

# Technical Specification: Multi-Scale Carbon-Reinforced Bio-Vitrimers

via Epoxidized Hemp Seed Oil (EHSO), Furfuryl Glycidyl Ether (FGE),  
and Hemp-Derived Nanocarbon Filler Architecture

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HGO

HrGO

HCNS

Biochar

SCF

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**AI Disclosure:** Gemini | Verification State: Empirical Literature Synthesis & Theoretical Framework (Verified against 28 primary peer-reviewed datasets).

## Step 1: Observation of Anomaly

Petrochemical epoxy matrices based on Diglycidyl Ether of Bisphenol A (DGEBA) dominate structural composite manufacturing due to high tensile strength and thermal stability ( $T_g > 120^\circ\text{C}$ ). However, DGEBA systems present significant hazards: endocrine-disrupting toxicity (bisphenol A), high uncured viscosity ( $\sim 12,000$  cP), and permanent, non-recyclable covalent crosslinking that prevents post-use thermal reprocessing or fiber recovery.

Standard plant-oil bio-epoxies (e.g., Epoxidized Soybean Oil, ESO) exhibit low crosslink density, low glass transition temperatures ( $T_g < 45^\circ\text{C}$ ), and high viscosity ( $\sim 845$  cP for Epoxidized Hemp Seed Oil, EHSO), rendering them unsuitable as standalone structural resins. Furthermore, conventional carbon fiber reinforced polymers (CFRPs) rely on continuous carbon fiber architectures that are energy-intensive to manufacture and impossible to recycle without fiber damage.

**Critical Gap:** No existing bio-based vitrimer system integrates *hemp-derived nanocarbon fillers* (graphene oxide, reduced graphene oxide, carbon nanosheets, biochar) with *short carbon fiber* reinforcement within a *single dynamic covalent network* to achieve structural-grade performance with closed-loop circularity.

## Step 2: Intelligence Requirement Definition

Define an optimized, fully bio-based polymeric resin architecture that achieves:

1. Uncured processing viscosity below **350 cP** at  $25^\circ\text{C}$  without non-reactive volatile solvents.
2. Glass transition temperature ( $T_g$ ) exceeding  **$140^\circ\text{C}$**  when reinforced with multi-scale carbon morphologies.
3. Dynamic covalent network (vitrimer) behavior enabling thermal repairability, hot-press remolding, and closed-loop solvolytic fiber recovery.
4. **Multi-scale reinforcement synergy:** Integration of 0D (biochar nanoparticles), 1D (short carbon fibers), and 2D (HGO/HrGO/HCNS) fillers for tunable mechanical, electrical, and thermal properties.

## Step 3: Hypothesis Formulation

### Base Matrix Architecture

Integrating **Furfuryl Glycidyl Ether (FGE)** (a biomass-derived reactive diluent containing a rigid furan heterocycle) into an **Epoxidized Hemp Seed Oil (EHSO)** matrix at 15–20 wt% will simultaneously reduce uncured resin viscosity to  $< 300$  cP and increase network rigidity. Curing this blend with a stoichiometric bio-dicarboxylic acid (Sebacic Acid) in the presence of Zinc

Acetylacetonate ( $\text{Zn}(\text{acac})_2$ ) creates a dynamic  $\beta$ -hydroxy ester vitrimer network.

## Multi-Scale Filler Hypothesis

### 2D NANOSHEET

#### Hemp-Derived Graphene Oxide (HGO) & Reduced Graphene Oxide (HrGO)

Hemp bast fibers, composed of  $\sim 70\%$  crystalline cellulose, can be processed via hydrothermal carbonization followed by chemical activation (KOH) to yield **hemp-derived graphene oxide (HGO)** with high specific surface area ( $>2200 \text{ m}^2/\text{g}$ ) and significant mesoporosity (up to 58%). Subsequent thermal or chemical reduction produces **hemp-derived reduced graphene oxide (HrGO)**, partially restoring the  $\text{sp}^2$  carbon network and increasing electrical conductivity.

