal arrays were employed to determine the number of specimens needed in the flexural testing. The investigation involved varying the iron powder contents (15%, 20%, 25%) and the powder grain sizes (92 micrometers, 64 micrometers, and 32 micrometers). The use of orthogonal arrays facilitated a systematic and efficient exploration of the combined effects of these factors, optimizing the experimental design for flexural testing. This approach not only allowed for a comprehensive assessment of the influence of iron powder content and grain size

on the flexural properties but also ensured resource optimization by minimizing the number of specimens required for a thorough analysis.

Within the context of this study, three factors influencing Flexural Strength (FS) and Young's Modulus (E) were taken into consideration, with their respective parameters and levels detailed in Table 2. Utilizing Taguchi's orthogonal array L9 (3x3), a total of 9 experiments and combinations of variables were derived, as illustrated in Table 3. To ascertain the optimal configuration for achieving the highest FS and Young's modulus, these parameters were treated as responses in Taguchi's design analysis.

The experimental data underwent a transformation into a signal-to-noise ratio (S/N) for analysis, adopting the assumption that a larger value indicates a better outcome (LB). This transformation is accounted for in each combination of factor levels through a logarithmic transformation of the loss function using the equation below:

$$\eta = -10 \log_{10} \left[\frac{1}{n} \sum_{i=1}^n \frac{1}{Y_i^2} \right]$$

Where η is the signal-to-noise ratio (dB), n is the number of observations under the same experimental conditions, and Y is the response value obtained in the test (output).

Table II.4. Taguchi factors and runs.

Taguchi Array	L9 (3x3)
Factors	3
Runs	9

Table II.5. Factors and levels used in Taguchi's method

Factor	Symbol	Level 1	Level 2	Level 3
Content (%)	A	15	20	25
Size (µm)	B	92	64	32
Velocity (mm/min)	C	1	5	10

The dimensions of the bending specimens, adhere to the specifications outlined in the ISO 14125 standard. A CROWN cutting machine, boasting an 1100 W power capacity and equipped with a 250mm diameter diamond disc, was employed for precision cutting. To aid in the cutting process and maintain optimal conditions, a lubrication system was utilized to cool the specimens. The average dimensions of the specimens are detailed in Table II.6



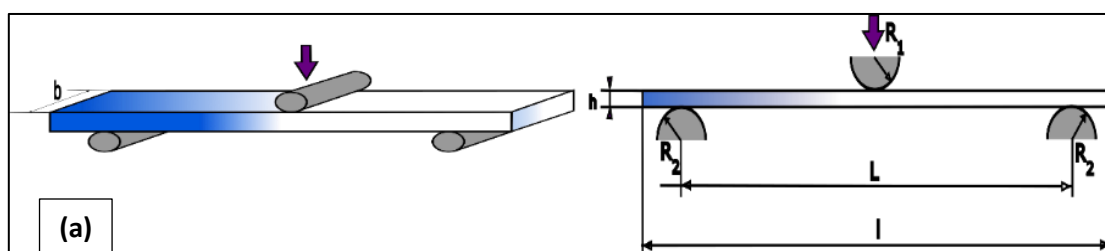
Figure II.6. Bending specimen's preparation.

(a) plates cutting; (b) and (c) bending test samples;

Table II.6. Specimen mean dimensions.

Specimen length (l) mm	Outer span (L) mm	Width (b) mm	Thickness (h) mm
80	64	14.64	3.83

The bending tests were conducted at the facilities of the "Mining and Metallurgy Research Unit, formerly URMA," within the "Research Center in Industrial Technologies" (URMM/CRTI) in Annaba. A universal machine, specifically the MTS 43 was utilized for the experiments, which is a mechanical testing machine, equipped with a force sensor and computer control, offering force capacities ranging from 1 to 600 kN (220 to 132,000 lbs), featuring high-speed, low-vibration electromechanical drives and integrated closed-loop digital controls for greater fidelity during testing. These reliable and user-friendly systems deliver accurate results and support a wide range of specimen types with forces from 600 kN to as low as 1 N. Key features include a 5,000 Hz data acquisition rate for detailed material analysis, compact AC servomotors for high-speed, low-vibration testing, TEDS-compatible force sensors for automatic calibration data upload, and TestSuite TW software with customizable test templates for managing complex testing. Each run involved a minimum of six (6) tests, following the specifications outlined in an L9 (3 × 3) Taguchi orthogonal array, and were carried out at an ambient temperature of 24°C. The load cell, functioning as a dynamometer, was connected to an acquisition chain that facilitated the simultaneous recording of displacement time, load, and deformation. Data collection and result processing were conducted using computer-based MTS TestSuite software.



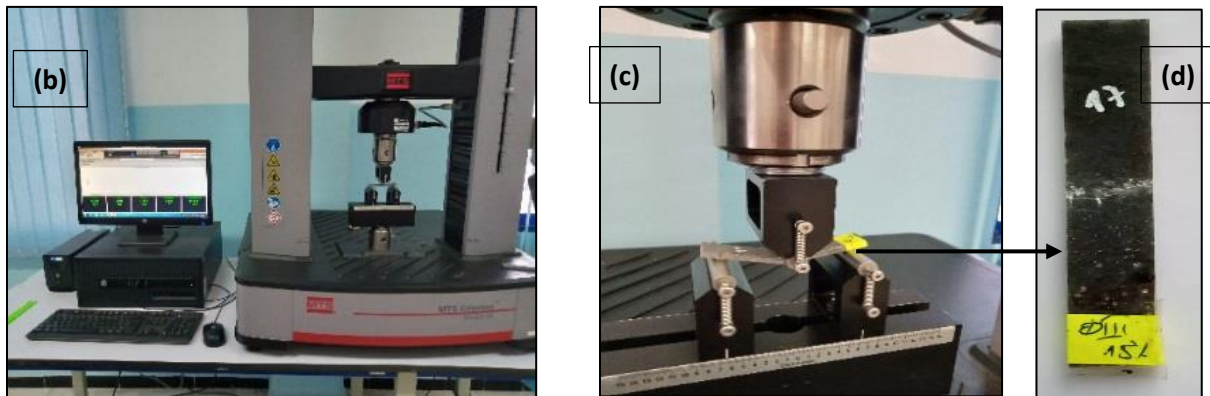


Figure II.7. Bending test experimental procedure.

(a) three-point bending scheme; (b) photography of experimental test on MTS 43 universal machine, (c) loading force, (d) damaged specimens after flexure test

The results obtained were juxtaposed with those of glass epoxy specimens, which were designed similarly and subjected to three-point bending at a rate of 1mm/min.

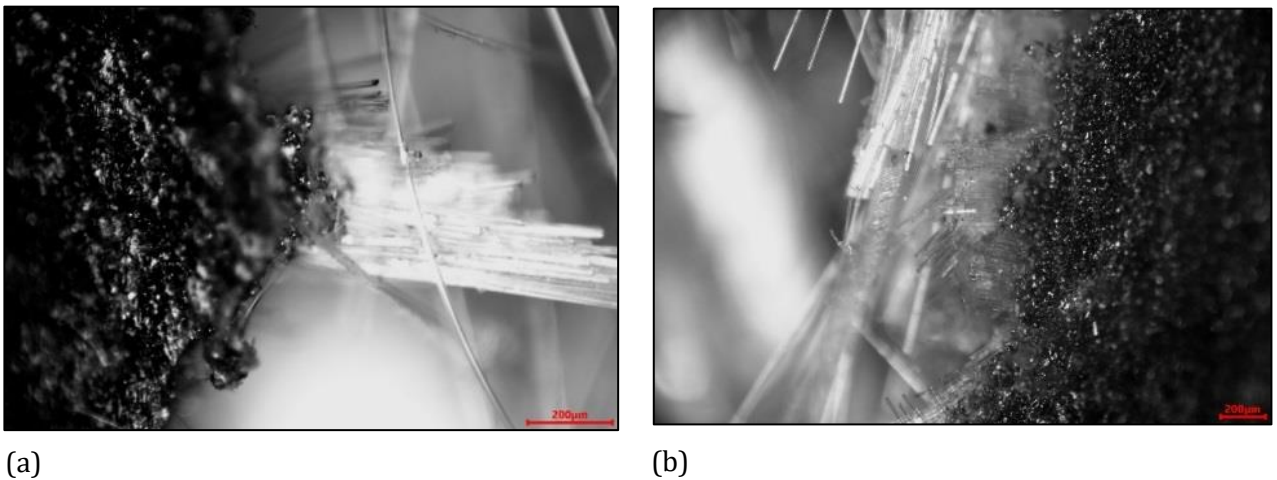


Figure II.8. Microscopic image of the specimen's rupture zone (a) left (b) right

II.6.4. Tensile tests

The tensile test is a fundamental and widely employed mechanical testing method used to assess the mechanical properties of materials, particularly their response to axial loading. This test provides crucial insights into a material's strength, ductility, and other key mechanical characteristics. In this study, the tensile test was conducted under ambient room temperature conditions (25°C) to evaluate the material's behavior in a typical environmental setting.

The testing procedure was executed with a controlled testing speed of 1 mm/min, ensuring a consistent and standardized approach. This controlled speed allows for a systematic evaluation of the material's response to increasing tensile forces, enabling the characterization of its mechanical performance.

The specimens used in the tensile test were precision-cut using a computer numerical control (CNC) machine equipped with a diamond-disc cutting tool. This advanced cutting method ensures accurate and reproducible specimen dimensions, contributing to the reliability and precision of the test results. The use of a diamond-disc cutting tool adds an additional level of precision, minimizing any potential alterations to the material's structure during the specimen preparation process.

Conforming to the ISO 527-2 standard, the dimensions of the specimens were carefully adhered to, ensuring alignment with internationally recognized testing standards. Standardizing specimen dimensions is critical for enabling accurate comparisons and facilitating the interpretation of results within a broader scientific and engineering context.

This tensile test, conducted under carefully controlled conditions and following established standards, serves as a foundational component for evaluating the mechanical behavior of the material under investigation. The systematic approach, from specimen preparation to testing parameters, enhances the reliability and significance of the results, providing valuable insights for material characterization and potential applications.

The tensile tests were conducted using the Zwick Z050 electromechanical fatigue testing machine, designed for mechanical characterization of materials and structures under quasi-static tensile/compressive loading conditions. The key technical specifications of this machine include a loading capacity of 0.5 kN, 5 kN, or 50 kN, with a piston displacement of 1520 mm. The acquisition frequency is set at 150 Hz, and the system allows for servo-controlled loading based on either force or displacement. The machine utilizes mechanical flat grips (1 kN, 2.5 kN, 10 kN, and 50 kN) for specimen fixation during testing, at least 5 specimens were tests for each run.

The primary objective of this tensile test is to investigate potential variations in tensile strength and Young's modulus in composites loaded with pure iron powder. Specifically, the study aims to discern if the addition of pure iron powder, varied in weight percentage, plays a significant role in influencing the mechanical behavior of the composites. This exploration is motivated by the need to understand the impact of pure iron powder on the overall tensile properties of the composite material, with a focus on elucidating any evolution or trends in tensile strength and Young's modulus.

By systematically altering the weight percent of added iron powder, we seek to establish correlations between these variations and the observed changes in mechanical properties. This endeavor contributes not only to a fundamental understanding of composite material behavior but also holds practical implications for optimizing material formulations with enhanced tensile performance.

The standard force in tension relative to stress refers to the relationship between the force applied to a material during a tensile test and the resulting induced stress in the material. Stress is defined as force per unit area and is expressed in Pascals (Pa) in the International System of Units (SI).

Hooke's Law is often used to describe this relationship in the elastic range of a material. In this range, stress (σ) is directly proportional to the applied force (F) and the initial cross-sectional area of the specimen (A). The mathematical formula associated with this relationship is as follows:

$$\sigma = F/A$$

Where:

σ represents stress,

F represents the applied force, and

A represents the initial cross-sectional area of the specimen.

II.7. Experimental Procedure in Aggressive Environment

When studying composites in extreme settings, it is essential to follow a systematic experimental strategy to thoroughly evaluate how the material reacts to difficult conditions. The procedure commences by meticulously choosing composite materials, taking into account variables like as reinforcing fibers, matrix composition, and any protective coatings or additives. Subsequently, standardized methods are followed to prepare uniform specimens, taking into consideration the specific circumstances of the harsh environment. The experimental setup sets precise and regulated circumstances for testing, which include determining the period of exposure and closely monitoring environmental factors. Regular evaluations are carried out for the duration of the exposure, encompassing visual inspections, testing of material properties, microscopic observations, and chemical analysis. A thorough collection and analysis of data, encompassing mechanical properties and microstructural changes, is conducted in a systematic manner. This process involves reaching relevant findings by utilizing statistical assessments. Post-exposure assessments offer valuable insights into the degradation of the composite material and its overall impact following exposure to harsh conditions. Correlating experimental findings with real-world settings improves comprehension of the material's performance and durability in practical applications. This methodical experimental protocol guarantees a comprehensive examination of

composites in hostile environments, providing significant observations for the selection of materials, optimization of design, and evaluation of durability in demanding operational conditions.

Modifications to the approach might be implemented to correspond with the particular aggressive elements being examined and the primary objectives of the study.

The chosen environment in this study closely mirrors the conditions seen in the Annaba region, where industrial pollution is present in both seawater and wastewater. As a result, there is a significantly low pH level, indicating acidity. Moreover, the coastal location of Annaba underscores the importance of investigating the efficacy of materials when immersed in ocean or sea water. This study is especially pertinent for comprehending the processes of deterioration and long-lasting quality of materials in challenging environments, owing to the unique amalgamation of industrial pollutants and coastal circumstances. The prevalence of acidic water conditions, primarily resulting from industrial pollutants, underscores the need for a comprehensive examination of the materials employed in such environments. The close proximity to the ocean amplifies the potential impacts of saltwater exposure on the materials under study, emphasizing the imperative of performing this examination for its relevance to marine and coastal engineering applications. The results of this study will not only improve our understanding of material behavior in the particular environmental conditions of Annaba, but also have important implications for other coastal regions worldwide grappling with the challenges posed by industrial waste and sea exposure.

