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REVIEW ARTICLE

A Review on the Edible Bio-Particulates-Filled Polymer Composites

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ABSTRACT:
This literature review is focused on the progress and the uses of bio-particulate reinforcement in epoxy composites with an emphasis on the associated characterization techniques and a broad range of applications. The study begins by explaining the environmental degradation and remediation processes due to the application of convention reinforcement materials. It then goes on to suggest the development of bio-particulate reinforcements as a more sustainable alternative. This study, through a thorough analysis of the current literature, aims to articulate the novel developments and interests associated with the employment of bio-particulate reinforcements in epoxy composites, and the possible characterization strategies as well as potential utilization.

KEYWORDS: Bio-particulate reinforcement, epoxy composites, characterization techniques, mechanical properties, sustainability, applications.

1. Introduction

In recent decades, the global shift towards environmentally sustainable manufacturing processes has catalyzed profound transformations within materials science. Traditionally, the composites industry has relied heavily on synthetic reinforcements, such as glass, carbon, and aramid fibers, embedded within thermosetting or thermoplastic matrices [1]. While these synthetic composites offer exemplary specific strength, high modulus, and excellent thermal stability, they are plagued by severe environmental drawbacks. Their production processes are highly energy-intensive and rely on depleting petroleum reserves. More critically, at the end of their service life, synthetic composites present monumental recycling challenges, often resulting in incineration or accumulation in landfills, thereby exacerbating global pollution.

To address these pressing environmental concerns and promote circular economy principles, researchers have increasingly directed their attention towards natural reinforcements. Early efforts focused primarily on long natural fibers like jute, sisal, flax, and hemp. However, a newer and highly promising frontier has emerged: the utilization of edible bio-particulates and agro-waste powders as filler materials in polymer matrices. These bio-particulates include materials such as walnut shells, peanut shells, eggshells, rice husks, and spent coffee grounds [2].

Unlike long fibers, which often require complex extraction, retting, and weaving processes, bio-particulates are typically derived directly from industrial food waste and agricultural by-products. They require minimal processing—often just washing, drying, and grinding—making them incredibly cost-effective. Furthermore, utilizing these edible wastes solves a dual problem: it reduces the burden on municipal waste management systems while providing a sustainable, renewable, and biodegradable filler for polymer composites [3].

This literature review aims to provide a comprehensive analysis of the progress, properties, and applications of edible bio-particulates reinforced epoxy composites. Epoxy resin is

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chosen as the focal matrix due to its widespread industrial use, excellent adhesion properties, and high mechanical strength, although its inherent brittleness often necessitates the inclusion of toughening fillers. We will explore the chemical composition of various bio-particulates, the fabrication techniques employed, and the critical surface modification strategies necessary to ensure adequate interfacial bonding. Furthermore, we will systematically review the mechanical, thermal, tribological, and morphological characterization of these composites, concluding with a discussion on their potential applications and future research trajectories.

2. Edible Bio-Particulates as Reinforcement

The utilization of edible and agro-waste bio-particulates is driven by their abundant availability, low density, non-abrasive nature (which reduces wear on processing equipment), and eco-friendliness [4]. The physical and mechanical properties of the resulting composite are heavily dependent on the intrinsic chemical composition of the chosen particulate.

2.1. Chemical Composition of Bio-Particulates

Most plant-based bio-particulates are lignocellulosic, meaning they are primarily composed of cellulose, hemicellulose, and lignin.

- **Cellulose:** A crystalline polymer that provides structural strength and stiffness to the particulate. High cellulose content generally correlates with higher composite tensile strength.
- **Hemicellulose:** An amorphous, highly hydrophilic polymer network that binds cellulose microfibrils. It is primarily responsible for the moisture absorption issues in natural composites.
- **Lignin:** A complex, cross-linked phenolic polymer that acts as a natural binder and provides rigidity and thermal stability to the plant structure.

Table 1 outlines the typical chemical compositions of various edible bio-particulates discussed in recent literature.

2.2. Prominent Bio-Particulates in Literature

Walnut Shells: Walnut shells are uniquely hard and possess a high lignin content (up to 50%), which imparts excellent thermal stability and dimensional rigidity. Singh et al. [7] demonstrated that finely ground walnut shell particulates significantly improve the hardness and wear resistance of epoxy composites, making them suitable for abrasive environments.

