

Comprehensive Investigation of the Mechanical, Thermal, and Morphological Properties of Hemp-Derived Graphene Nanosheets in Epoxy and Bio-Epoxy Composite Systems

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Abstract

Background: While the exceptional electrochemical properties of hemp-bast-fiber-derived carbon nanosheets for energy storage applications are established, a significant research gap exists concerning their performance as a reinforcing agent in polymer composites. The mechanical, thermal, and morphological properties of these sustainable, low-cost nanomaterials have not been systematically investigated.

Objective: This study outlines a comprehensive experimental plan to synthesize and characterize hemp-derived graphene nanosheets (HGNs) and to evaluate their efficacy as a reinforcing filler in both conventional petroleum-based epoxy and sustainable bio-epoxy composite systems. The primary goal is to test the hypothesis that HGNs can significantly enhance the thermomechanical properties of these polymer matrices.

Methods: A detailed Standard Operating Procedure (SOP) is presented for a two-stage process to synthesize HGNs via hydrothermal carbonization followed by chemical activation. The research plan includes the fabrication of composite samples with varying weight percentages of HGNs (0.1%, 0.5%, 1.0%) in both DGEBA epoxy and epoxidized linseed oil (ELO) bio-epoxy matrices. A suite of characterization techniques is proposed, including tensile testing (ASTM D638), flexural testing (ASTM D790), dynamic mechanical analysis (DMA), thermogravimetric analysis (TGA), and scanning electron microscopy (SEM) to assess the mechanical performance, viscoelastic behavior, thermal stability, and filler-matrix morphology.

Anticipated Outcomes: It is hypothesized that the incorporation of HGNs will lead to a dose-dependent increase in tensile strength, Young's modulus, storage modulus, and thermal degradation temperature for both composite systems. This paper serves as a complete pre-experimental protocol, with placeholders provided in the Results and Discussion sections for the insertion and interpretation of empirical data. The findings from this proposed work are expected to validate HGNs as a viable, green alternative to conventional graphene for developing high-performance, sustainable composite materials.

1 Introduction

The quest for advanced materials that are both high-performance and environmentally sustainable is a defining challenge of 21st-century materials science. Graphene, a two-dimensional allotrope of carbon, has garnered immense interest due to its extraordinary mechanical, thermal, and electrical properties [1]. However, its production through methods like chemical vapor deposition or exfoliation of graphite remains costly and energy-intensive, limiting its widespread application. This has spurred research into alternative, sustainable sources for graphene-like materials.

One of the most promising avenues is the conversion of biomass into carbon nanomaterials. In a seminal work, Mitlin et al. demonstrated that hemp bast fiber, a low-cost agricultural byproduct, could be converted into unique carbon nanosheets with exceptional electrochemical performance, rivaling that of commercial graphene in supercapacitor applications [2]. Their work focused exclusively on the electrical properties and the material’s suitability for energy storage. While groundbreaking, this study left a critical knowledge gap: the mechanical and thermal properties of these hemp-derived graphene nanosheets (HGNs) remain uncharacterized. Furthermore, their potential as a reinforcing agent in polymer composites—a major application area for traditional graphene—has not been explored.

Polymer composites, particularly those using epoxy resins, are ubiquitous in aerospace, automotive, and civil engineering due to their high strength-to-weight ratio and chemical resistance [3]. The incorporation of nanofillers like graphene has been shown to significantly enhance their mechanical and thermal performance [4]. Concurrently, there is a strong industrial and academic push towards “green composites,” which utilize bio-based resins, such as epoxidized vegetable oils, to reduce reliance on petroleum feedstocks [5].

This research aims to bridge the gap identified in the literature by conducting the first systematic investigation into the mechanical and thermal properties of HGNs as a reinforcement phase in both a conventional petroleum-based epoxy and a sustainable bio-epoxy.

1.1 Research Questions

Based on the identified research gap, this study will address the following FINER (Feasible, Interesting, Novel, Ethical, Relevant) questions:

1. What are the quantitative effects of incorporating varying weight percentages (0.1%, 0.5%, 1.0%) of HGNs on the tensile strength, Young’s modulus, and flexural strength of a standard DGEBA epoxy composite?
2. How does the inclusion of HGNs influence the thermomechanical properties (storage modulus, glass transition temperature) and thermal stability (degradation temperature) of epoxy and bio-epoxy composites?
3. Is HGN an effective reinforcing agent in a sustainable bio-epoxy matrix (e.g., epoxidized linseed oil), and how does its performance compare to its role in a conventional epoxy

system?

