

Experimental Protocol: Hemp Nanosheets in Epoxy

Systematic Empirical Evaluation of Hemp-Derived Carbon Nanosheets (HDCNS) in Standard and Bio-Epoxy Matrices

Objective: Bridge the transition from theoretical framework to concrete empirical data by establishing a rigorous testing blueprint for compounding hemp-derived carbon nanosheets into thermoset and bio-based epoxy networks.

1. Research Rationale & Scope

While generic hemp fibers and biomass biochar have been widely researched, embedding exfoliated hemp-derived carbon nanosheets (HDCNS) into epoxy matrices represents a frontier in sustainable high-performance polymers. This protocol outlines material preparation, dispersion methodologies, and standard mechanical/thermal characterization steps to validate or refute performance hypotheses.

2. Phase 1: Material Selection & Pre-Treatment

Matrix Formulations

- Standard Epoxy:** DGEBA (Diglycidyl Ether of Bisphenol A) with amine hardener baseline.
- Bio-Epoxy Variant:** Epoxidized plant-oil blends or lignin-integrated bio-resins to maximize sustainability index.

Filler Processing

- HDCNS Synthesis:** Hydrothermal carbonization of hemp bast fibers followed by chemical activation (KOH).
- Surface Functionalization:** Silane coupling agents (e.g., GPTMS) to improve interfacial bond strength.

3. Phase 2: Loading & Dispersion Strategy

Agglomeration prevention is critical due to van der Waals forces acting on nanosheets. Batches are formulated at strict mass fractions: 0.5%, 1.0%, 2.0%, and 5.0% wt to identify percolation thresholds before structural degradation occurs.

Batch ID	Matrix Type	HDCNS Loading (wt%)	Processing Protocol
B-00 (Control)	DGEBA / Amine	0.0%	Standard mechanical mixing
H-05	DGEBA / Amine	0.5%	High-shear + Probe Sonication
H-10	DGEBA / Amine	1.0%	High-shear + Probe Sonication
E-05	Bio-Epoxy Blend	0.5%	High-shear + Probe Sonication

4. Compounding & Curing Workflow

The step-by-step physical processing sequence ensures uniform dispersion and void-free casting:

- **High-Shear Mixing:** Disperse HDCNS into liquid resin (Part A) at 3,000 RPM for 30 minutes using a Cowles blade disperser.
- **Ultrasonication:** Subject the pre-mixture to probe ultrasonication (20 kHz) in an ice-bath to prevent premature exotherm and break down remaining agglomerates.
- **Vacuum Degassing:** Place mixture under vacuum (-0.1 MPa) at room temperature for 45 minutes to evacuate entrapped micro-voids.
- **Stoichiometric Curing:** Add hardener (Part B), cast into precision PTFE molds, cure at room temperature for 24 hours, followed by post-curing at 80°C for 4 hours.

5. Phase 3: Characterization & Testing Matrix

Empirical validation relies on standardized ASTM testing protocols to quantify mechanical reinforcement and thermal property shifts:

Property Tested	Standard Method	Key Metrics Evaluated
Tensile Properties	ASTM D638	Young's Modulus, Tensile Strength, Elongation at Break
Flexural Rigidity	ASTM D790	Three-point bending strength and flexural modulus
Impact Toughness	ASTM D256	Izod impact energy absorption capacity
Thermal Analysis	ASTM E1312 (DSC) / ASTM E1131 (TGA)	Glass transition temperature (T_g), char yield, degradation threshold
Microstructure	SEM Imaging	Fractography, dispersion uniformity, crack deflection analysis

6. Success Criteria & Deliverables

Validation Target: A successful formulation will demonstrate a $\geq 15\%$ increase in tensile modulus and a noticeable upward shift in char yield without severe viscosity spikes or catastrophic particle agglomeration visible under SEM.