Peanut Shells: Peanut shells are highly abundant and lightweight. Kumar et al. [6] found that peanut shell powder acts as an effective filler to reduce the overall density of epoxy composites while maintaining acceptable flexural properties, highly desirable for lightweight automotive interior panels.

Eggshells: Unlike plant-based particulates, eggshells are primarily composed of calcium carbonate ($CaCO_3$). Zindani et al. [8] highlighted that eggshell particulates act as bioceramic fillers. Their incorporation drastically increases the micro-hardness and compression strength of epoxy matrices, while also serving as a natural flame retardant.

Rice Husk: Rice husks are unique due to their high silica (SiO_2) content, which remains in the ash after burning. This natural silica provides excellent thermal insulation and hardness. Chen et al. [9] successfully utilized rice husk particulates to enhance the thermal degradation threshold of epoxy resins.

3. Matrix Material: Epoxy Resin

Epoxy resins are a class of reactive prepolymers and polymers containing epoxide groups. For high-performance composite applications, epoxy is the undisputed matrix of choice among thermosetting polymers [10].

3.1. Properties of Epoxy

Epoxy resins undergo a cross-linking process (curing) when mixed with a hardener or curing agent (such as amines or anhydrides). This results in a rigid 3D network that provides:

- Excellent specific strength and stiffness.
- Outstanding adhesion to a wide variety of fillers due to the presence of polar hydroxyl and ether groups.
- Low shrinkage during curing, ensuring dimensional stability of the final molded part.
- High resistance to chemical degradation and moisture.

3.2. Challenges with Epoxy Matrices

The primary limitation of highly cross-linked epoxy is its inherent brittleness and low fracture toughness. Consequently, bare epoxy is highly susceptible to crack propagation under impact loading. The introduction of bio-particulates, if properly bonded, can act as crack arrestors. The particles force the crack to deviate, branch, or pin, thereby absorbing energy and enhancing the impact toughness of the otherwise brittle matrix.

4. Fabrication Techniques

The dispersion and distribution of bio-particulates within the epoxy matrix are critical. Poor dispersion leads to agglomeration, creating severe stress concentration points that initiate premature failure.

Table 1: Typical Chemical Composition of Edible Bio-Particulates (% by weight) [5, 6]

Bio-Particulate	**Cellulose (%)**	**Hemicellulose (%)**	**Lignin (%)**	**Ash/Others (%)**
Walnut Shell	25 - 30	22 - 28	40 - 50	1 - 3
Peanut Shell	35 - 40	15 - 20	25 - 30	5 - 10
Rice Husk	30 - 38	15 - 20	18 - 22	15 - 20 (Silica)
Coffee Grounds	8 - 15	35 - 40	20 - 25	10 - 15 (Lipids)
Coconut Shell	30 - 35	20 - 25	40 - 45	1 - 5

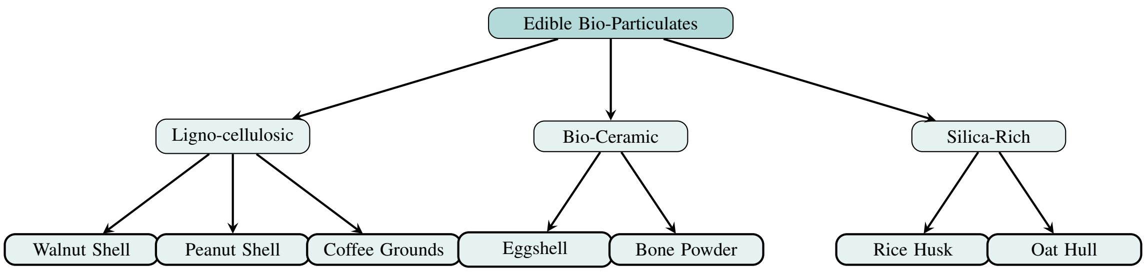

Figure 1: Classification of edible bio-particulates used in polymer composites.

4.1. Open Mold / Hand Lay-Up

The hand lay-up technique is the simplest and oldest composite fabrication method. The bio-particulates are mechanically stirred into the liquid epoxy resin and hardener mixture. The slurry is then poured into a prepared mold and allowed to cure at room temperature. To remove entrapped air bubbles—a major issue when mixing fine powders—the mixture is often degassed in a vacuum desiccator before pouring [11].