II.7.1. Solution preparation

In preparation for our experimental investigations, two distinct media formulations were meticulously crafted. The first medium involved the utilization of seawater sourced from a coastal area in the Annaba region. Following the meticulous collection process, the specimens underwent a rigorous sterilization procedure, ensuring the elimination of any potential contaminants. Subsequently, they were carefully dried within specialized ovens to maintain optimal conditions. The second medium, illustrated in Figure II.9, was prepared by diluting pure hydrochloric acid (HCl) with distilled water until it reached a targeted pH value of 2. Similar to the initial medium, this subsequent solution underwent a comprehensive sterilization process and subsequent drying within dedicated ovens.

These intricate preparations were undertaken with the specific aim of facilitating the submersion of the meticulously prepared test specimens in our forthcoming experiments. By adhering to these rigorous protocols, we aim to ensure the integrity and reliability of the experimental conditions, thereby contributing to the robustness of our research outcomes.

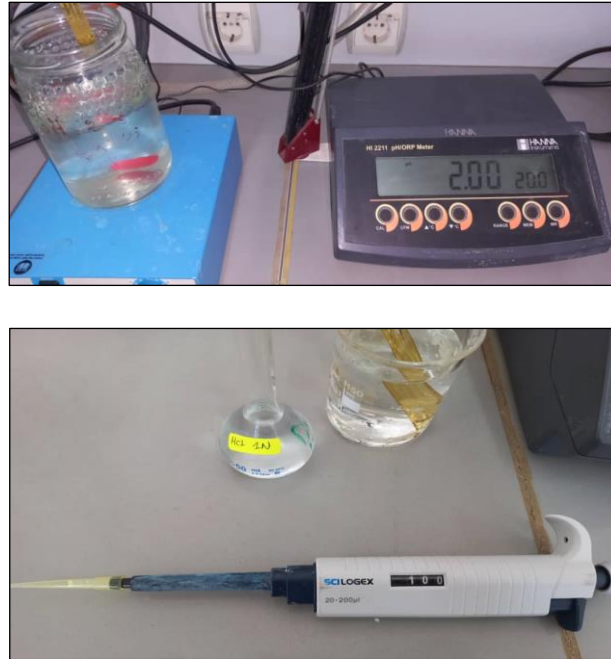


Figure II.9. Acidic Solution preparation

The specimens were categorized into three distinct groups, as illustrated in Table II.7, identified as group A, group B, and group C. Within group A, three sub-groups were subjected to exposure to seawater, while the three sub-groups in group B underwent immersion in an acidic solution with a pH value of 2. The remaining sub-groups in both group A and group B were kept unsubmerged for comparative analysis.

Table II.7. Specimen's grouping

Group	Sub-groups	Iron content (W%)	Solution
A	A-1	15	Seawater
	A-2	20	Seawater
	A-3	25	Seawater
B	B-1	15	Acid
	B-2	20	Acid
	B-3	25	Acid
C	C-1	15	/
	C-2	20	/
	C-3	25	/

II.7.2. Submersion's Impact on Specimen Weight

The dry, non-immersed, and immersed specimens were initially weighed to determine their initial weight (W_0). Subsequently, these specimens were submerged in two different solutions for a minimum of 1440 hours. At specific time intervals, the samples were extracted, and their individual weights (W_i) were measured using an analytical balance with a resolution of 0.0001g. The recorded weight measurements are comprehensively presented in Table 3. In this table:

- **T0 (Time Zero):** Refers to the initial time when the specimens are submerged or subjected to the experimental conditions.
- **T1 to T6:** These time points represent specific intervals during the experiment, indicating when measurements or observations were taken. For instance, T1 might signify measurements taken after 10 days, T2 after 20 days, and so forth. Each subsequent time point (T3, T4, T5, and T6) would similarly denote additional intervals or milestones in the experiment, helping to track changes over time.



Figure II.10. Weight measurement operation

Table II.8. Specimen's weight measurement detailed

Group	Sub-group	T0	T1	T2	T3	T4	T5	T6
A	1	7.6049	7.6070	7.6085	7.6098	7.6123	7.6246	7.6655
	2	7.957	7.9608	7.9620	7.9636	7.9649	7.9755	7.9989
	3	8.4760	8.4807	8.4835	8.4837	8.4865	8.4964	8.5227

B	4	7.5305	7.5275	7.5244	7.5253	7.5175	7.513933	7.5152
	5	7.8348	7.8326	7.8303	7.8301	7.8231	7.8163	7.8026
	6	8.8055	8.8005	8.7974	8.7889	8.7817	8.7808	8.7616

II.7.2.1. Moisture absorption

Measuring moisture absorption of the studied specimens has been done using the simple formula as follows: Moisture absorption ability in % = $(W_i - W_0)/W_0$

Where:

W_0 (g) is the initial weight of samples;

W_i (g) is the weight of samples after uptaking moisture.

II.7.3. Flexure testing

The 3-points flexure testing schematized in Figure 4, was conducted at the “Mining and Metallurgy Research Unit”, formerly known as “URMA”, which is a part of the “Research Center in Industrial Technologies” (URMM/CRTI) located in Annaba (Algeria). The MTS 43, a universal machine, is outfitted with a force sensor and is under the control of a computer. A minimum of five (5) tests were applied for each sub-group, using specimens mean dimensions displayed in Table II.9 In this case, speed and temperature were 10 mm/min and 25 °C, respectively. The load cell illustrated in Figure 5, also known as a dynamometer, is linked to an acquisition chain that enables the concurrent measurement of displacement time, load, and deformation. The data collection and result processing were conducted using computer technology, specifically the MTS TestSuite software. A total of 6 experiments and combinations of variables were conducted, in order to measure the FS and Young’s modulus.

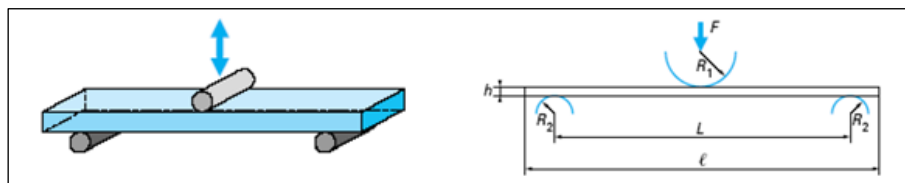


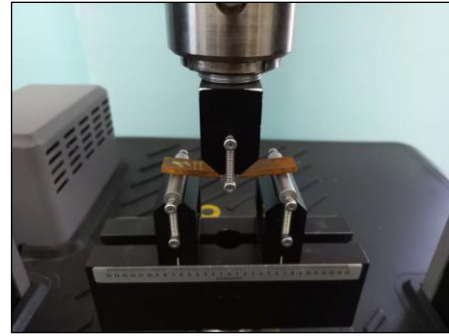
Figure II.11. Three points bending scheme

Table II.9. Specimen mean dimensions

Length (l)	Outer span (L)	Width (b)	Thickness (h)
mm	mm	mm	mm
80	64	14	4



(a)



(b)

Figure II.12. Bending test procedure (a) and (b)

II.7.4. ANOVA in Material Studies

The ANOVA is a robust statistical tool used to evaluate the mechanical properties of composite materials. The implementation of this method is especially relevant in the field of composite production, where multiple factors might impact the ultimate mechanical properties of the material. ANOVA is a robust analytical framework that enables researchers to identify the origins of variability in mechanical parameters, providing a more profound comprehension of how various elements contribute to the observed differences.

ANOVA is utilized in the analysis of composite materials to examine the influence of many factors, including material composition, manufacturing conditions, and the addition of substances such as iron powder. ANOVA allows researchers to assess the statistical significance of reported changes in mechanical parameters by carefully analyzing the variability within and between various groups or circumstances. It is essential to determine the primary components that have a substantial impact on the composite's performance.

ANOVA is highly versatile as it can effectively manage numerous factors at the same time, making it ideal for complicated experimental designs commonly found in composite research. ANOVA is useful for identifying the elements that have a statistically significant impact on mechanical properties such as tensile strength, flexural modulus, and impact resistance. This statistical method serves the purpose of both validating the success of various manufacturing strategies and providing guidance for optimizing composite formulas to satisfy specified performance requirements.

Moreover, ANOVA enables the detection of interactions among factors, providing insight into how the collective impact of variables may deviate from their impacts. Having a nuanced understanding is crucial for customizing composite materials accurately, guaranteeing that the mechanical properties match the desired uses.

II.7.4.1. Key Aspects of ANOVA

Comparison of Means

ANOVA is designed to compare the means of three or more groups to determine if there are statistically significant differences among them.

In material studies, ANOVA can be applied to compare the mean values of material properties across various experimental conditions, treatments, or formulations.

Factorial Experiments

ANOVA is particularly useful in factorial experiments where multiple factors are manipulated simultaneously.

For example, in a material study, ANOVA might be applied when varying factors like temperature, pressure, or composition to observe their combined effects on material properties.

Identification of Sources of Variation

ANOVA helps identify sources of variation within a dataset, allowing researchers to understand which factors contribute significantly to observed differences.

In material studies, this aids in pinpointing influential variables affecting material performance.

Partitioning Variance

ANOVA partitions the total variance in the data into components associated with different factors and their interactions.

This breakdown assists in quantifying the relative impact of each factor on the observed variations in material properties.

F-Test and Significance Levels

ANOVA employs the F-test to determine whether the observed differences among group means are statistically significant.

Significance levels (often denoted by alpha, α) are chosen to assess whether the p-value is below a predetermined threshold (e.g., 0.05), indicating statistical significance.

II.7.4.2. Applications in Material Science

Material Characterization

ANOVA aids in comparing the means of material properties such as hardness, tensile strength, or conductivity under different conditions or treatments.

Quality Control

ANOVA is utilized in quality control processes to assess whether variations in material properties are within acceptable limits.

Experimental Design

Researchers use ANOVA to analyze experimental designs involving multiple factors, helping to optimize conditions for desired material outcomes.

Optimization Studies

In studies aiming to optimize material formulations or processing parameters, ANOVA guides the identification of influential factors.

II.7.5. Microscopic and FTIR Analysis

Various methods are employed to observe microfibrils, surface topography, and rupture areas in material studies, each offering unique advantages and capabilities.

Scanning Electron Microscopy: SEM provides high-resolution images by scanning a sample's surface with a focused electron beam. It offers detailed observations of microfibrils, surface features, and fracture surfaces at magnifications beyond the capability of light microscopy.

Transmission Electron Microscopy (TEM): TEM involves transmitting electrons through an ultra-thin sample, enabling nanoscale observations of microfibrils and internal structures. It is particularly powerful for detailed examinations of composite materials at the atomic and molecular levels.

Atomic Force Microscopy (AFM): AFM utilizes a sharp tip to scan the sample's surface, providing topographical information at the nanoscale. It is highly effective in characterizing surface roughness, measuring forces, and mapping material features with high-resolution.

Fractography Techniques: Techniques such as fractography involve the detailed analysis of fracture surfaces using scanning techniques or optical microscopes. These methods aid in understanding the mechanisms of material failure, including crack initiation and propagation.

Confocal Microscopy: Confocal microscopy utilizes a focused laser beam to create optical sections of a sample, allowing three-dimensional imaging. It is valuable for studying surface topography and microfibril arrangements in transparent or fluorescently labeled materials.

Interference Microscopy: Interference microscopy measures variations in optical path length to visualize surface topography with high precision. It is often used for non-destructive and quantitative assessments of materials.

Infrared Microscopy: Infrared microscopy analyzes the absorption or reflection of infrared light by materials, providing information about composition and molecular structures. It is particularly useful for studying the chemical composition of microfibrils and surfaces.

Each of these methods contributes to a comprehensive understanding of material properties, offering varying levels of resolution, depth, and specificity. The choice of technique depends on the research objectives, the scale of features under investigation, and the nature of the material being studied. Combining multiple methods often provides a more thorough and nuanced characterization of microfibrils, surface topography, and rupture areas in material studies.

Light microscopy, despite its limitations in achieving nanoscale resolution, holds significant relevance in certain material studies, including the examination of microfibrils, surface topography, and rupture areas. Several factors contribute to the rationale for using light microscopy in specific investigations:

Accessibility and Cost-Effectiveness: Light microscopes are widely available and more cost-effective compared to advanced microscopy techniques. This accessibility makes them suitable for routine observations, preliminary examinations, and educational purposes.

Macroscopic Observations: Light microscopy provides a macroscopic view of the sample, allowing researchers to observe and document general features such as the alignment and distribution of microfibrils, overall surface texture, and the extent of rupture areas.

Speed and Efficiency: Light microscopy offers rapid and real-time observations. Researchers can quickly scan large sample areas and obtain immediate visual feedback, facilitating a swift overview of material characteristics.

Non-Destructive Nature: Light microscopy is non-destructive, allowing repeated observations without altering the sample. This is particularly advantageous when conducting initial assessments or when preservation of the sample's integrity is critical.

Surface Features and Macroscopic Characteristics: Light microscopy is effective for studying surface topography, identifying gross features, and assessing macroscopic characteristics. This is especially relevant in material studies where a broader understanding of the sample's overall appearance is essential.

In the specific study at hand, the use of light microscopy may be justified based on the need for a preliminary examination of microfibrils, surface topography, and rupture areas cost-effectively and efficiently. This initial observation can guide researchers in identifying regions of interest for more detailed analyses using higher-resolution techniques, such as SEM or TEM. Light

microscopy, in this context, serves as a valuable tool for obtaining a quick overview and establishing a foundation for further investigations.

Observations of microfibrils, surface topography, and the rupture area were conducted using a light microscope. The type of microscope equipment used for this research was the Leica ATC 2000 Microscope, Wetzlar.

II.7.5.1. Fourier-Transform Infrared (FTIR) spectroscopy

It is a powerful analytical technique used in material studies to elucidate the chemical composition, molecular structures, and bonding characteristics of a wide range of materials. In the context of microfibrils, surface topography, and rupture areas, FTIR spectroscopy provides valuable information about the molecular constituents and chemical interactions within the studied materials.

FTIR spectroscopy is widely used in material studies and plays a crucial role in characterizing and evaluating different materials. FTIR is crucial in polymer and composite characterization as it provides a thorough comprehension of chemical composition and structural attributes. FTIR is a crucial instrument in quality control operations since it allows for quick assessments of material composition. This capability enables the prompt discovery of variances or inconsistencies that could affect the overall quality of the product.