1.2 Hypotheses

To address these questions, the following primary alternative hypothesis (H_a) and corresponding null hypothesis (H_0) have been formulated:

- **H_a (Alternative Hypothesis):** The addition of hemp-derived graphene nanosheets to epoxy and bio-epoxy resins will result in a statistically significant, dose-dependent improvement in the material’s mechanical properties (tensile strength, modulus) and thermal properties (storage modulus, thermal stability) compared to the neat polymer matrices.
- **H_0 (Null Hypothesis):** The addition of hemp-derived graphene nanosheets will have no statistically significant effect on the mechanical and thermal properties of either the epoxy or bio-epoxy matrices.

This work will provide a comprehensive evaluation of HGNs as a multifunctional, sustainable reinforcement material, potentially unlocking new applications for this promising biomaterial in the field of high-performance composites.

2 Materials and Methods

This section details the Standard Operating Procedure (SOP) for the synthesis of HGNs, fabrication of composites, and the characterization techniques to be employed.

2.1 Materials and Reagents

- **Hemp Bast Fiber:** Sourced locally (e.g., from TerrAscend Canada).
- **Potassium Hydroxide (KOH):** ACS reagent grade, $\geq 85\%$.
- **Hydrochloric Acid (HCl):** 37%, ACS reagent grade.
- **Epoxy Resin:** Diglycidyl ether of bisphenol A (DGEBA), (e.g., Epon 828).
- **Epoxy Curing Agent:** Jeffamine D-230 or similar polyetheramine.
- **Bio-Epoxy Resin:** Epoxidized linseed oil (ELO).
- **Bio-Epoxy Curing Agent:** Anhydride-based or bio-based amine.
- **Solvent:** Acetone, ACS reagent grade.
- Deionized (DI) Water.

2.2 Equipment

- Hydrothermal synthesis reactor (autoclave)
- Tube furnace with inert gas (Ar, N₂) supply
- Probe sonicator
- High-speed mechanical stirrer (shear mixer)
- Vacuum oven
- Universal Testing Machine (UTM) with tensile and flexural grips
- Dynamic Mechanical Analyzer (DMA)
- Thermogravimetric Analyzer (TGA)
- Scanning Electron Microscope (SEM)
- Analytical balance, glassware, and standard laboratory safety equipment

2.3 Experimental Variables

- **Independent Variables:**

1. Nanofiller concentration (wt% HGNs): 0% (control), 0.1%, 0.5%, 1.0%.
2. Polymer matrix type: DGEBA Epoxy, ELO Bio-Epoxy.

- **Dependent Variables:**

1. Mechanical: Tensile strength (MPa), Young's modulus (GPa), Elongation at break (%), Flexural strength (MPa).
2. Thermomechanical: Storage modulus (E'), Loss modulus (E''), Tan delta, Glass transition temperature (T_g) (°C).
3. Thermal Stability: Onset degradation temperature (T_{onset}) (°C).
4. Morphology: Dispersion quality, fracture surface features.

- **Controlled Variables:**

1. Curing schedule (time and temperature) for each resin system.
2. Sample geometry and dimensions (per ASTM standards).
3. Testing parameters (e.g., strain rate, temperature ramps).
4. HGN synthesis parameters.

2.4 Step-by-Step Procedure

2.4.1 Part 1: Synthesis of Hemp-Derived Graphene Nanosheets (HGNs)

1. Clean and dry raw hemp bast fiber.
2. Subject the fibers to hydrothermal carbonization in a sealed autoclave at 180 °C for 8 hours to produce hydrochar.
3. Wash the resulting hydrochar with DI water until a neutral pH is achieved and dry overnight in an oven.
4. Chemically activate the hydrochar by impregnating it with a KOH solution (1:4 mass ratio of char to KOH).
5. Dry the mixture and then pyrolyze it in a tube furnace at 800 °C under an inert Argon atmosphere for 1 hour.
6. After cooling, wash the activated carbon extensively with 0.1M HCl to remove inorganic salts, followed by DI water until the filtrate is neutral.
7. Dry the final HGN powder in a vacuum oven at 80 °C.
8. Characterize the HGN powder using SEM for morphology and Raman spectroscopy for structural quality (D/G band ratio).

2.4.2 Part 2: Fabrication of Polymer Composites

1. For each composite formulation, calculate the required mass of HGNs and resin.
2. Disperse the desired amount of HGN powder into the epoxy (or bio-epoxy) resin using a combination of shear mixing and probe sonication in an acetone solvent carrier to ensure homogeneous dispersion and prevent agglomeration.
3. Evaporate the acetone using a rotary evaporator or gentle heating in a fume hood.
4. Add the stoichiometric amount of curing agent to the HGN-resin mixture and mix thoroughly but gently to avoid introducing excessive air bubbles.
5. Place the mixture in a vacuum chamber to degas and remove any entrapped air.
6. Pour the degassed mixture into pre-heated, release-agent-coated silicone molds conforming to ASTM standard geometries (e.g., dumbbell shape for ASTM D638).
7. Cure the samples in an oven according to the manufacturer's recommended schedule (e.g., 80 °C for 2 hours followed by 125 °C for 3 hours for a standard epoxy). A separate curing profile will be used for the bio-epoxy system.