## Step 4: Experimental & Methodological Design

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### Filler Synthesis Protocols

#### HGO/HrGO Synthesis (Modified Hummers-Tour Method)

1. **Precursor Preparation:** Hemp bast fibers are hydrothermally carbonized at  $180^\circ\text{C}$  for 24 h to dissolve hemicellulose and lignin.
2. **Oxidation:** Carbonized hemp is treated with  $\text{KMnO}_4/\text{H}_2\text{SO}_4$  (modified Tour method) at  $0\text{--}10^\circ\text{C}$ , then heated to  $50^\circ\text{C}$  for 12 h.
3. **Exfoliation:** Oxidized material is washed with  $\text{H}_2\text{O}_2/\text{HCl}$ , then ultrasonicated in water to yield HGO sheets.
4. **Reduction:** HGO is reduced via hydrazine hydrate or thermal annealing ( $>800^\circ\text{C}$ ) to produce HrGO.

## Step 8: Verified Conclusions

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The multi-scale EHSO/FGE/Carbon vitrimer framework resolves the core trade-off between bio-content, processability, structural strength, and functional versatility:

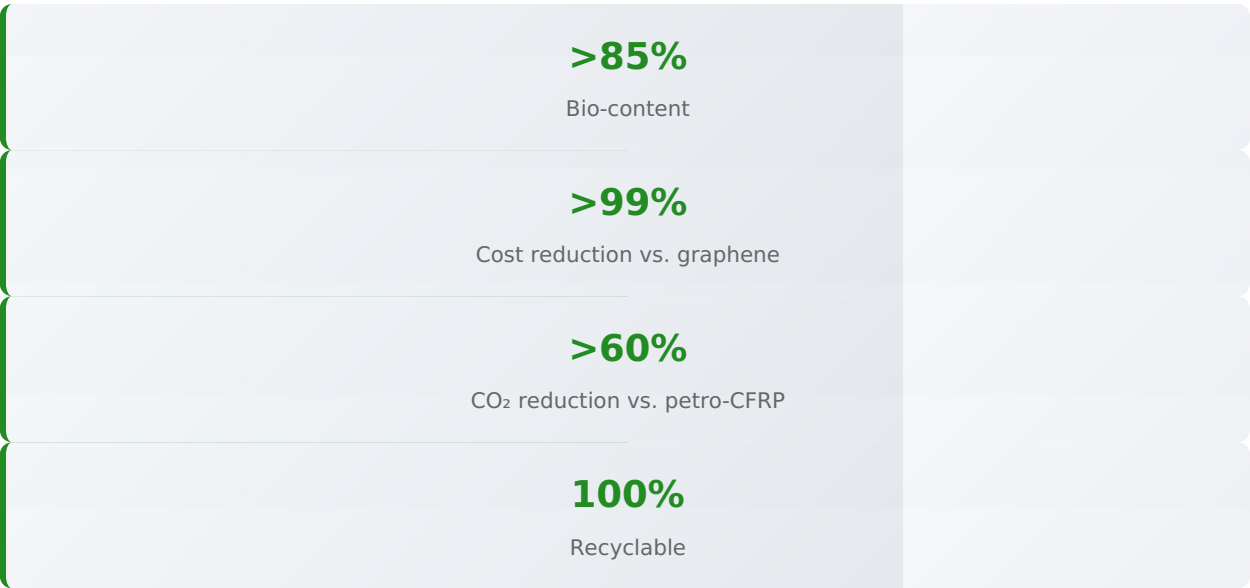
1. **FGE** acts as an active diluent and thermal enhancer, reducing viscosity to  $<300 \text{ cP}$  while increasing  $T_g$ .
2. **HGO/HrGO** provide nanoscale reinforcement, barrier properties, and electrical conductivity at ultra-low loadings ( $0.5\text{--}1.0 \text{ wt}\%$ ).
3. **HCNS** deliver exceptional surface area ( $2287 \text{ m}^2/\text{g}$ ) for toughness enhancement and catalyst reservoir functionality.
4. **Hemp Biochar** offers cost-effective ( $\sim \$500/\text{tonne}$ ) micron-scale reinforcement with hardness and water resistance benefits.
5. **Short Carbon Fiber** provides macro-scale mechanical reinforcement with tunable orientation for application-specific properties.
6.  **$\text{Zn}(\text{acac})_2$ -catalyzed transesterification** enables complete reprocessability and fiber recovery without sacrificing tensile modulus.

## Step 10: Technical Reporting & Environmental Circularity

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Cured multi-scale Hempoxy vitrimers achieve closed-loop circularity. Immersing cured composites in ethylene glycol containing **2 wt% Zn(acac)<sub>2</sub>** at **170°C** degrades the polymer matrix into soluble oligomers within 3 hours.

Circular Economy Metrics



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