Surface modification studies utilize FTIR analysis to investigate the complexities of surface topography, yielding insights into the impact of alterations on chemical functions, adhesion qualities, and overall surface features. Furthermore, FTIR enables the evaluation of compatibility in composite structures, guaranteeing the most favorable interactions between microfibrils and matrices. This element is crucial in attaining the appropriate mechanical and chemical characteristics in composite materials.

Furthermore, FTIR is utilized for environmental degradation studies, specifically for examining chemical alterations linked to degradation processes like oxidation or hydrolysis. Through the process of elucidating these changes, Fourier-Transform Infrared Spectroscopy (FTIR) plays a crucial role in evaluating the stability of materials under different environmental conditions. This, in turn, helps in developing ways to improve durability and prolong the lifespan of the materials.

FTIR spectroscopy is a highly adaptable and essential instrument in the field of material sciences. It provides useful information about the chemical composition, structural characteristics, and dynamic properties of materials. FTIR is a versatile analytical technique that plays a leading role in materials research. It is used for many purposes such as describing polymers and composites, maintaining quality control, assessing surface changes, evaluating compatibility, and investigating environmental degradation.

Key Aspects of FTIR Spectroscopy

Identification of Functional Groups: FTIR spectroscopy is particularly adept at identifying functional groups present in a material. By analyzing the absorption of infrared radiation, it reveals characteristic peaks associated with different chemical bonds, such as C-H, O-H, N-H, C=O, and others.

Polymer Analysis: In studies involving microfibrils, FTIR is instrumental in characterizing polymers. It helps identify the specific polymer types, variations in chain structures, and any modifications or additives present.

Surface Chemistry: FTIR can be employed to investigate the surface chemistry of materials, providing insights into chemical changes, functionalization, or reactions that may occur at the microfibril surfaces.

Quantitative Analysis: FTIR spectroscopy allows for quantitative analysis, enabling the determination of the concentration of specific functional groups or components in a material. This is valuable for assessing compositional variations.

Material Compatibility: FTIR is used to evaluate the compatibility of different materials in composites. It helps assess the bonding between microfibrils and matrices, offering information on the effectiveness of material integration.

Failure Analysis: In studies focused on rupture areas, FTIR aids in failure analysis by identifying changes in chemical composition at fracture surfaces. It helps determine whether the rupture is associated with chemical degradation, fatigue, or other factors.

Applications in Material Studies

Polymer and Composite Characterization: FTIR is widely applied to characterize polymers and composite materials, providing a comprehensive understanding of their chemical composition and structural features.

Quality Control: FTIR serves as a valuable tool in quality control processes, enabling rapid assessment of material composition and detecting variations or inconsistencies.

Surface Modification Studies: Studies involving surface topography benefit from FTIR analysis to understand how surface modifications impact chemical functionalities, adhesion, and overall surface characteristics.

Compatibility Assessment: FTIR helps evaluate the compatibility of different materials in composite structures, ensuring optimal interactions between microfibrils and matrices.

Environmental Degradation Analysis: FTIR is used to investigate chemical changes associated with environmental degradation, such as oxidation or hydrolysis, aiding in the assessment of material stability.

Meanwhile, the FTIR spectra were recorded at the “Water and Environmental Science and Technology Laboratory” located at University Mohamed Cherif Messaidia, Souk Ahras, Algeria, using an IRAffinity-1S FTIR at a measuring range between 500 and 4000 cm^{-1}

II.7.6. Fatigue cyclic flexure

Fatigue cyclic flexure testing is widely recognized as a vital method in material science and engineering for evaluating the behavior of materials under repetitive bending or flexural loading. It provides critical insights into the formation of cracks, the initiation and propagation of fractures, and the eventual failure mechanisms of materials subjected to dynamic stress. However, in this study, fatigue testing was deemed impractical due to the significant degradation of mechanical properties observed in the composites after exposure to harsh environmental conditions, including immersion in acidic solutions and seawater. The severe reduction in strength and durability of the materials under these conditions rendered the data from fatigue testing non-representative and potentially unreliable for meaningful analysis.

Furthermore, the primary objective of this research is to investigate the impact of extreme environmental conditions on the mechanical behavior of the composites, rather than their cyclic fatigue performance in their current state. Performing fatigue tests on already degraded samples would not provide valuable insights and could lead to conclusions that do not align with the intended scope of the study.

In future work, modifications to the matrix composition and the characteristics of the reinforcing powders will be explored to enhance the durability and environmental resistance of the composites. These improvements are expected to facilitate the incorporation of fatigue cyclic flexure testing into subsequent studies, enabling a comprehensive analysis of the composites' long-term performance and allowing for the prediction of their service life under cyclic loading. This approach ensures that the focus remains on understanding and mitigating the degradation mechanisms while paving the way for a more robust investigation of fatigue behavior in optimized material systems.

II.8. Conclusion

In summary, the chapter commenced with an examination of composite components, delving into matrix types with a particular focus on epoxy resin as a noteworthy option. The discussion extended to reinforcement materials, specifically different types of fiberglass, highlighting their role in enhancing the composite's properties. Additionally, the incorporation of additives,

specifically iron powder, was meticulously explored, addressing granulometry through microscopic analysis, sieving, and assessing purity.

The material processing section elucidated the production methods, leading seamlessly into the experimental procedure. Mechanical testing, including hardness tests, bending strength evaluations, and tensile tests, was comprehensively covered. The inclusion of statistical methods in mechanical testing added a robust analytical dimension to the assessment.

Furthermore, the chapter expanded its scope to investigate the impact of aggressive environments on the composite material. Experimental procedures for solution preparation and the subsequent submersion's effect on specimen weight were discussed, along with key aspects of ANOVA, microscopic, and FTIR analysis. The consideration of moisture absorption and flexure testing rounded out the comprehensive exploration.

As a final note, the chapter culminated with a focused analysis of the effects of harsh environmental conditions on the mechanical behavior of the composites. While fatigue cyclic flexure testing was not conducted due to the significant degradation observed after exposure to acidic and saline environments, this decision highlights the need for future material optimization to improve durability and enable meaningful fatigue testing. This thorough investigation into composite production and environmental performance not only lays the foundation for subsequent chapters but also establishes a robust framework for understanding the material's properties and limitations under both controlled and extreme conditions.

Chapter III

Mechanical Behavior

Results and Discussion

This section explores the extensive range of mechanical tests carried out in this study to assess the performance and structural properties of the manufactured laminated composite plates. Three essential mechanical tests were utilized, specifically Hardness, Flexure, and tensile tests, each offering vital information about the material qualities and behavior. The comprehensive evaluation conducted through these tests is essential for comprehending the appropriateness and efficacy of the composite in practical scenarios.

III.1. Experimental Procedure in Normal Conditions

III.1.1. Hardness Test

An analysis was conducted on the acquired hardness values to establish connections between the composition and manufacturing characteristics of the composite. This research offers valuable insights into the surface integrity of the material and its ability to withstand wear and abrasion. It gives crucial data for applications where hardness is a major performance aspect.

The information displayed in Figure III.1 provides a comprehensive understanding of the hardness characteristics for various compositions. The epoxy resin initially exhibits a rather low degree of hardness, consistent with its known properties. However, adding iron powder as a filler result in a significant increase in the hardness of the material. The fiberglass reinforcement greatly improves the epoxy resin, leading to extremely impressive hardness values between 17.5 and 22.5.

Examining the influence of iron powder in more detail reveals that one important aspect affecting material hardness is the size of the powder's grains. The measurements of the epoxy resin plate taken at various times show a progressive rise in hardness values, from 12 to 16. The hardness increases in proportion to the decrease of iron particle diameter. Significantly, the addition of powder with a diameter of 64 micrometers yields a hardness range of 24 to 25, while the addition of powder with a diameter of 92 micrometers results in a hardness range of 21 to 23. With a diameter of 32 micrometers, the plate containing the tiniest powder grains exhibits the highest hardness values, ranging from 27 to 28. It is evident from this relationship that harder materials have smaller grains, highlighting the significance of powder size in controlling the mechanical characteristics of composite materials.

The data displayed in Figure III.2 represents the highest levels of hardness achieved for each plate. Specifically, these numbers represent the maximum levels of hardness attained under the testing conditions. The epoxy resin has a measured hardness of 13.7, whereas the GFRE has a hardness of 19.52, demonstrating the strengthening impact of fiberglass. The incorporation of iron powder additionally augments material hardness, as evidenced by precise tests that demonstrate the impact of powder particle size. The composite loaded with iron powder of 92 micrometers obtains a hardness of 23.3. The variation with a grain size of 64 micrometers records a hardness of 24.38. The lowest grain size, measuring 32 micrometers, produces the maximum hardness at 27.64. The maximum hardness values are important reference points that give a complete overview of the composite material's mechanical strength when different formulations and situations are considered.

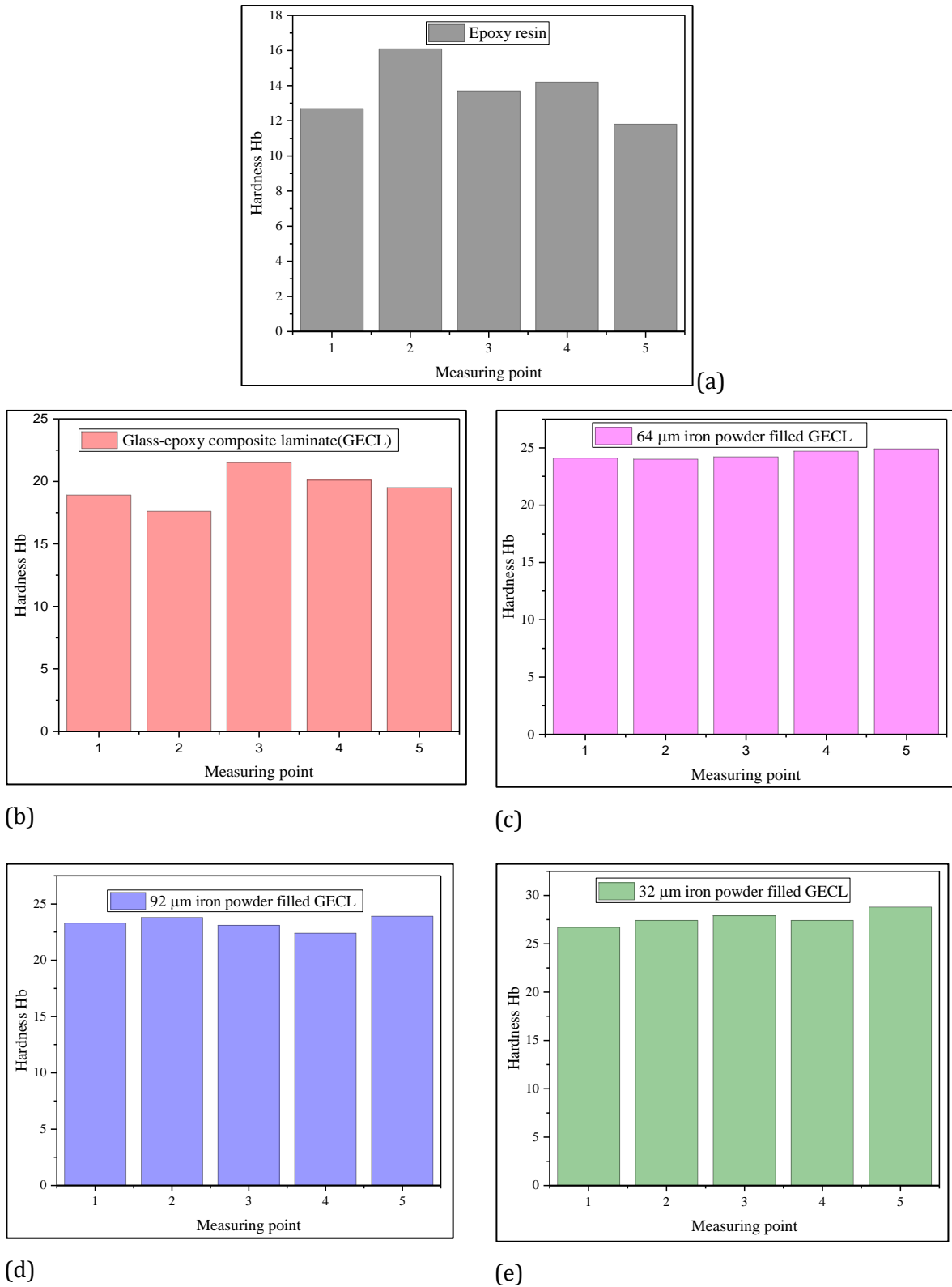


Figure III.1. Hardness measurement points for each plate (a), (b), (c), (d) and (e)

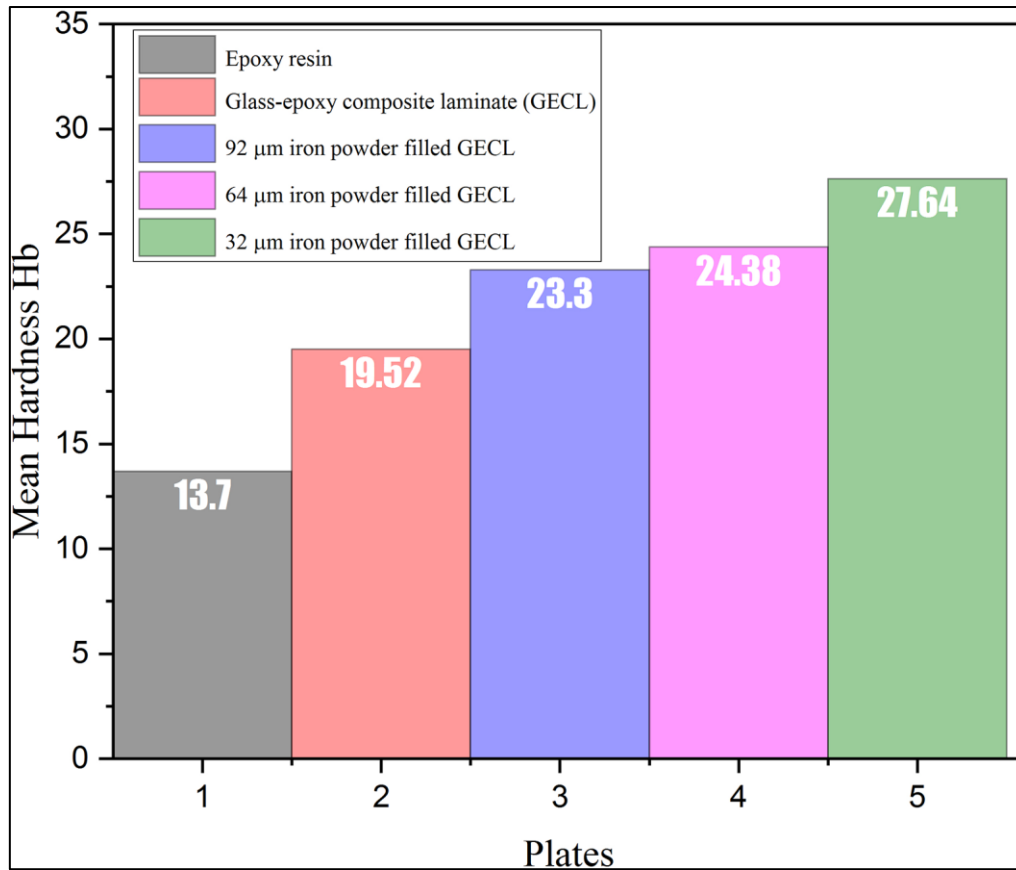


Figure III.2. Brinell Hardness mean value for all composites

In conclusion, the experiment findings show that iron powder works very well as a filler to raise a material's hardness. The size of the powder grains is a significant component of this augmentation and is essential to its accomplishment. These results not only advance our knowledge of the mechanical behavior of the composite but also offer practical information that can be utilized to improve material formulation for a range of applications where hardness is a crucial factor.