4.2. Ultrasonic Dispersion

To prevent the agglomeration of micro- and nano-sized bio-particulates (such as fine eggshell powder), ultrasonic dispersion is employed. High-frequency sound waves generate cavitation bubbles in the resin, which forcefully break apart particulate clusters, ensuring a highly homogenous distribution.

4.3. Compression Molding

For structural applications requiring higher volume fractions of particulates, compression molding is utilized. The particulate-resin mixture is placed in a heated metallic mold. Hydraulic pressure is applied during the curing cycle, which minimizes void content, ensures excellent surface finish, and consolidates the composite structure, yielding superior mechanical properties compared to hand lay-up.

5. Surface Modification Techniques

The fundamental bottleneck in all natural fiber and bio-particulate composites is the inherent incompatibility between the hydrophilic filler and the hydrophobic polymer matrix. Edible bio-particulates contain abundant surface hydroxyl (-OH) groups, which eagerly absorb moisture but repel the non-polar epoxy resin, leading to weak interfacial adhesion [12].

To bridge this gap, chemical surface modifications are mandatory.

5.1. Alkaline Treatment (Mercerization)

Alkaline treatment involves immersing the bio-particulates in an aqueous solution of sodium hydroxide (NaOH), typically between 2% to 10% concentration, for several hours. This process dissolves and removes the amorphous hemicellulose and lignin, as well as surface impurities like waxes and oils.

$$Particulate{-}OH + NaOH \rightarrow Particulate{-}O^{-}Na^{+} + H_2O \tag{1}$$

This treatment dramatically increases the surface roughness and porosity of the particulate, allowing the liquid epoxy resin to mechanically interlock (wet out) with the filler surface.

5.2. Silane Coupling Agents

Silane treatment provides chemical rather than just mechanical bonding. Organosilanes act as molecular bridges. They possess two different reactive functional groups: one end reacts with the hydroxyl groups on the bio-particulate surface, and the other end co-cures with the epoxy matrix. This covalent bonding drastically improves interfacial shear strength, preventing the particulates from being easily pulled out of the matrix under stress.

6. Mechanical Properties Characterization

The viability of bio-particulate composites for engineering applications is primarily assessed through their mechanical properties. The performance is fundamentally governed by the particulate size, weight/volume fraction, and the strength of the filler-matrix interface.

6.1. Tensile Properties

The tensile strength of bio-particulate/epoxy composites is heavily dependent on the filler loading. Studies consistently show that the addition of particulates initially increases or maintains the tensile strength of the epoxy up to an optimal weight percentage (typically between 10% and 20% wt).

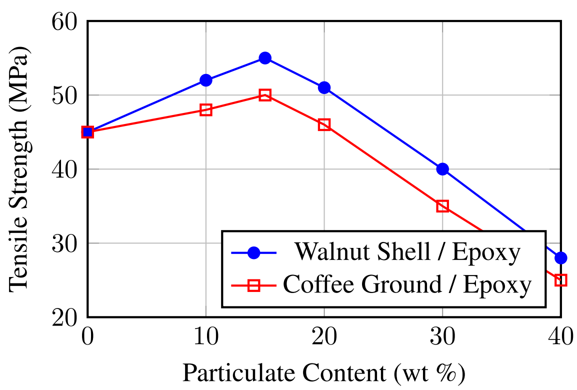

Figure 2: Typical trend of tensile strength variation with bio-particulate loading.

As illustrated in Figure 2, beyond the optimal loading threshold, the tensile strength declines sharply. This is attributed to particulate agglomeration, which creates weak zones where the matrix cannot fully coat the filler, leading to stress concentrations and premature crack initiation [7].

Conversely, the Young's Modulus (stiffness) typically increases linearly with higher particulate loading, as the rigid lignocellulosic or bio-ceramic particles restrict the mobility of the epoxy polymer chains.

6.2. Flexural Properties

Flexural strength, measured via three-point or four-point bending tests, evaluates the composite's resistance to bending and delamination. Bio-particulates excel in enhancing the flexural modulus. For instance, the incorporation of highly rigid eggshell powder significantly improves the bending stiffness of epoxy [8].

6.3. Impact Strength

Impact strength measures the energy absorbed by a material during high-speed fracture (e.g., Izod or Charpy tests). Unfilled epoxy is brittle and shatters easily. The inclusion of bio-particulates introduces crack-pinning and crack-deflection mechanisms. When an advancing crack front hits a hard bio-particulate (like a walnut shell fragment), it must expend additional energy to detour around the particle or pull the particle out of the matrix. This increases the total fracture toughness of the composite.