8. After curing, allow the samples to cool slowly to room temperature to minimize residual thermal stresses.
9. De-mold the samples and post-cure if required. Lightly sand the edges to remove any flash.

2.4.3 Part 3: Characterization and Testing

1. **Mechanical Testing:** Conduct tensile tests according to ASTM D638 and three-point bending (flexural) tests according to ASTM D790 using the UTM. Test a minimum of five samples per formulation to ensure statistical validity.
2. **Dynamic Mechanical Analysis (DMA):** Analyze samples using a DMA instrument in a single-cantilever or three-point bending mode. Sweep the temperature from room temperature to above the glass transition temperature (e.g., 30 to 180 °C) at a heating rate of 3 °C/min to determine storage modulus, loss modulus, and T_g .
3. **Thermogravimetric Analysis (TGA):** Evaluate the thermal stability of the composites by heating a small sample from room temperature to 700 °C at 10 °C/min under a nitrogen atmosphere.
4. **Scanning Electron Microscopy (SEM):** Examine the cryo-fractured surfaces of the tested composite samples to assess the quality of HGN dispersion, the nature of the fracture (brittle/ductile), and the interfacial adhesion between the filler and the matrix.

2.5 Data Collection and Statistical Analysis Plan

Data for all tests will be recorded in organized spreadsheets. The primary statistical tool will be a two-way Analysis of Variance (ANOVA) to determine the significance of the independent variables (HGN concentration and matrix type) on the various dependent properties. If significant effects are found ($p \leq 0.05$), a Tukey's Honestly Significant Difference (HSD) post-hoc test will be performed to identify which specific groups differ from one another. Results will be presented as mean $\pm$ standard deviation.

3 Results

This section is a template with placeholders. It must be populated with the empirical data collected during the experimental execution phase (Step 8 of the workflow).

3.1 Mechanical Properties

The mechanical properties of the neat epoxy, neat bio-epoxy, and their respective HGN-reinforced composites were evaluated. The results for tensile strength, Young's modulus, and flexural strength are summarized in Table 1 and visualized in Figures 1-3.

- [PLACEHOLDER FOR RESEARCHER: Insert Table 1 here. The table should summarize the mean and standard deviation for tensile strength, Young’s modulus, and flexural strength for all 8 formulations (2 matrices x 4 concentrations).]
- [PLACEHOLDER FOR RESEARCHER: Insert Figure 1 (Bar chart of Tensile Strength vs. HGN wt%), Figure 2 (Bar chart of Young’s Modulus vs. HGN wt%), and Figure 3 (Bar chart of Flexural Strength vs. HGN wt%). Each figure should show data for both epoxy and bio-epoxy systems with error bars.]

A two-way ANOVA was performed on the data. A statistically significant effect of HGN loading on tensile strength was observed ($F(df1, df2) = X.XX$, $p < 0.05$).

- [PLACEHOLDER FOR RESEARCHER: Objectively describe the trends observed in the figures and table. For example: "The addition of HGNS resulted in a monotonic increase in tensile strength for the DGEBA epoxy system, with the 1.0 wt% loading showing a X% improvement over the neat epoxy. Report the specific p-values from the Tukey post-hoc test comparing the control group to the reinforced groups."]

3.2 Thermomechanical and Thermal Properties

DMA was used to investigate the viscoelastic behavior of the composites. The storage modulus (E') at room temperature and the glass transition temperature (T_g , determined from the peak of the $\tan \delta$ curve) are presented in Figure 4.

- [PLACEHOLDER FOR RESEARCHER: Insert Figure 4 here (Bar chart showing Storage Modulus and T_g for all formulations).]
- [PLACEHOLDER FOR RESEARCHER: Describe the DMA results. For example: "The storage modulus below the T_g increased with HGN loading, indicating a stiffening effect. The T_g of the composites showed a slight increase/decrease of X°C at 1.0 wt% loading compared to the neat resin."]

The thermal stability was assessed using TGA, with the onset degradation temperature (T_{onset}) serving as the primary metric.

- [PLACEHOLDER FOR RESEARCHER: Insert Figure 5 here (Overlaid TGA curves for all formulations) and a corresponding table for T_{onset} values.]
- [PLACEHOLDER FOR RESEARCHER: Describe the TGA results. For example: "The incorporation of HGNS enhanced the thermal stability of both matrices, with the T_{onset} for the 1.0 wt% epoxy composite increasing by Y°C compared to the neat epoxy."]

3.3 Morphological Analysis

SEM was used to examine the cryo-fractured surfaces of the composite samples to understand the structure-property relationships.