III.1.2. Flexure Test

The results from the three-point bending test unveil critical insights into the mechanical behavior of composite materials, with a specific focus on flexure strength and Young's modulus. The study compares two sets of specimens: the first without iron powder (Comparison specimen) and the second incorporating pure iron powder based on an L9 Taguchi orthogonal array. This comparison allows for a detailed examination of how the addition of iron powder, guided by the Taguchi method, influences the material's response to bending forces. The subsequent analysis of flexure strength and Young's modulus provides valuable insights into the impact of iron particles on the structural behavior of the composite, offering a nuanced understanding of the interplay between material composition and mechanical performance.

III.1.2.1. Fiberglass matt epoxy comparison specimen

Table III.1. Comparison of specimen’s characteristics

Content (%)	Velocity (mm/min)	Flexure strength (Mpa)	Young’s modulus (Mpa)
45% fiberglass E + 55% epoxy	1	154.275	8903.355

Table III.1 presents a comparison of the characteristics of the test specimens, including the content percentage, testing velocity, FS, and Young’s modulus. Specifically, the table shows the results for a specimen containing 45% fiberglass and 55% epoxy, tested at a velocity of 1 mm/min. The FS for this specimen was found to be 154.275 MPa, while Young’s modulus was measured at 8903.355 MPa. These values provide insight into the mechanical behavior of the composite material under bending stress and highlight its overall rigidity and strength.

III.1.2.2. Taguchi’s analysis

Table III.2. Experimental results detailed for Taguchi L9 array

Run	A	B	C	Flexure strength (Mpa)	S/N Ratio (dB)	MEAN	Young’s modulus (Mpa)	S/N Ratio (dB)	MEAN
1	15	92	1	236.201	47.4656	236.201	14975.714	83.5078	14975.7
2	15	64	5	230.961	47.2708	230.961	12199.886	81.7271	12199.9
3	15	32	10	310.822	49.8502	310.822	15107.267	83.5837	15107.3
4	20	92	5	341.198	50.6601	341.198	16366.777	84.2793	16366.8
5	20	64	10	322.625	50.1740	322.625	16030.615	84.0990	16030.6
6	20	32	1	261.265	48.3416	261.265	16599.690	84.4020	16599.7
7	25	92	10	259.450	48.2811	259.450	13308.677	82.4827	13308.7
8	25	64	1	279.731	48.9348	279.731	14653.074	83.3186	14653.1
9	25	32	5	311.998	49.8830	311.998	15097.248	83.5780	15097.2

In this context, 'A' denotes the weight percentage of iron content, ranging from 15% to 25%. 'B' signifies the iron grain size in micrometers (μm), with values of 92, 64, and 32. Additionally, 'C' represents the velocity in millimeters per minute (mm/min), with options of 1, 5, and 10.

Table III.2 presents the experimental results for the Taguchi L9 array, detailing the influence of three factors (A, B, and C) on the FS and Young's modulus of the composite material. The table includes values for FS, signal-to-noise (S/N) ratio, and the mean of each measurement for each run. The runs represent various combinations of factor levels, with varying content and conditions, providing insights into how these factors influence the mechanical properties of the material. The results show that FS and Young's modulus fluctuate based on different factor combinations, and the corresponding S/N ratios help identify optimal conditions for material performance. This data is crucial for determining the most effective parameters for achieving high mechanical strength and rigidity in the composite.

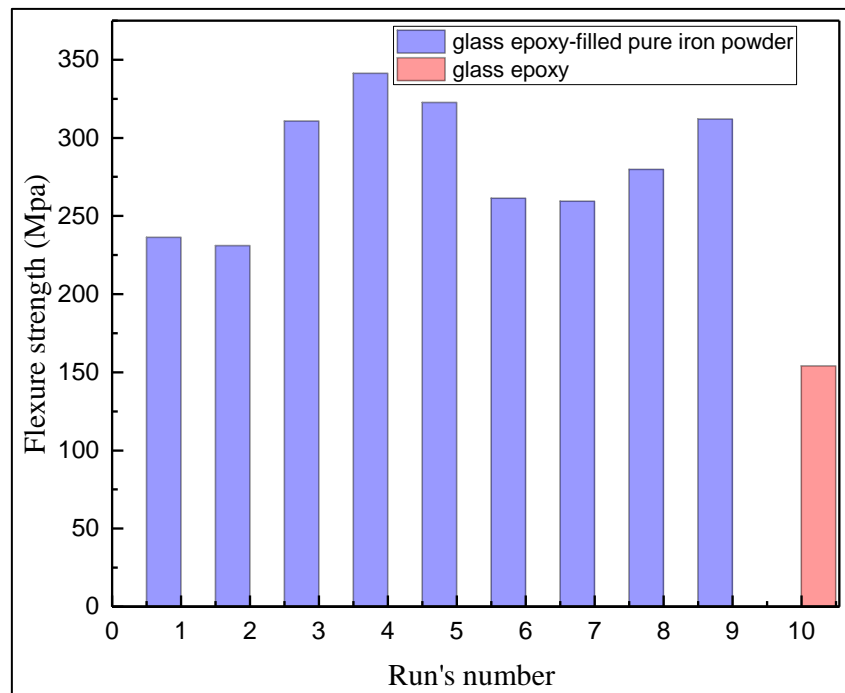


Figure III.3. Bar chart for comparison between all runs flexure's strength and glass epoxy specimens' one.

Figure III.3 presents a bar chart comparing the FS of all the experimental runs with that of the glass epoxy specimens. This chart highlights the variations in FS across different runs, providing a visual representation of how each combination of factors influences the material's mechanical properties. The comparison underscores the performance of the glass epoxy specimens relative to the other test configurations, aiding in the identification of optimal conditions for superior FS.

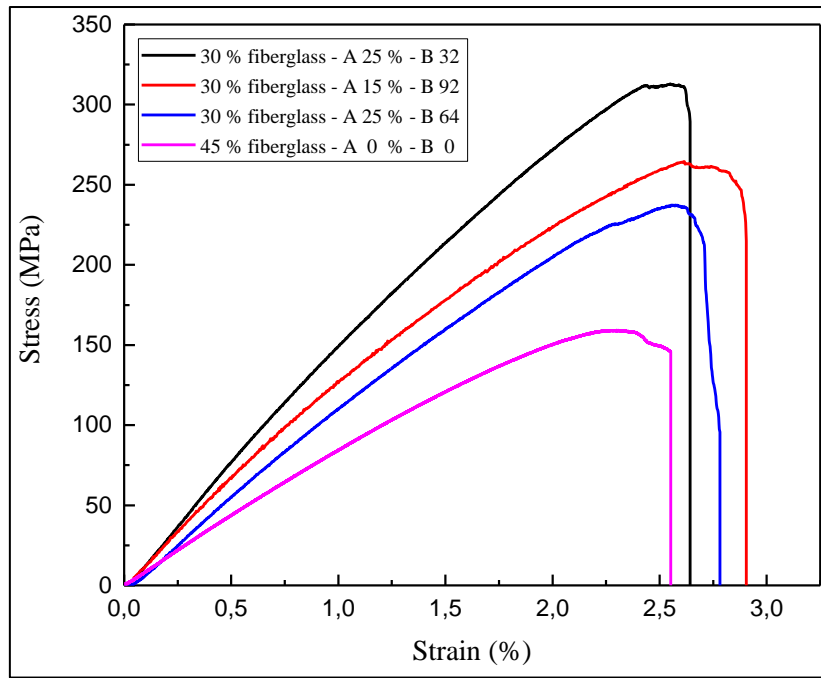


Figure III.4. Comparison of Typical Flexural Stress-Strain Curves for Fiber-Reinforced Composites with and without Powder Loading

The plotted curves in Figure III.4 represent the flexural stress-strain behavior of the three best-performing specimens compared to the specimen containing 45% fiber without powder loading. Notably, the curve for the non-powder-loaded specimen is consistently lower than those of the specimens loaded with pure iron powder. This highlights the significant contribution of powder reinforcement in enhancing flexural performance, emphasizing the critical role of powder loading in improving the mechanical properties of the composites.

Taguchi’s Analysis: Flexure Strength (Mpa) Versus Content (%); Size (μm); Velocity (Mm/min)

By effectively employing this concept, optimal settings for control factors can be determined to achieve peak performance (high signal) in a given process or product, all the while minimizing the impact of uncontrollable influences (low noise). This information is presented in both Table III.3 and Table III.4.

Table III.3. Response Table for Signal-to-Noise Ratios

Level	Content (%)	Size (μm)	Velocity (mm/min)
1	48,20	49,36	48,25

2	49,73	48,79	49,27
3	49,03	48,80	49,44
Delta	1,53	0,57	1,19
Rank	1	3	2

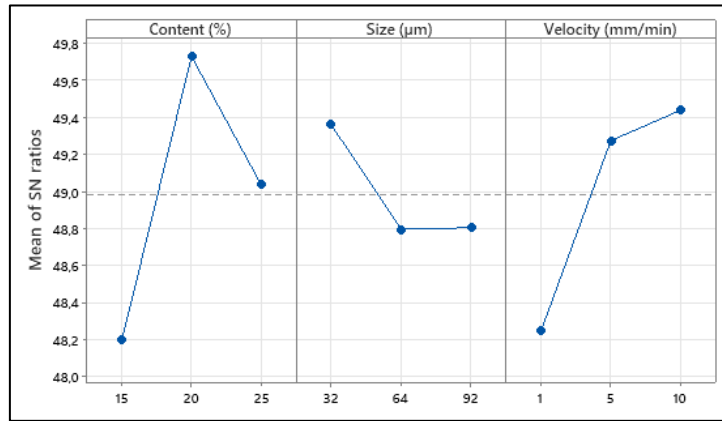


Figure III.5. Main effects plot for SN ratios

Regression Equation for Flexure Strength

$$\text{Flexure Strength (Mpa)} = 229.8 + 2.44 \text{ Content} - 0.269 \text{ Size} + 4.13 \text{ Velocity}$$

Taguchi Analysis: Young's modulus (Mpa) versus Content (%); Size (µm); Velocity (mm/min)

The Signal-to-Noise Ratios (SNR) presented in Table 3 and Table 4 provide a detailed analysis of the effect of content (%), Size (µm), and Velocity (mm/min) on FS and Young's modulus. These tables notably highlight the Delta values, offering a full knowledge of the impact of these components.

Table III.3 displays the Response Table for SNR for FS (Mpa), with the Delta values indicating the relative impact of each element. A Delta value of 1.53 for content (%) signifies a significant influence on FS, highlighting the crucial role of content percentage in determining the observed differences. The Delta values of 0.57 for Size (µm) and 1.19 for Velocity (mm/min) offer more understanding of the importance of these elements, assisting in the determination of ideal conditions for improving FS.

Table III.4. Response Table for SNR

Level	Content (%)	Size (µm)	Velocity (mm/min)
1	82,94	83,85	83,74
2	84,26	83,05	83,19
3	83,13	83,42	83,39
Delta	1,32	0,81	0,55
Rank	1	2	3

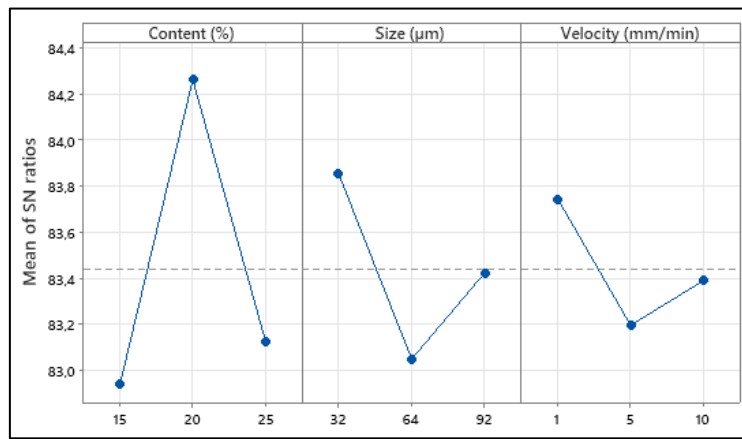


Figure III.6. Main effects plot for SN ratios

Simultaneously, Table III.4 displays the Response Table for SNR for Young's modulus (Mpa). The Delta values, namely 1.32 for content (%), 0.81 for Size (µm), and 0.55 for Velocity (mm/min), provide a detailed understanding of the elements that affect Young's modulus. Although content (%) remains influential, Size (µm) and Velocity (mm/min) also have significant but comparatively smaller effects. The utilization of this dual assessment allows for strategic analysis in identifying the most favorable conditions for Young's modulus, hence enhancing our comprehension of the mechanical properties of the composite material.

The combination of these tables and Delta values are essential tools in our investigation of the Taguchi Method. They help us make informed decisions about optimizing factors to enhance the FS and Young's modulus of the composites we are studying.