7. Thermal and Dynamic Mechanical Analysis

Understanding the thermal stability and viscoelastic behavior of these composites is vital for high-temperature applications.

7.1. Thermogravimetric Analysis (TGA)

TGA measures the mass loss of the composite as a function of increasing temperature. Epoxy resin typically degrades completely between 350°C and 450°C.

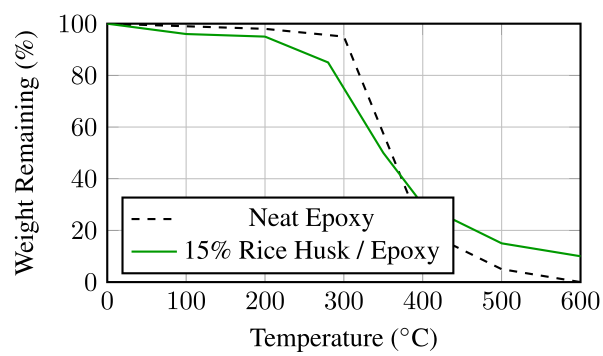

Figure 3: TGA curves demonstrating the thermal degradation of neat epoxy vs. bio-particulate composite.

The inclusion of bio-particulates alters the degradation profile. Lignocellulosic fillers typically introduce an early weight loss step around 250°C due to hemicellulose breakdown. However, silica-rich fillers like rice husk leave a significant char residue at 600°C, acting as a thermal barrier that protects the underlying epoxy matrix [9].

7.2. Dynamic Mechanical Analysis (DMA)

DMA characterizes the storage modulus (E'), loss modulus (E''), and damping factor ($\tan\delta$) under oscillating loads. Rathore et al. [12] demonstrated that agro-waste filled epoxies exhibit a higher storage modulus in the glassy region compared to neat epoxy, confirming effective stress transfer. Furthermore, the glass transition temperature (T_g), indicated by the peak of the $\tan\delta$ curve, often shifts slightly to higher temperatures, indicating that the particulates constrain the segmental motion of the polymer chains.

8. Tribological Properties

Tribology encompasses the study of friction and wear. In engineering applications like brake pads, gears, and flooring, composites must resist abrasive wear.

Hard bio-particulates (e.g., walnut shells, coconut shells) are exceptionally effective solid lubricants and wear resistors. Chandraker et al. [13] investigated the tribological characteristics of bio-waste particulate composites and found that specific wear rates decreased significantly with the addition of treated particulates. The particles act as load-bearing asperities, preventing direct contact between the softer epoxy matrix and the abrasive counter-face. Furthermore, as the particles eventually wear down, they often form a thin, protective tribo-film on the sliding surface, further lowering the coefficient of friction.

9. Morphological Characterization (SEM)

Scanning Electron Microscopy (SEM) is the standard tool for analyzing the fractured surfaces of tested composites. SEM micrographs provide indisputable visual evidence of the failure mechanisms and the quality of the fiber-matrix interface.

In untreated bio-particulate composites, SEM images typically reveal clear "pull-out" holes, deep interfacial gaps, and smooth particulate surfaces, indicating poor adhesion. Conversely, in silane-treated composites, SEM reveals particles heavily coated with the epoxy matrix, rough fracture topographies, and matrix shear yielding, confirming strong covalent bonding and effective load transfer [2].

10. Water Absorption and Swelling Behavior

One of the most persistent drawbacks of utilizing plant-based bio-particulates is their hydrophilic nature. When exposed to humid environments or immersed in water, the free hydroxyl groups in the cellulose structure attract water molecules via hydrogen bonding.

Water absorption in NFRPCs follows Fickian diffusion kinetics. The absorbed water acts as a plasticizer inside the epoxy matrix, disrupting intermolecular hydrogen bonds and reducing the glass transition temperature. More destructively, the water causes the bio-particulates to swell. This swelling exerts tremendous internal stress on the matrix, leading to micro-cracking and complete interfacial debonding, severely degrading the mechanical properties over time [14].