- [PLACEHOLDER FOR RESEARCHER: Insert Figure 6 here. This should be a multi-panel figure showing representative SEM micrographs of the fracture surfaces of the neat epoxy and the 1.0 wt% HGN-epoxy composite. Use arrows to highlight key features.]
- [PLACEHOLDER FOR RESEARCHER: Describe the SEM images. For example: "The fracture surface of the neat epoxy (Fig. 6a) exhibits smooth, river-like patterns characteristic of brittle fracture. In contrast, the 1.0 wt% HGN composite (Fig. 6b) shows a much rougher, more tortuous fracture path, indicating effective crack deflection by the nanosheets. At higher magnification, individual HGNS can be seen pulled out from the matrix, suggesting good load transfer."]

4 Discussion

This section is a template with placeholders for your interpretation and analysis, connecting your results to the initial hypotheses and existing literature.

The results of this study must be analyzed to determine if they support the alternative hypothesis (H_a).

- [PLACEHOLDER FOR RESEARCHER: Begin by restating your main finding and whether it supports or refutes your hypothesis. For example: "The experimental data strongly support the alternative hypothesis that the addition of hemp-derived graphene nanosheets significantly enhances the thermomechanical properties of both epoxy and bio-epoxy systems."]
- [PLACEHOLDER FOR RESEARCHER: Interpret the mechanical property enhancements. Why did they improve? Relate this to the SEM findings. For example: "The observed increase in tensile strength and modulus can be attributed to the effective transfer of applied stress from the polymer matrix to the high-stiffness HGNS. The rough fracture surfaces seen in SEM imaging provide clear evidence that the nanosheets act as obstacles to crack propagation, thereby increasing the energy required for fracture."]
- [PLACEHOLDER FOR RESEARCHER: Compare the performance in the epoxy vs. the bio-epoxy system. Was one better than the other? Speculate on why. For example: "Interestingly, the percentage improvement in mechanical properties was more pronounced in the ELO bio-epoxy system. This

could be due to better interfacial adhesion resulting from potential chemical interactions between the oxygen-containing functional groups on the HGN surface and the ester groups in the ELO matrix.”]

- [PLACEHOLDER FOR RESEARCHER: Discuss the thermal analysis results. What is the mechanism for the improved thermal stability and storage modulus? For example: ”The increase in T_g and T_{onset} is likely due to the ’nanoconfinement effect,’ where the presence of the HGNS physically restricts the mobility of polymer chains, requiring more thermal energy to induce segmental motion (glass transition) and bond scission (degradation).”]]
- [PLACEHOLDER FOR RESEARCHER: Acknowledge the limitations of your study. What were the potential sources of error or confounding variables? For example: ”A key limitation of this work is the potential for HGN agglomeration at higher concentrations. Although sonication was used, small aggregates might still exist, acting as stress concentrators rather than reinforcing agents. Future work should explore the use of surfactants or functionalization of the HGNS to improve dispersion further. Additionally, any deviations from the SOP noted during the experiment should be discussed here.”]

5 Conclusion

This section is a template for you to summarize the project’s key takeaways and suggest future work.

- [PLACEHOLDER FOR RESEARCHER: Summarize the main finding in one or two clear sentences. For example: ”This study successfully demonstrated that hemp-derived graphene nanosheets, synthesized from agricultural waste, are a highly effective multifunctional reinforcing agent for both petroleum-based and bio-based epoxy composites.”]
- [PLACEHOLDER FOR RESEARCHER: State the broader implications of your work. Why is this important for the field of materials science or for industry? For example: ”This research validates a low-cost, sustainable pathway for producing high-performance, lightweight composite materials. The use of HGNS in bio-epoxies, in particular, represents a significant step towards developing fully ’green’ composites with engineering-grade performance, with potential applications in automotive interiors, sporting goods, and consumer electronics.”]
- [PLACEHOLDER FOR RESEARCHER: Propose specific next steps for future research based on your findings and the limitations you identified. For example: ”Future research should focus on optimizing the HGN dispersion

and interfacial adhesion through surface functionalization. Furthermore, the electrical conductivity and EMI shielding effectiveness of these composites should be investigated to explore their potential for multifunctional applications. Long-term durability studies, including fatigue and hygrothermal aging tests, are also necessary before these materials can be considered for structural applications.”]

References

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- [3] H. P. S. A. Khalil et al., "A review on nanocellulosic polymer composites: properties, processing and applications," *Express Polymer Letters*, vol. 11, no. 6, pp. 445–463, 2017. DOI: [10.3144/expresspolymlett.2017.43](https://doi.org/10.3144/expresspolymlett.2017.43).
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- [5] I. Javni, W. Zhang, and Z. S. Petrović, "Biobased polyurethanes from vegetable oils," *Journal of Polymers and the Environment*, vol. 11, pp. 123–129, 2003. DOI: [10.1023/A:1025800007353](https://doi.org/10.1023/A:1025800007353).