Regression Equation for Young's modulus

$$\text{Youngs modulus (Mpa)} = 15528 + 26 \text{ Content} - 12,6 \text{ Size} - 61 \text{ Velocity}$$

III.1.2.3. Discussion

The results obtained from the laminate bending test, together with the analysis utilizing Taguchi's approach, have shown important insights into the elements that affect FS. When evaluating several aspects, the iron powder concentration stands out as the most significant, with test speed being the second most influential, and powder grain size having the least impact.

After careful analysis of the data, a significant pattern becomes apparent: the FS of the laminated composite increases as the iron powder content rises, reaching an optimal weight percentage where the outcomes are most favorable. After reaching this optimal point, flexure strength undergoes a further decrease.

The most effective combination of parameters that produces the highest quality outcomes consists of a powder content of 20%, a powder grain size of 32 μm , and a test speed of 10 mm/min. These particular conditions correspond to the arrangement that optimizes the bending strength of the laminated composite, as determined by the Taguchi method analysis. This comprehensive comprehension of the interaction among these aspects' aids in the strategic adjustment of parameters, directing the development of laminated composites with improved flexure strength for real-world uses.

III.1.3. Tensile Test

III.1.3.1. Results

The tensile test results offer crucial insights into the mechanical performance of four distinct specimens subjected to axial loading. The study encompasses one specimen with epoxy resin and three additional glass epoxy laminates, each filled with varying percentages of iron powder (15%, 20%, and 25%).

This detailed examination of tensile properties across these distinct specimens allows for a comprehensive understanding of how the addition of iron powder, at different concentrations, influences the mechanical behavior of the glass epoxy laminates under axial loading conditions. The results contribute to a nuanced characterization of the tensile performance of these composite materials, guiding insights into the optimal composition for specific applications.

Table III.5. Young’s Modulus and Tensile Strength experimenting results

Group	Specimen	Surface	Young’s Modulus	Tensile Strength
A	1	49.1454	3.391966709	48.68064559
	2	49.1454	3.303741844	48.04974773
	3	49.1454	3.88676993	38.9049239
	4	49.1454	3.632165727	45.22012404
	5	49.1454	3.512066218	46.63493589
B	1	29.784	8.863744506	138.0004391
	2	28.482	8.083658914	136.2869223
	3	27.2468	6.517165409	134.5741282
	4	28.5042	7.377609248	139.4728807
	5	27.5766	7.764630881	136.2786353
C	1	30.433	15.15899612	152.1597528
	2	30.651	15.2044286	151.3940126
	3	29.301	14.81158406	141.9094885
	4	31.7822	15.18171236	151.7768827
	5	30.8823	14.41873952	148.4877513
D	1	31.731	9.886521791	131.395752
	2	31.0503	9.464182003	129.3528975
	3	33.0924	11.01315512	135.646306
	4	30.12	10.30886158	130.3743247
	5	30.3696	8.061243471	128.8079831

The provided Table III.5 displays the surface, Young's modulus, and tensile strength of each specimen. It is worth noting that the powder-loaded specimens exhibit substantial improvements in their tensile mechanical properties when compared to the resin specimen without any powder. Simultaneously, a noticeable rise in tensile strength is noted as the amount of powder increases, reaching its maximum at 20%, where both the highest tensile strength and Young's modulus are attained. Nevertheless, when the value reaches 25%, it decreases significantly, roughly approximating the outcomes reported at a loading of 15%. The observed pattern emphasizes the complex correlation between the amount of powder used and the physical characteristics of the material. It demonstrates that the best tensile performance is reached when the powder loading is at 20%. This finding is important for customizing composite formulations to improve their mechanical properties.

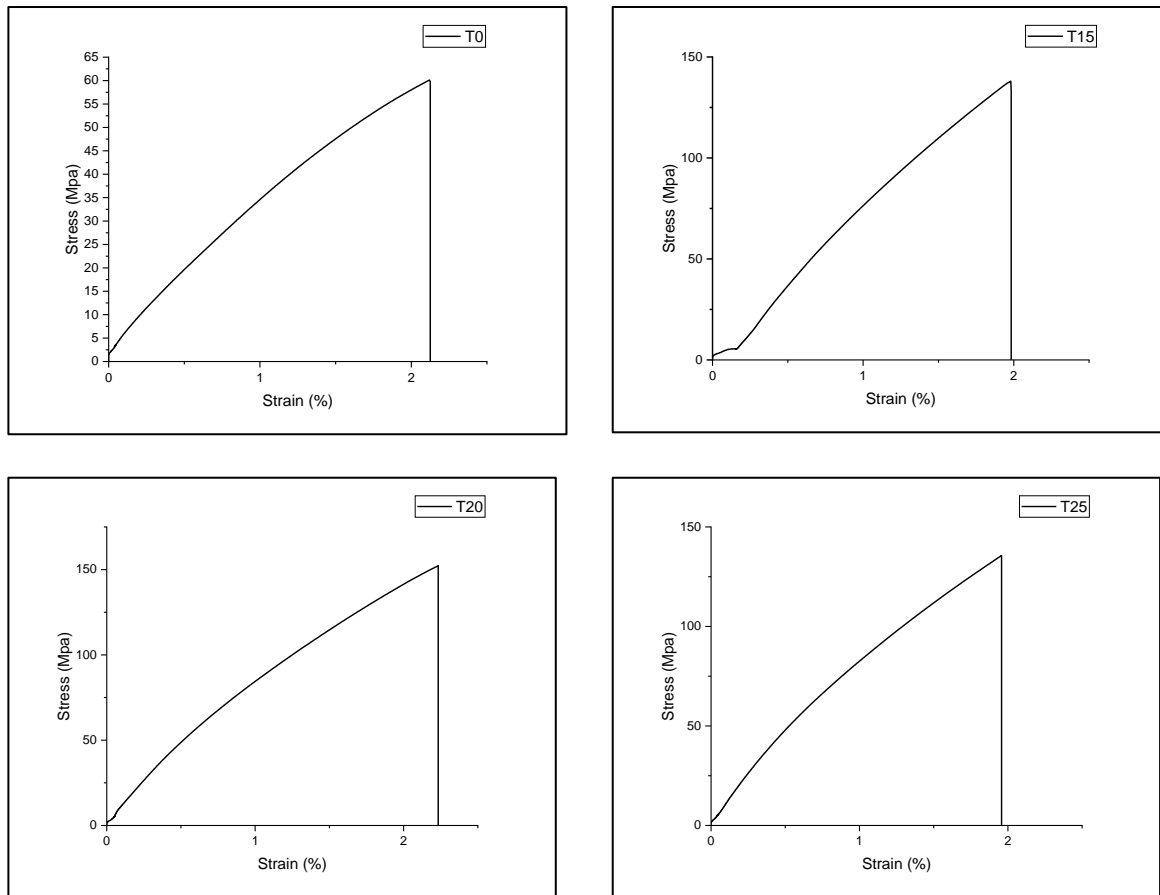


Figure III.7. Tensile Test Analysis: Effect of Iron Powder Content on Stress-Strain Behavior

III.1.3.2. Discussion

The observed results from the tensile tests provide valuable insights into the mechanical behavior of the composite specimens, shedding light on the impact of iron powder loading on key properties. Notably, the loaded specimens exhibit a substantial improvement in tensile characteristics compared to the unloaded epoxy resin specimen. This enhancement is particularly evident in terms of tensile strength and Young's modulus.

The progressive increase in tensile strength with rising iron powder loading up to 20% is a noteworthy trend. At this loading level, the specimens showcase the highest tensile strength and Young's modulus. This indicates that the inclusion of iron powder, up to a certain concentration, acts as a reinforcing agent, enhancing the material's ability to withstand tensile forces and promoting stiffness.

However, the decrease in tensile strength and Young's modulus at 25% loading, approaching values observed at 15%, suggests a saturation or potential threshold effect. This finding implies that beyond a certain concentration, additional iron powder may not contribute significantly to further improvement and may even lead to a slight reduction in mechanical properties.

This nuanced understanding of the relationship between iron powder loading and tensile properties is pivotal for optimizing composite formulations. The results suggest that loading of 20% iron powder achieves the best compromise between enhanced tensile strength and stiffness. Beyond this point, diminishing returns or potential negative effects may be anticipated.

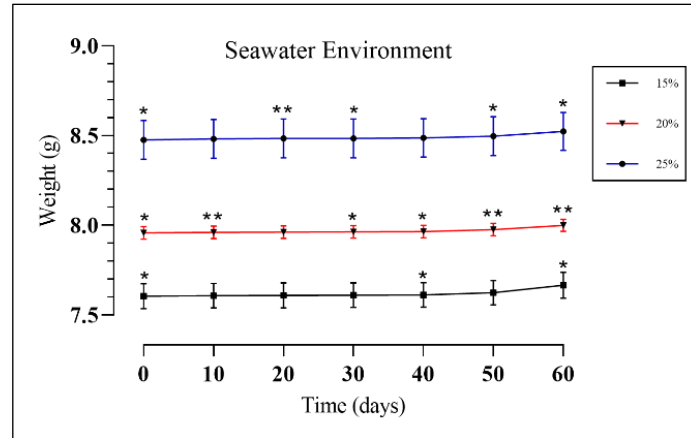
These findings are crucial for guiding the formulation of composite materials, allowing for the tailored design of materials with optimal mechanical performance for specific applications. The nuanced interpretation of the results underscores the importance of carefully balancing the concentration of reinforcing agents in composite materials to achieve desired mechanical properties.

III.2. Experimental Procedure in Aggressive Environment

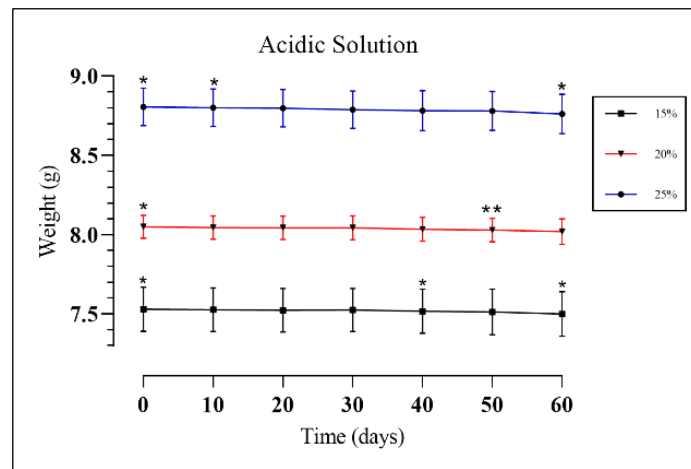
III.2.1. Submersion's Impact on Specimen Weight

This study employed a Two-Way ANOVA approach to examine the variations in the weight of the specimens when subjected to immersion in two distinct solutions for no less than 60 days: a seawater environment and an acidic solution. The present investigation took into account the variables of immersion duration and the proportion of iron powder loading. The outcomes exhibited noteworthy disparities in comparison to the specimens that were not subjected to immersion, which can be referred to as the control group (C).

Compared with the control, the weight change illustrated in Figure 6 was significant ($p < 0.05$), proportional to the time of immersion. In such a way a noticeable increase in weight was observed over time in specimens immersed in seawater Figure III.8-a, indicating significant water absorption. This aligns with findings who reported increased water absorption in glass/epoxy composites exposed to seawater over a year, supporting the impact of seawater on weight gain in composite materials [45]. On the other hand, specimens that were fully submerged in the acidic solution exhibited a weight reduction Figure III.8-b that was directly related to the duration of exposure. This echoes the findings who investigated the effects of concentrated HCl on aged fiberglass polyester composites [65]. This weight decrease phenomenon could be attributed to corrosion and material degradation. In corrosive environments like acidic solutions, the chemical reactions involving the iron powder within the composite may release iron ions, leading to material corrosion. This corrosion process contributes to the observed loss of material mass, aligning with researcher's observations of significant destructive impacts in acid-immersed composites.



(a)



(b)

Figure III.8. Charts (a) and (b) display the evaluation of the masses of various test specimens over time.

III.2.1.1. Moisture absorption and Weight loss

The process of moisture absorption was quantified for specimens that were submerged in seawater Table III.6. It was shown that these specimens exhibited moisture absorption, leading to a subsequent increase in their overall weight. On the other hand, the specimens that were submerged in the acidic solution exhibited a reduction in mass as shown in Table III.7

$$\% = (W_i - W_0)/W_0$$

Table III.6. Sub-groups moisture absorption

Sub-group	Absorption (%)
1	0.79

2	0.52
3	0.55

Table III.7. Sub-groups weight loss

Sub-group	Weight loss (%)
1	0.20
2	0.41
3	0.50

A direct correlation is noted between the quantity of iron powder present in the laminate and the corresponding reduction in moisture absorption. Stated otherwise, an increase in resin content corresponds to an increase in the moisture absorption capacity of the composite material. Furthermore, it was observed that the test specimens, when subjected to immersion in the acidic solution, exhibited a weight reduction that was directly proportionate to the quantity of iron contained within them. The observed reduction in weight can be ascribed to the deterioration of the composite material caused by the influence of hydrochloric acid (HCl), as mentioned before.

III.2.2. Flexure strength and Young Modulus

III.2.2.1. Experimental results detailed

The analysis focused on the results obtained from a three-point bending experiment carried out on specimens that underwent immersion for a minimum period of 60 days. Figure III.9 illustrates the typical stress/strain curve for each group, with detailed results of FS and Young's modulus for each sub-group presented in Table III.8.

In such a case that

Figure III.9(a) depicts the stress/strain plot for the sub-group submerged in seawater. In Figure III.9(b), the stress/strain plot illustrates the performance of the sub-group submerged in an acidic solution. Finally, Figure III.9(c) presents the stress/strain plot for the non-submerged sub-group, referred to as the control group."