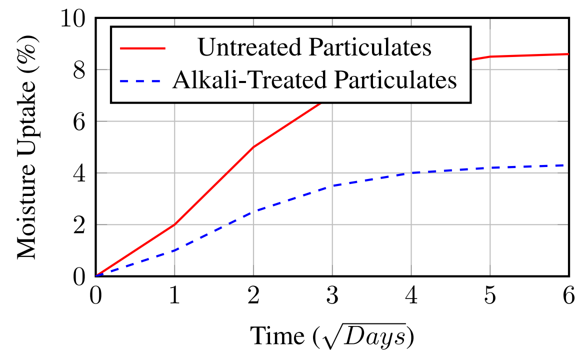

Figure 4: Water absorption kinetics demonstrating Fickian diffusion.

As shown in Figure 4, chemical treatments like alkalization or acetylation are highly effective in mitigating this issue by chemically blocking the hydrophilic sites on the particulates.

10.1. Viscoelastic Properties of Particulate Composites

Dynamic Mechanical Analysis (DMA) not only determines the glass transition temperature (T_g) but also provides deep insights into the viscoelastic properties under varying temperature conditions. In bio-particulate/epoxy composites, the storage modulus (E') is a critical indicator of the stiffness of the material in the glassy region (below T_g). When hard particulates like walnut shells or coffee grounds are uniformly dispersed within the epoxy matrix, they severely restrict the mobility of the epoxy polymer chains. This physical hindrance forces the composite to maintain a much higher storage modulus than the neat resin as the temperature increases towards T_g.

Furthermore, the loss modulus (E'')—which represents the viscous response or energy dissipation of the material—also peaks higher in particulate-filled composites. The internal friction generated between the bio-particulates and the epoxy matrix during cyclic loading contributes significantly to this energy dissipation.

11. Fractography and Interfacial Adhesion

Understanding how a composite fails is just as crucial as knowing when it fails. Fractography, primarily conducted using Scanning Electron Microscopy (SEM), provides a microscopic view of the fracture surface, revealing the intricacies of the fiber-matrix or particulate-matrix interface.

11.1. Brittle vs. Ductile Failure Mechanisms

Neat epoxy typically exhibits a classic brittle fracture, characterized by a smooth, mirror-like topography with distinctive "river lines" pointing toward the crack origin. However, the introduction of edible bio-particulates radically transforms this topography.

When observing the fracture surface of an untreated walnut shell/epoxy composite, one commonly sees massive particulate pull-outs. The presence of deep spherical voids matching the shape of the particulates indicates that the crack propagated directly around the particles rather than through them. This suggests that the interfacial adhesion was weaker than the cohesive strength of the matrix itself, allowing the particles to easily debond and pull out under stress.

11.2. Evidence of Enhanced Adhesion

Conversely, when evaluating silane-treated or alkali-treated particulates, the fractographic evidence shifts entirely. Instead of clean pull-outs, the SEM micrographs typically reveal particles that are fractured cleanly in half, lying perfectly flush with the fracture plane of the matrix. Furthermore, the remaining particles are often coated with a thick layer of epoxy resin. This is the hallmark of excellent interfacial adhesion. It proves that the stress transfer from the epoxy matrix to the bio-particulate was so efficient that the failure occurred cohesively through the particle itself, rather than adhesively at the boundary.

12. Cost Analysis and Commercial Viability

For any novel engineering material to transition from the laboratory to large-scale industrial application, it must demonstrate not only technical superiority or environmental benefits but also strict economic viability.

12.1. Raw Material Economics

The primary economic advantage of edible bio-particulates is their status as industrial or agricultural waste. For example, coffee grounds are produced in millions of tons annually by the beverage industry, and peanut shells are a massive byproduct of the agricultural sector. Acquiring these materials often incurs zero raw material costs, requiring only logistical expenditures for transportation and basic processing (washing, grinding, sieving). In stark contrast, producing synthetic E-glass or carbon fibers involves highly energy-intensive melting, drawing, and carbonization processes, driving up the cost per kilogram significantly.

12.2. Processing Costs

While the raw material is essentially free, the cost of chemical surface treatments (such as silane coupling agents or massive quantities of NaOH) must be factored into the final unit price of the composite. To maximize commercial viability, researchers are actively investigating "green" or physical surface modification techniques—such as plasma treatment, ozone treatment, or microwave-assisted surface roughening—that do not require expensive chemical baths or hazardous waste disposal protocols.