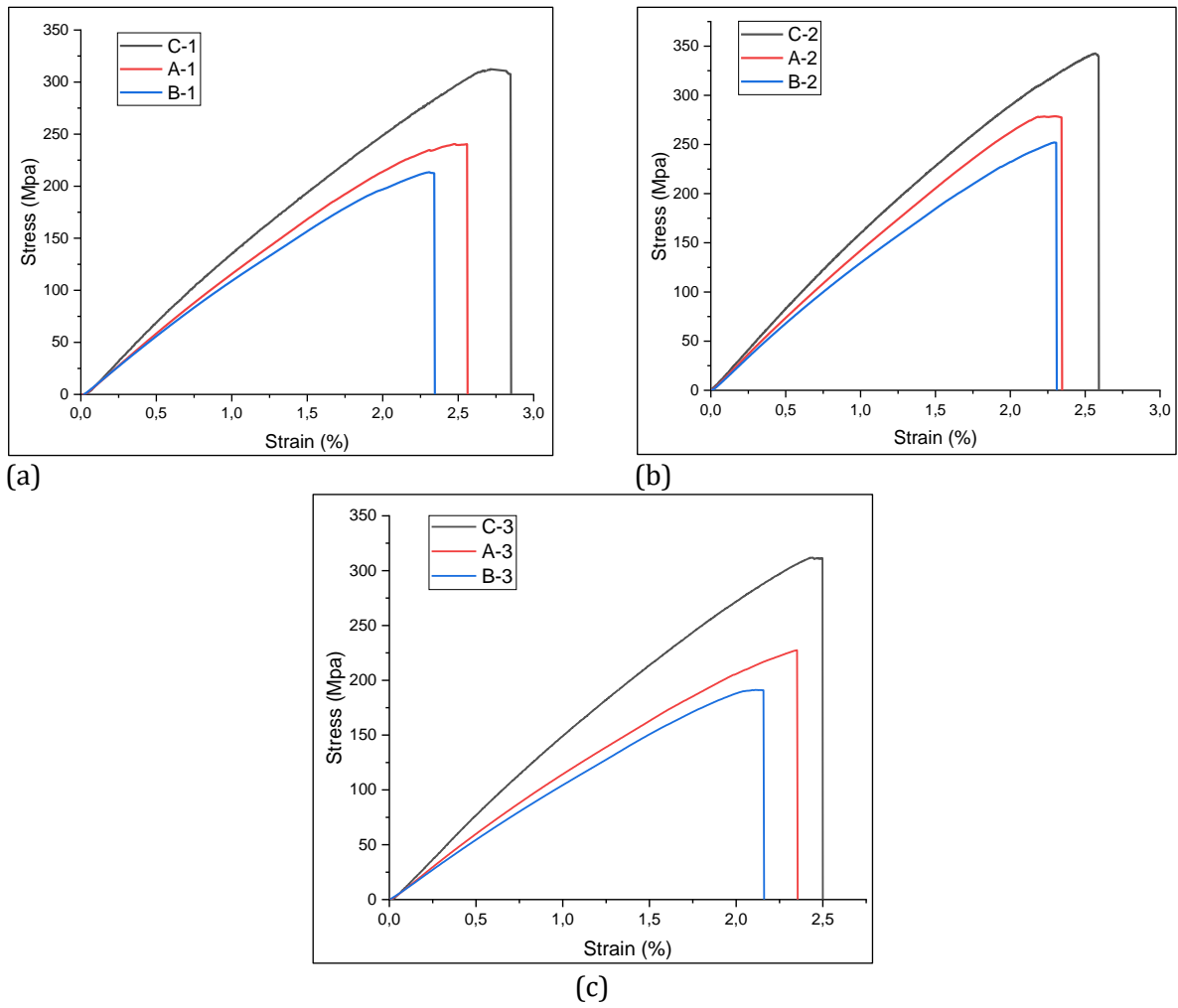


Figure III.9. Stress-Strain Behavior of Sub-groups under Different Environmental Conditions: Seawater, Acidic Solution, and Unsubmerged Control

Table III.8. Three Points Bending Test Results Detailed

Group	Sub-groups	Iron content (W%)	Solution	Flexure Strength (Mpa)	Young's Modulus (Mpa)
A	1	15	Seawater	240.5714	10211.82
	2	20	Seawater	263.7119	11162.67
	3	25	Seawater	219.1429	9830.429
B	1	15	Acid	201.5714	8961.479
	2	20	Acid	243.9256	9588.857

	3	25	Acid	193.5714	8200.84
C	1	15	/	310.822	15107.267
	2	20	/	341.198	16366.777
	3	25	/	311.998	15097.248

The present investigation utilized a Two-Way ANOVA approach to investigate the changes in flexure strength and Young's modulus.

The current study considered the factors of FS and Young's modulus, as well as the solution type and iron powder loading %. The results (as shown in Figure III.10) displayed significant differences when compared to the samples that were not exposed to immersion, which can be referred to as the control group (C). The observed reduction in both flexure strength and Young's modulus was found to be statistically significant when compared to the control group ($p < 0.05$).

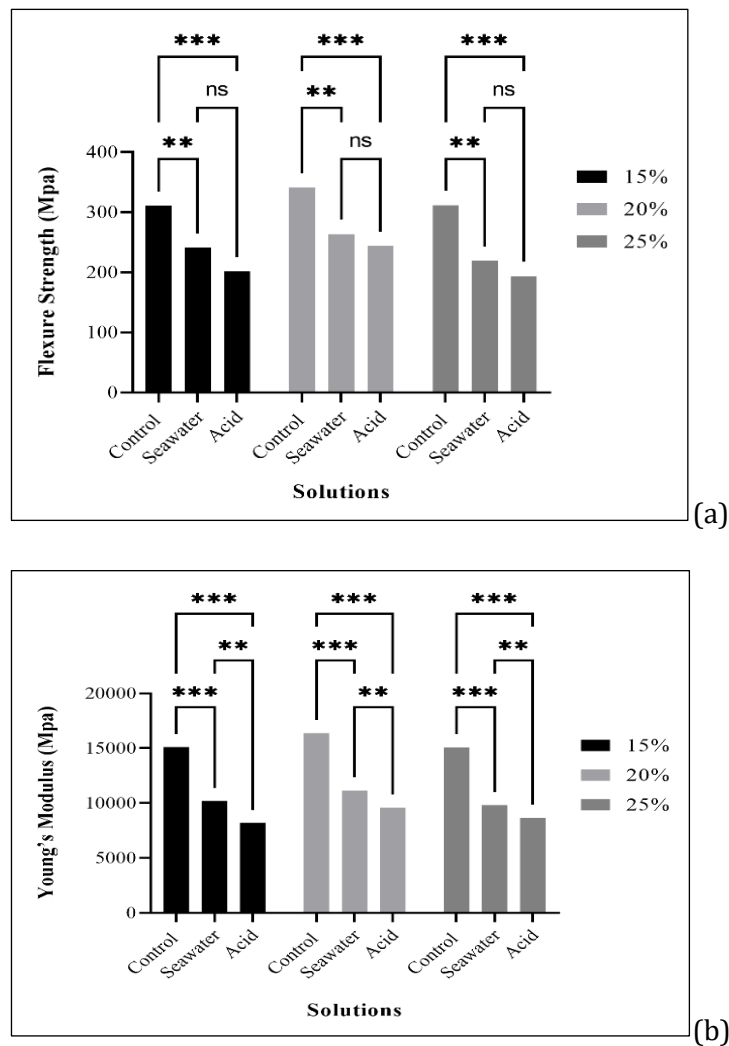


Figure III.10. ANOVA Analysis of FS (a) and Young's modulus (b) for composite materials

After the results were evaluated using the ANOVA method, the findings of the study (Figure III.10) demonstrated a significant decrease in both FS and Young's modulus in the immersed composites as compared to the composites that were not subjected to immersion.

The Flexure Strength of Composites 1, 2, and 3 (Figure III.10-a) in group A decreased by 22.6%, 22.7%, and 29.7%, respectively. Likewise, within group B, laminates 1, 2, and 3 demonstrated a reduction in their FS of 35.1%, 28.5%, and 37.9% correspondingly.

When examining Young's modulus (Figure III.10-b), it was observed that group A, immersed in seawater, experienced reductions of 32.4%, 31.8%, and 34.8% in sub-groups 1, 2, and 3, respectively, when compared to their non-immersed counterparts. In contrast, group B, exposed to the acidic solution, demonstrated a more substantial decline, with decreases of 40.6%, 41.4%, and 45.6% in sub-groups 1, 2, and 3, respectively, compared to their non-immersed counterparts. These findings underscore the significant impact of the acidic solution on Young's modulus in comparison to the specimens not subjected to immersion.

A notable observation was made while comparing the specimens that underwent immersion in an acidic solution with those that were exposed to seawater, as a more prominent reduction in both FS and Young's modulus was observed. In line with the observations from the study [66], in which the results obtained similarly highlight a notable reduction in FS and Young's modulus for specimens immersed in an acidic solution compared to those exposed to seawater. This correspondence further underscores the higher reactivity of acidic solutions and the consequent mechanical degradation observed in laminates, as indicated in the mentioned study.

The results presented above emphasize the need to consider environmental factors when evaluating the durability and effectiveness of composite materials in real-world situations.

III.2.3. Microscopic and FTIR analysis

The microscopic images in Figures III.11-a and III.11-b capture the specimen's behavior upon immersion in an acidic solution, illustrating the conditions after the exposure. These images depict the formation of pores on the epoxy layer, which serves as the matrix material for the specimen. The pore formation within the matrix indicates a degradation process that directly correlates with the significant reduction in mechanical strength observed in the specimens subjected to acidic immersion. The chemical interaction between the epoxy matrix and the acidic solution likely weakens the matrix structure, leading to the material's deterioration. In contrast, Figure III.11-c provides a detailed view of the specimen submerged in seawater, where excessive corrosion is evident. This corrosion is primarily due to the absorption of seawater into the epoxy matrix, which accelerates the breakdown of the material's surface and structure. The prolonged exposure to seawater not only facilitates the corrosion process but also worsens the overall integrity of the composite, leading to further mechanical weakening.

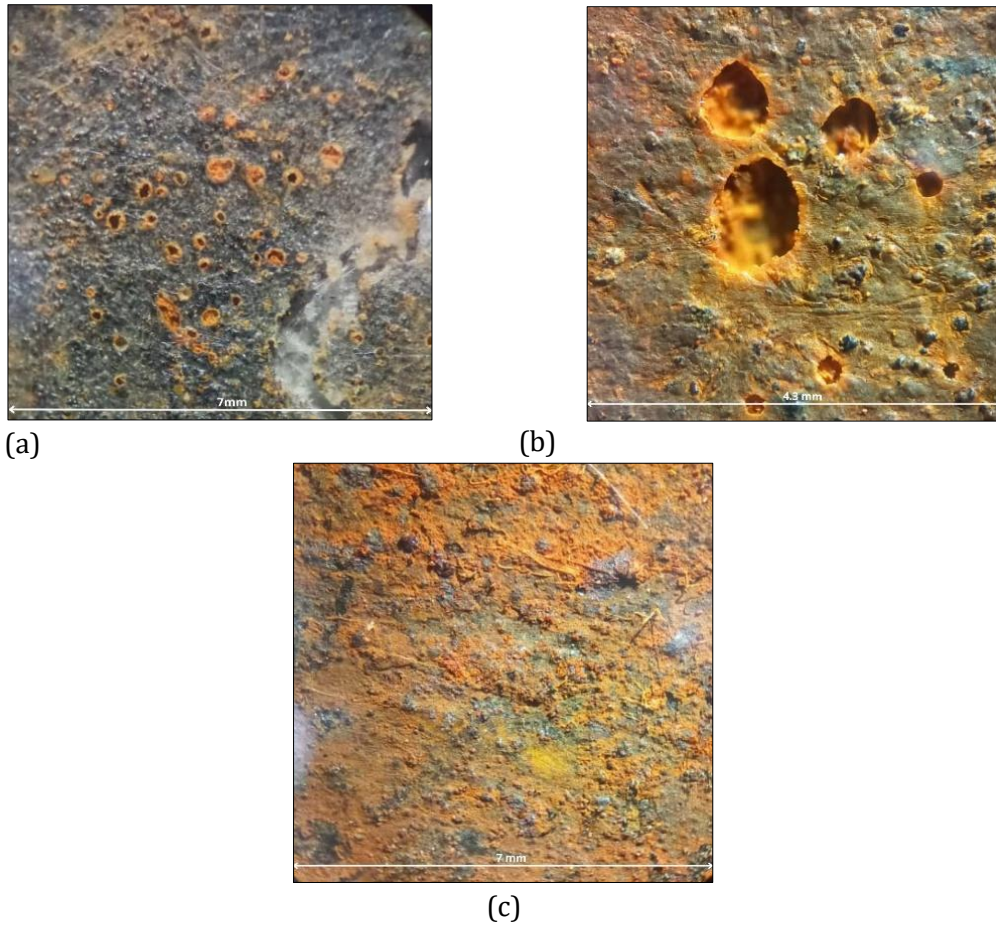


Figure III.11. Microscopic images of the surfaces of the specimens immersed in the acid solution (a) (b) and seawater (c).

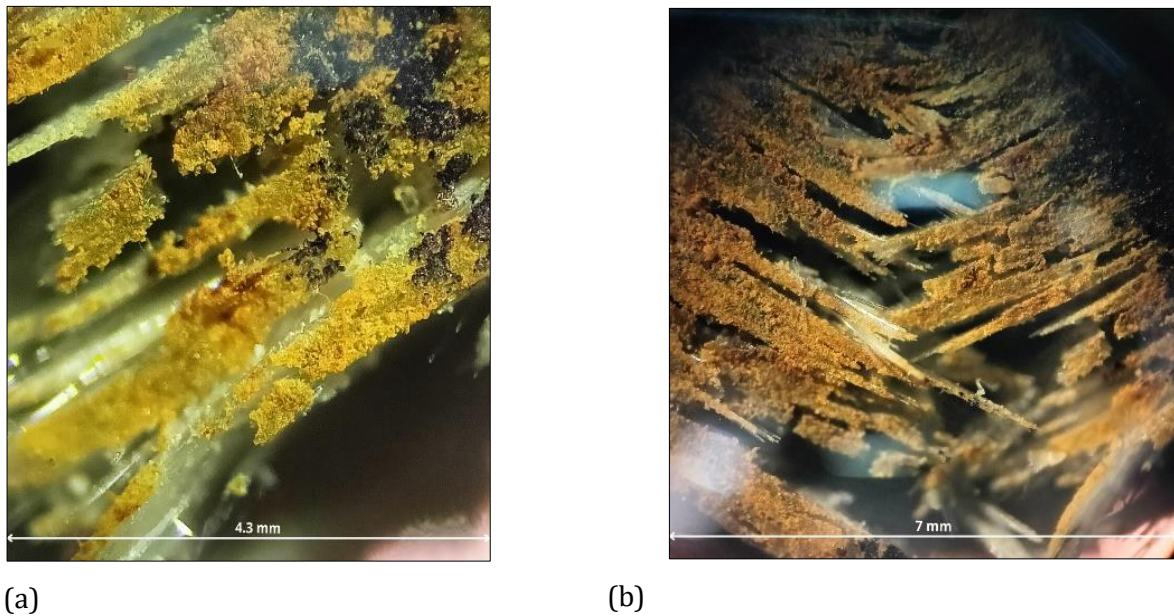


Figure III.12. Analysis of Fracture Surface in Test Specimens after Exposure

The microscopic images in Figure III.12 reveal the oxidation of iron grains within the glass fibers, which led to a reduction in the mechanical properties of the various specimens.

The diagram of Figure III.13 shown below displays the FTIR spectra of distinct specimens while immersed in different solutions: seawater, a pH 2 acid medium, and specimens that were not immersed. The absorption peaks occur at a frequency of 3865 cm^{-1} , which is specifically related to the stretching vibration of hydroxyl (-OH) bonds found in functional groups like water or other hydroxylated compounds. This frequency range also includes values between 3800 and 3900 cm^{-1} . Seawater inherently contains hydroxyl groups.

The frequency at 3734 cm^{-1} corresponds to the stretching vibration of a carbonyl (C=O) bond present in specific organic or inorganic compounds, including corrosion products prevalent in the maritime environment or due to the iron powder presence in our composite.

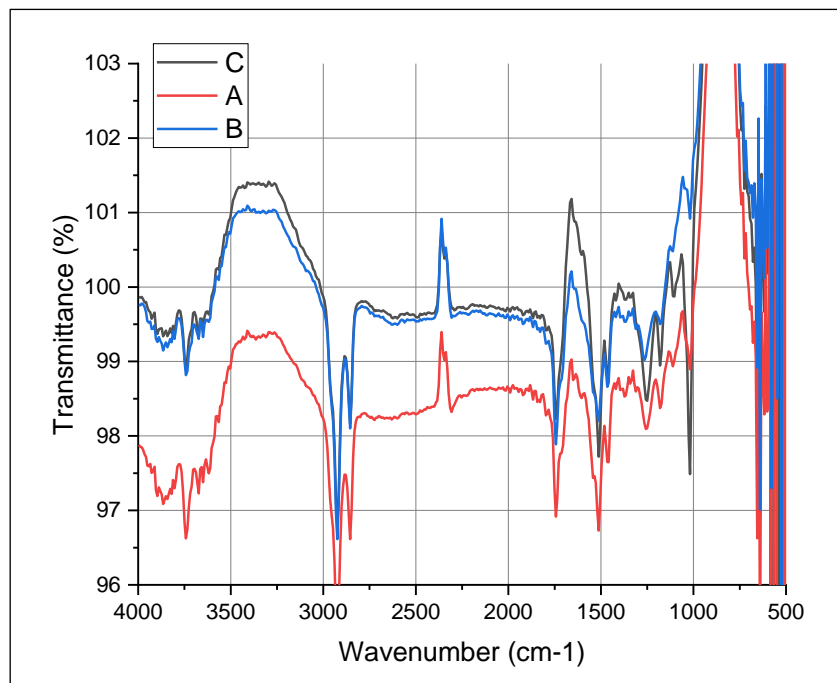


Figure III.13. Diagram of FTIR spectra for different specimens.

The peak observed at 1743 cm^{-1} corresponds to the stretching vibration of the C=O bond in the -COO- functional group. Additionally, the peaks at 1473 cm^{-1} and 1373 cm^{-1} are associated with the bending vibration of the -CH₃ groups in the -CH (CH₃)₂ moiety. The peak observed at 1242 cm^{-1} corresponds to the stretching vibration of the C-O bond in the -COOH functional group, while the peak at 1172 cm^{-1} is attributed to the stretching vibration of a C-O bond. These could be found in functional groups such as esters, ethers, or the vibration of the C-O-C skeleton.

The predominant peak belongs to the epoxy cycle within the region of 1270 - 1240 cm^{-1} . The epoxy functions exhibit strong absorption at 1257 cm^{-1} , which corresponds to the symmetrical vibrations of C-O-C. Additionally, two peaks at 1153 cm^{-1} correspond to the vibrations of CH₂

groups in the epoxy. The absorption at 3410 and 3382 cm^{-1} in amines is attributed to the vibration of primary amine groups (-NH₂) in the hardener structure [67].

The intense peak observed at 2931 cm^{-1} and 2870 cm^{-1} in other groups is associated with the CH groups of the alkane function, and its absorption limit occurs at 1567 cm^{-1} . Ether groups exhibit two distinct absorption peaks at 1257 and 1233 cm^{-1} . The first peak corresponds to C-O-C groups that are connected to aromatic cycles, while the second peak arises from the phatic stretching of carbon-oxygen (-O-CH₂) bonds [68].

Epoxy contains functional groups known as epoxy groups (C-O-C) which can undergo a reaction with water. The three spectra exhibit a high degree of similarity. Nevertheless, alterations in the absorption bands linked to these functional groups, particularly in the range of 1018 to 1735 cm^{-1} , may suggest chemical modifications associated with water absorption. This is evident in the changes observed in the bands at 1018 cm^{-1} , 1118 cm^{-1} , the 1249 cm^{-1} vibration band, and the 1365 cm^{-1} band, which affect the bonding bands.

Water absorption can cause the polymeric matrix to become softer, and acid can affect iron powder, leading to alterations in bonding vibration bands. The absorption bands related to C-C and C-O-C groups in the epoxy matrix may exhibit alterations that indicate modifications in the polymeric structure, as evidenced by the images showing cavities in the material matrix.

The absorption peak observed at 1404 cm^{-1} corresponds to the presence of carbon-nitrogen bonds (CN). Additionally, the range between 1172 and 1200 cm^{-1} is commonly linked to the stretching vibrations of C-N bonds in primary amines. Secondary and tertiary amines can also display bands in this region, however, the position of these bands may vary significantly depending on the characteristics of the group.

The presence of an absorption band at 1249 cm^{-1} in an infrared spectrum can be indicative of many chemical bonds: CO (carbon-oxygen bonds): The stretching vibrations of CO bonds can be linked to this particular location. It may also exist in functional groups like esters, ethers, or other molecules that have carbon-oxygen linkages. The presence of amines or nitriles can also result in stretching vibrations of CN bonds, which can contribute to this region.

Research demonstrates that a spectral peak at 1100 cm^{-1} , accompanied by a smaller peak at 1198 cm^{-1} , can be attributed to the vibrational motion of Si-O bonds in SiO₂ [69].

III.2.3. Conclusion

In conclusion, the comprehensive examination of various mechanical properties in this chapter provides valuable insights into the behavior and performance of composite materials, particularly those incorporating iron powder as a filler.

The hardness test results underscore the effectiveness of iron powder as a filler, demonstrating a notable increase in material hardness. The size of the powder grains emerges as a critical factor influencing this enhancement. These findings not only contribute to a deeper understanding of composite mechanical behavior but also offer practical insights for optimizing material formulations in applications where hardness is a crucial parameter.

Moving to the flexure test, the analysis reveals a distinct pattern in the influence of iron powder concentration on FS. The optimal combination of parameters, identified through Taguchi's approach, showcases the highest quality outcomes. This nuanced understanding allows for the strategic adjustment of parameters, facilitating the development of laminated composites with improved flexure strength tailored for real-world applications.

The tensile test discussion further enriches our understanding, highlighting the positive impact of iron powder loading on tensile characteristics up to a concentration of 20%. Beyond this threshold, diminishing returns or potential negative effects may be anticipated, emphasizing the need for a balanced approach in composite formulations.

Shifting the focus to the aggressive environment analysis, the immersion of composites in seawater and acidic solutions revealed significant reductions in both FS and Young's modulus. The comparison between these environments emphasizes the higher reactivity of acidic solutions, correlating with prior studies and emphasizing the importance of environmental considerations in assessing composite material durability.

In essence, this chapter contributes not only to the scientific understanding of composite materials but also offers practical guidance for optimizing formulations, considering environmental factors, and making informed decisions in research methodologies. These insights pave the way for the development of advanced composites tailored for specific applications, aligning with the ever-evolving demands of real-world scenarios.

Chapter IV
General Conclusion and
Perspectives

IV.1. General Conclusion

The thesis extensively explores the effects of incorporating pure iron powder as a filler in glass epoxy composite laminates, with a specific focus on its impact on FS, tensile strength, and hardness. This involved creating laminate plates with varying iron content (15%w, 20%w, and 25%w) and grain sizes (92 μm , 64 μm , and 32 μm) while maintaining a consistent 30% fiberglass content.

The research reveals that changes in iron powder content and grain size significantly influence the mechanical characteristics of the composite material. Specific filler percentages and reduced grain sizes notably enhance hardness and FS. Comparative analyses with a 45% glass epoxy laminate and assessments against resin and the 45% glass epoxy laminate further emphasize the superior performance of the iron powder-enhanced composites.

Additionally, the thesis examines the tensile properties of various specimens, providing insights into how different concentrations of iron powder shape the mechanical behavior of glass epoxy laminates under axial loading conditions. The results show a progressive increase in tensile strength up to 20% loading, beyond which diminishing returns or potential negative effects are observed.

Moreover, the thesis investigates the impact of seawater and acidity on glass epoxy composites filled with pure iron powder, revealing significant declines in FS under harsh environmental conditions. The section dedicated to exploring the cyclic fatigue behavior of materials under flexural loading analyzes the results of static bending tests conducted in challenging conditions, demonstrating a substantial decrease, up to 40%, in the mechanical properties of the materials following static bending.

Overall, this thesis advances our understanding of iron powder-enhanced composite materials, providing valuable insights into their mechanical performance under various conditions. The findings offer important guidance for optimizing composite formulations and designing materials with optimal mechanical performance.

IV.2. Perspectives

In the perspective section, we would suggest stating:

1. Explore chemical treatment of iron powder to enhance its resistance to aggressive environments and study the mechanical fatigue behavior of this material.
2. Investigate mechanical treatment of grains to improve the contact surface with the resin.
3. Consider switching to a higher density resin such as HDPE to potentially enhance material properties.

References

References

- [1] Narasimha Boddeti, David W. Rosen, Kurt Maute, Martin L. Dunn, "Multiscale optimal design and fabrication of laminated composites," *Composite Structures*, vol. 228, no. 1, p. 111366, 2019.
- [2] Dipen Kumar Rajak, Durgesh D Pagar, Pradeep L Menezes and Emanoil Linul., "Fiber-Reinforced Polymer Composites: Manufacturing, Properties, and Applications.," *Polymers*, vol. 11, no. 10, p. 1667, 2019.
- [3] N. Mortas, O. Er, P. N. B. Reis and J. A. M. Ferreira, "Effect of corrosive solutions on composites laminates subjected to low velocity impact loading.," *Composite Structures*, vol. 108, pp. 205-211, 2014.
- [4] Deng, X. Zhang and Z., "Effects of Seawater Environment on the Degradation of GFRP Composites by Molecular Dynamics Method," *Polymers*, vol. 14, no. 14, p. 2804, 2022.
- [5] Gülsoy, M. Taşdemir and H. Ö., " Mechanical Properties of Polymers Filled with Iron Powder.," *ternational Journal of Polymeric Materials and Polymeric Biomaterials*, vol. 57, no. 3, pp. 258-265, 2008.
- [6] A. Gungor, "Mechanical properties of iron powder filled high density polyethylene composites.," *Materials & Design*, vol. 28, no. 3, pp. 1027-1030, 2007.
- [7] S. Basavarajappa, K. V. Arun and J. Davim., "Effect of Filler Materials on Dry Sliding Wear Behavior of Polymer Matrix Composites – A Taguchi Approach," *Journal of Minerals & Materials Characterization & Engineering*, vol. 8, no. 5, pp. 379-391, 2009.
- [8] Sihama Issa Salih, Safaa Nayyef, Alyaa H.Abd Alsalam, Ammar Mousa Hasan, " Evaluation Mechanical Properties of Polymer Composites," *Eng. &Tech.Journal*, vol. 33, no. 7, pp. 1348-1360, 2015.
- [9] M. Ghorbanzadeh Ahangari, A. Fereidoon, M. Jahanshahi and N. Sharifi, "Effect of nanoparticles on the micromechanical and surface properties of poly(urea-formaldehyde) composite microcapsules," *Composites Part B: Engineering*, vol. 56, pp. 450-455, 2014.
- [10] Nallusamy, Dr, and Karthikeyan, "Synthesis and Wear Characterization of Reinforced Glass Fiber Polymer Composites with Epoxy Resin Using Granite Powder," *Journal of Nano Research*, vol. 49, pp. 01-09, 2017.
- [11] "What Are Composites? - Romeo RIM," 2023. [Online]. Available: <https://romeorim.com/what-are-composites/>.
- [12] "Les composites, c'est quoi?," l'agence Drupal bluedrop.fr, 2024. [Online]. Available: <https://www.polyvia-formation.fr/actualites/la-plasturgie-cest-quoi/les-composites-cest-quoi>. [Accessed Avril 2024].