13. Optimization using AI and Machine Learning

As the number of variables in composite design increases—including particulate type, size distribution, weight fraction, treatment duration, and epoxy curing cycles—the traditional "trial and error" experimental approach becomes excessively time-consuming and prohibitively expensive.

13.1. Predictive Modeling

Recent advancements have seen the successful integration of Artificial Intelligence (AI) and Machine Learning (ML) algorithms into the design of bio-particulate composites. By feeding large datasets of historical experimental results into models like Artificial Neural Networks (ANN), Random Forests, or Support Vector Regression (SVR), researchers can accurately predict the ultimate tensile strength, flexural modulus, and wear rates of new, untested composite formulations.

For instance, an ANN model can map the highly nonlinear relationship between the percentage of silane coupling agent used and the resulting impact toughness of a rice husk/epoxy composite. By navigating this multidimensional parameter space computationally, engineers can pinpoint the exact formulation that yields the highest performance-to-cost ratio, thereby drastically accelerating the commercialization pipeline of these sustainable materials.

14. Applications of Bio-Particulate Epoxy Composites

The unique balance of low density, adequate mechanical strength, acoustic damping, and eco-friendliness makes these composites attractive across several industrial sectors.

Table 2: Potential Applications of Bio-Particulate Composites

Sector	Specific Applications
Automotive	Interior door panels, dashboard components, parcel shelves, sound-damping engine covers.
Construction	Non-load bearing partition walls, acoustic ceiling tiles, decking, and faux-wood flooring.
Packaging	Biodegradable rigid packaging enclosures, shock-absorbing pallets.
Consumer Goods	Furniture components, sporting goods, tool handles, protective casings.

The automotive sector is the primary driver for this technology, utilizing the low density of agro-waste composites to achieve lightweighting goals, which directly translates to improved fuel efficiency and lower emissions.

15. Future Challenges and Perspectives

While the promise of edible bio-particulate composites is vast, significant research gaps must be addressed to facilitate large-scale commercialization:

1. **Standardization of Raw Materials:** Unlike synthetic fibers, agricultural waste possesses high inherent variability depending on the geographic location, climate, and soil conditions where the plant was grown. Developing standardized extraction and grading protocols is essential for industrial quality control.

2. **Fire Retardancy:** Lignocellulosic materials are highly flammable. While some particulates like eggshell provide natural resistance, intensive research is needed into eco-friendly, halogen-free fire retardant coatings to meet strict automotive and aerospace safety standards.

3. **Long-Term Environmental Degradation:** While biodegradability is a feature, premature degradation is a flaw. Comprehensive life-cycle assessments (LCA) and long-term weathering studies (UV exposure, thermal cycling) are required to accurately predict the service life of these composites in outdoor environments.

4. **Advanced Hybridization:** Exploring the synergy between micro-scale bio-particulates and nano-fillers (e.g., graphene, nano-clays) to create hierarchical composites that offer the best of both worlds—sustainability and ultra-high performance.

16. Conclusion

The integration of edible bio-particulates and agro-industrial waste as reinforcing fillers in epoxy polymer matrices represents a highly significant paradigm shift toward sustainable materials engineering. This comprehensive review has detailed the progression from traditional synthetic reinforcements to eco-friendly alternatives such as walnut shells, peanut shells, rice husks, and eggshells.

Through detailed analysis of current literature, it is evident that these bio-particulates are not merely cheap diluents; when properly processed, they actively enhance specific properties of the epoxy matrix. Hard particulates like walnut and coconut shells drastically improve wear resistance and micro-hardness, while lightweight fillers like peanut shells aid in density reduction for automotive applications.

However, the realization of these benefits hinges entirely on overcoming the hydrophilic-hydrophobic mismatch between the filler and the matrix. Surface modification techniques—particularly alkaline and silane treatments—are absolutely mandatory to ensure strong interfacial bonding, prevent catastrophic moisture absorption, and enable effective load transfer.

While challenges regarding natural variability, fire retardancy, and long-term outdoor durability remain, the economic and environmental imperatives driving this research are undeniable. The utilization of bio-particulate composites offers a closed-loop, circular economy approach: converting waste into high-value structural materials. Continued interdisciplinary research focusing on advanced hybridization, nano-reinforcement, and automated manufacturing processes will undoubtedly cement the role of bio-particulate polymer composites as foundational materials in the sustainable industrial landscape of the future.

16. Acknowledgement

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