- [13] D. Kumar, S. R. Boopathy, D. Sangeetha and G. Bharathiraja, "Investigation of Mechanical Properties of Horn Powder-Filled Epoxy Composites," *Journal of Mechanical Engineering*, vol. 63, no. 2, pp. 138-147, 2017.
- [14] Ozsoy, Iskender; Demirkol, Askin; Mimaroglu, Abdullah; Unal, Huseyin; Demir, Zafer, "The Influence of Micro- and Nano-Filler Content on the Mechanical Properties of Epoxy Composites," *Journal of Mechanical Engineering*, vol. 61, pp. 601-609, 2015.
- [15] Fu, Shao-Yun; Feng, Xi-Qiao; Lauke, Bernd; Mai, Yiu-Wing, "Effects of particle size, particle/matrix interface adhesion and particle loading on mechanical properties of particulate-polymer composites," *Composites: Part B*, vol. 39, no. 6, p. 933-961, 2008.
- [16] Sui, G.; Jana, S.; Salehi-khojin, A.; Neema, S.; Zhong, W. H.; Chen, H.; Huo, Q., "Thermal and Mechanical Properties of Epoxy Composites Reinforced by a Natural Hydrophobic Sand," *Journal of Applied Polymer Science*, vol. 109, no. 1, p. 247-255, 2008.
- [17] Valášek, Petr; Müller, Miroslav; Hloch, Sergej, "RECYCLING OF CORUNDUM PARTICLES TWO-BODY ABRASIVE WEAR OF POLYMERIC COMPOSITES BASED ON WASTE," *Tehnički vjesnik*, vol. 22, no. 3, pp. 567-572, 2015.
- [18] B. Vincent, S. Natesan and G. Anand., "Investigation on Thermo-Mechanical Behaviour of E Glass Fibre and Iron Oxide Filler Particles Reinforced Epoxy Composite," *International Journal for Research in Applied Science and Engineering Technology*, vol. 8, pp. 1522-1526, 2020.
- [19] M. Megahed, A. Fathy, D. Morsy and F. Shehata, "Mechanical Performance of glass/epoxy composites enhanced by micro- and nanosized aluminum particles.," *Journal of Industrial Textiles*, vol. 51, no. 1, 2019.
- [20] B.Aravinth, G.Vivek, S.Gokulakrishanan, D.Manikandan, "Synthesis and Mechanical Analysis of on iron powder of mixed glass fiber reinforced polymer composite Material," *International Research Journal on Advanced Science Hub*, vol. 2, no. 11, pp. 26-29, 2020.
- [21] Mao, H. Abushammala and J., "Waste Iron Filings to Improve the Mechanical and Electrical Properties of Glass Fiber-Reinforced Epoxy (GFRE) Composites," *Journal of Composites Science*, vol. 7, no. 3, p. 90, 2023.
- [22] Ala Tabiei, Wenlong Zhang, "Composite Laminate Delamination Simulation and Experiment: A Review of Recent Development," *Applied Mechanics Reviews*, vol. 70, 2018.
- [23] Runar Unnthorsson, Thomas Philip Runarsson, "NDT METHODS FOR EVALUATING CARBON FIBER COMPOSITES," *Materials Science, Engineering*, 2004.
- [24] Ab Ghani, A.F., Yaakob, M.Y., Salim, M.N., and Mahmud, J., "Hardness Assessment of Hybrid Composite CFRP and GFRP," *Int. J. Recent Technol. Eng.*, vol. 8, no. 1S5, p. 86-93, 2019.
- [25] Srinivasa, C.V. and Bharath, K.N., "Impact and Hardness Properties of Areca Fiber-Epoxy Reinforced Composites," *J. Mater. Environ. Sci.*, vol. 2, p. 351-356, 2011.

- [26] Alshabib, A., Silikas, N., and Watts, D.C., "Hardness and Fracture Toughness of Resin-Composite Materials with and without Fibers," *Dent. Mater. J.*, vol. 19, no. 8, p. 1194–1203, 2019.
- [27] K. Guseinov, S. B. Sapozhnikov & O. A. Kudryavtsev, "Features of Three-Point Bending Tests for Determining Out-of-Plane Shear Modulus of Layered Composites," *Mechanics of Composite Materials*, vol. 58, pp. 155-168, 2022.
- [28] Kim, J.-S., Lee, S.-Y., and Roh, J.-H., "Failure Analysis of Laminated Composites Subjected to Flexural Loadings," *Adv. Compos. Mater.*, vol. 29, no. 3, p. 1–16, 2020.
- [29] Dong, C. and Davies, I.J., "Flexural Strength of Bidirectional Hybrid Epoxy Composites Reinforced by E Glass and T700S Carbon Fibres," *Compos. B Eng.*, vol. 72, p. 65–71, 2014.
- [30] Al-Mosawi, A.I., Rijab, M.A., Abdullah, N., and Mahdi, S., "Flexural Strength of Fiber Reinforced Composite," *Int. J. All Res. Educ. Sci. Methods*, vol. 2, no. 1, 2013.
- [31] Bey, K., Tadjine, K., Khelif, R., Chemami, A., Benamira, M., and Azari, Z., "Mechanical Behavior of Sandwich Composites under Three-Point Bending Fatigue," *Mech. Compos. Mater.*, vol. 50, no. 6, p. 747–756, 2015.
- [32] J. Marszałek, "Span length effect on flexural properties of composite laminate reinforced with a plain weave carbon fiber fabric in a polymer matrix," *Science and Engineering of Composite Materials*, vol. 29, no. 1, pp. 322-334, 2022.
- [33] Paiva, J. M. F. de., Mayer, S., & Rezende, M. C., "Comparison of tensile strength of different carbon fabric reinforced epoxy composites," *Materials Research*, vol. 9, no. 1, p. 83–90, 2006.
- [34] Makeev A, Seon G, Nikishkov Y, Lee E., "Methods for assessment of interlaminar tensile strength of composite materials," *Journal of Composite Materials*, vol. 49, no. 7, pp. 783-794, 2015.
- [35] Armanios, Erian & Bucinell, Ronald & Dale, WW & O'Brien, TK & Chawan, AD & DeMarco, K., "Influence of Specimen Configuration and Size On Composite Transverse Tensile Strength and Scatter Measured Through Flexure Testing," *Journal of Composites Technology & Research*, vol. 25, 2003.
- [36] R. Ruzuqi, "Tensile Strength Analysis of Polymer Composite Materials Fiber Reinforced in The Fiber boat Application," *Journal of Research and Opinion*, vol. 7, no. 8, p. 2763–2769, 2020.
- [37] Ery Diniardi, Nelfiyanti Nelfiyanti, Kisman H Mahmud, Hasan Basri, A I Ramadhan, "Analysis of the Tensile Strength of Composite Material from Fiber Bags," *Journal of Applied Sciences and Advanced Technology*, vol. 2, no. 2, 2019.
- [38] Rahmani H, Najafi SHM, Ashori A., "Mechanical performance of epoxy/carbon fiber laminated composites," *Journal of Reinforced Plastics and Composites*, vol. 33, no. 8, pp. 733-740, 2014.

- [39] Taniguchi, N., Nishiwaki, T., & Kawada, H., "Tensile strength of unidirectional CFRP laminate under high strain rate," *Advanced Composite Materials*, vol. 16, no. 2, p. 167–180, 2007.
- [40] Md. Rashnal Hossain, Md. Aminul Islam, Aart Van Vuurea, Ignaas Verpoest, "Tensile Behavior of Environment Friendly Jute Epoxy Laminated Composite," *Procedia Engineering*, vol. 56, pp. 782-788, 2013.
- [41] S. Ebrahim Moussavi-Torshizi, Soheil Dariushi, Mojtaba Sadighi, Pedram Safarpour, "A study on tensile properties of a novel fiber/metal laminates," *Materials Science and Engineering: A*, vol. 527, no. 18–19, pp. 4920-4925, 2010.
- [42] "Uniform Elongation," World Auto Steel, 2021. [Online]. Available: <https://ahssinsights.org/tag/tensile-testing/>. [Accessed 2024].
- [43] B. Benmokrane, A. H. Ali, H. M. Mohamed, A. ElSafty and A. Manalo, "Laboratory assessment and durability performance of vinyl-ester, polyester, and epoxy glass-FRP bars for concrete structures," *Composites Part B: Engineering*, vol. 114, pp. 163-174, 2017.
- [44] C. L. Soles, F. T. Chang, D. W. Gidley and A. F. Yee, "Contributions of the nanovoid structure to the kinetics of moisture transport in epoxy resins," *Journal of Polymer Science Part B: Polymer Physics*, vol. 38, no. 5, 2000.
- [45] A.-H. I. Mourad, B. M. Abdel-Magid, T. El-Maaddawy and M. E. Grami, "Effect of Seawater and Warm Environment on Glass/Epoxy and Glass/Polyurethane Composites," *Applied Composite Materials*, vol. 17, no. 5, pp. 557-573, 2010.
- [46] M. A. G. Silva, B. S. da Fonseca and H. Biscaia, "On estimates of durability of FRP based on accelerated tests," *Composite Structures*, vol. 116, pp. 377-387, 2014.
- [47] Kaybal H B, Ulus H, Tatar A C, Demir O, "Influence of seawater on mechanical properties of SiO₂-epoxy polymer nanocomposites," *Res. Eng. Struct. Mat*, vol. 5, no. 2, pp. 147-154, 2019.
- [48] Emine Feyza SUKUR, Halil Burak KAYBAL, Hasan ULUS, Ahmet AVC, "Tribological behavior of epoxy nanocomposites under corrosive environment: effect of high performance boron nitride nanoplatelet," *Sigma Journal of Engineering and Natural Sciences*, vol. 41, no. 6, 2023.
- [49] L. Bian, J. Xiao, J. Zeng and S. Xing, "Effects of seawater immersion on water absorption and mechanical properties of GFRP composites," *Journal of Composite Materials*, vol. 46, no. 25, pp. 3151-3162, 2012.
- [50] H. Abrial, D. Kadriadi, A. Rodianus, P. Mastariyanto, Ilhamdi, S. Arief, et al., "Mechanical properties of water hyacinth fibers – polyester composites before and after immersion in water," *Materials & Design*, vol. 58, pp. 125-129, 2014.
- [51] S. Bagherpour, R. Bagheri and A. Saatchi, "Effects of concentrated HCl on the mechanical properties of storage aged fiber glass polyester composite," *Materials & Design*, vol. 30, no. 2, pp. 271-274, 2009.

- [52] A. M. Amaro, P. N. B. Reis, M. A. Neto and C. Louro, "Effects of alkaline and acid solutions on glass/epoxy composites," *Polymer Degradation and Stability*, vol. 98, no. 4, pp. 853-862, 2013.
- [53] N. Mortas, O. Er, P. N. B. Reis and J. A. M. Ferreira, "Effect of corrosive solutions on composites laminates subjected to low velocity impact loading," *Composite Structures*, vol. 108, pp. 205-211, 2014.
- [54] Anastasios P. Vassilopoulos, "The history of fiber-reinforced polymer composite laminate fatigue," *International Journal of Fatigue*, vol. 134, p. 105512, 2020.
- [55] Anyang Wang, Zhongqi Wang, Yang Zhao, Zhengping Chang, Xingmeng Shao, Yonggang Kang, "Fatigue behaviour and failure mechanism of the thin/thick-ply hybrid laminated composite bolted joints," *Composite Structures*, vol. 295, p. 115636, 2022.
- [56] Liaojun Yao and Mingyue Chuai and Jurui Liu and Licheng Guo and Xiangming Chen and R.C. Alderliesten and M. Beyens, "Fatigue delamination behavior in composite laminates at different stress ratios and temperatures," *International Journal of Fatigue*, vol. 175, p. 107830, 2023.
- [57] Ali Mahmoudi, Bijan Mohammadi, Hossein Hosseini-Toudeshky, "Damage behaviour of laminated composites during fatigue loading," *Fatigue & Fracture of Engineering Materials & Structures*, vol. 43, no. 4, pp. 698-710, 2019.
- [58] N.H. Padmaraj, Kini M. Vijaya, Pai Dayananda, "Experimental investigation on fatigue behaviour of glass/epoxy quasi-isotropic laminate composites under different ageing conditions," *International Journal of Fatigue*, vol. 143, p. 105992, 2021.
- [59] Pierre L. Chevalier, Christos Kassapoglou, Zafer Gürdal, "Fatigue behavior of composite laminates with automated fiber placement induced defects- a review," *International Journal of Fatigue*, vol. 140, p. 105775, 2020.
- [60] P. Smith, *Process Piping Design Handbook*, vol. 1, Houston, TX: Gulf Publishing Co, 2007.
- [61] Gwidon, S. and Batchelor, A.W., *Experimental Methods in Tribology*, vol. 44, Cambridge, MA: Academic Press, 2004.
- [62] H. Zhang, *Building Materials in Civil Engineering*, Sawston: Woodhead Publishing, 2011.
- [63] Goh, K.L., Thomas, S., De Silva, R.T., and Aswathi, M.K., *Interfaces in Particle and Fibre Reinforced Composites: Current Perspectives on Polymer, Ceramic, Metal and Extracellular Matrices*, Amsterdam: Elsevier Science & Technology, 2019.
- [64] R. Roy, *A Primer on the Taguchi Method*, Southfield: Society of Manufacturing Engineers, 2010.
- [65] S. Bagherpour, R. Bagheri and A. Saatchi, "Effects of concentrated HCl on the mechanical properties of storage aged fiber glass polyester composite," *Materials & Design*, vol. 30, no. 2, pp. 271-274, 2009.

- [66] A. M. Amaro, P. N. B. Reis, M. A. Neto and C. Louro., "Effects of alkaline and acid solutions on glass/epoxy composites," *Polymer Degradation and Stability*, vol. 98, no. 4, pp. 853-862, 2013.
- [67] Lahlali, D. and Naffakh, Mohammed and Dumon, Michel, "Cure kinetics and modeling of an epoxy resin cross-linked in the presence of two different diamine hardeners," *Polymer Engineering & Science*, vol. 45, no. 12, pp. 1581-1589, 2005.
- [68] G. A. George, G. A. Cash, L. Rintoul, "Cure monitoring of aerospace epoxy resins and prepregs by Fourier transform infrared emission spectroscopy," *Polymer International*, vol. 41, no. 2, pp. 169-182, 1996.
- [69] Voleník, K., Lettner, J., Hanousek, F. et al, "Oxides in plasma-sprayed chromium steel," *Journal of Thermal Spray Technology*, vol. 6, pp. 327-334, 1997.

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- **International:**
 - **Youcef Gheid**, Abdennacer Chemami, Djamel Gaagia, Hamza Aouaichia, “**Effects of Seawater and Acidic Environment on Mechanical Properties of Iron Powder-Loaded Glass-Epoxy Composite Laminates: Experimental and Analytical Investigation**” *Research on Engineering Structures & Materials* 2024; 10(3): 1109-1123
 - **Y. Gheid**, A. Chemami, H. Aouaichia & K. Belkaid “**EFFECT OF FILLER CONTENT AND SIZE ON THE FLEXURAL STRENGTH AND HARDNESS OF PURE IRON POWDER FILLED LAMINATED FIBERGLASS EPOXY COMPOSITES**” *Composites: Mechanics, Computations, Applications* (2024) 15(4):1-18

Communication

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