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HEMPOXIES 69



Hempseed-Oil Bio-Vitrimer Composite Research Platform

Design, Synthesis and Characterization of a Sustainable, Carbon-Negative Vitrimer Composite via a Hempseed-Oil Epoxy Matrix via a Hierarchical Biogenic Carbon Reinforcement System

Cited Research Dossier, Mechanistic Hypothesis, Prior-Art Analysis, and Experimental Protocol

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Research-stage disclosure (TRL 1–2). This document describes a *pre-experimental research hypothesis*. Every performance value, carbon balance, and mechanism described herein is **projected and unvalidated** and is presented as a forward-looking research programme, not as established or demonstrated technology. All volatile facts and literature values have been live-verified against the cited primary sources; projected design targets are flagged as such throughout. Citations follow a Retrieve = Store = Cite discipline — no claim is attributed without a resolvable source.

Abstract

Conventional carbon-fibre-reinforced polymers (CFRPs) couple outstanding specific mechanical properties to a permanently cross-linked thermoset matrix that cannot be reprocessed, repaired, or de-bonded from its fibres, rendering the composite effectively unrecyclable and committing the embodied carbon of the fibre to landfill or incineration at end-of-life. This dossier proposes — and lays out a falsifiable experimental programme to test — a closed-loop alternative built on three design pillars: (i) a *bio-based vitrimer matrix*, in which a hempseed-oil-derived epoxy is cured into an ester-rich covalent adaptable network (CAN) whose topology can rearrange by catalysed transesterification above a topology-freezing temperature T_v , following the associative-exchange paradigm introduced by Montarnal, Capelot, Tournilhac and Leibler [1]; (ii) a *hierarchical, predominantly biogenic carbon reinforcement*, pairing continuous polyacrylonitrile carbon fibre (PAN-CF) at the macro-scale with hemp-bast-derived carbon nanosheets [12] and pyrolytic biochar [14,15] at the nano- and micro-scales; and (iii) a *dual orthogonal dynamic-bond hypothesis*, in which furfuryl glycidyl ether (FGE) serves both as a fully bio-based reactive diluent [9,10] and as a furan donor for optional thermoreversible Diels–Alder bonds with maleimide-grafted fibres [11], superposing an interfacial self-healing mechanism on the bulk vitrimer. We position this concept against the state of the art — including the closely related 2025 demonstration of bio-based epoxy-vitrimer CFRP by Lin *et al.* [6] — articulate what is, to our knowledge, unreported, and develop a structured central hypothesis with six falsifiable sub-predictions spanning rheological vitrimer signatures, mechanical synergy, reprocessing, fibre reclamation, and a net-negative cradle-to-gate carbon balance.

1. Introduction

Thermosetting epoxy composites dominate high-performance aerospace, automotive, wind-energy, and sporting-goods markets because their permanently cross-linked networks confer dimensional stability, creep resistance, solvent resistance, and high specific stiffness and strength. That same permanence is their end-of-life liability: a fully cured thermoset cannot be melted, reshaped, or dissolved without destroying the network, so CFRP waste is overwhelmingly landfilled or incinerated, and the carbon-intensive fibre it contains is lost [6,17].

The *vitriimer* concept reframes this trade-off. In 2011, Montarnal, Capelot, Tournilhac and Leibler showed that an epoxy network cured with carboxylic acids or anhydrides, in the presence of a transesterification catalyst, could rearrange its own topology through associative bond-exchange reactions without ever depolymerising; the cross-link density stays constant while the network flows, and the viscosity follows an Arrhenius law reminiscent of vitreous silica — hence “vitriimer” [1]. Unlike dissociative covalent adaptable networks, which break a bond before forming a new one, associative vitrimers never lose integrity, so they behave as insoluble cured resins at service temperature yet become reprocessible above a characteristic topology-freezing temperature T_v [4]. The intervening decade has produced a large and maturing literature on vitriimer chemistries, catalysis, and, increasingly, fibre-reinforced structural composites [2,3,4,5,6,7,8].

This dossier asks a focused question: *can the vitriimer paradigm be combined with an all-hemp, carbon-negative materials palette to yield a structural CFRP that is recyclable by design and that stores more biogenic and pyrolytic carbon than its own manufacture emits?* The objective is not to claim such a material exists, but to specify it precisely enough to be built and falsified — to convert an aspiration into a testable hypothesis with defined formulations, processing windows, characterisation endpoints, and pass/fail criteria.

2. State of the Art and Prior Art

The proposed system sits at the intersection of seven established research threads. We summarise each, with its most load-bearing primary citations, so that the genuinely novel contribution (Section 3) can be stated against a verified baseline rather than asserted in a vacuum.

2.1 Associative vitrimers and transesterification CANs

The foundational demonstration is Leibler and co-workers’ silica-like malleable epoxy networks [1]. The mechanism was placed on a kinetic footing by Capelot *et al.*, who quantified metal-catalysed transesterification for healing and assembling thermosets [2] and showed that the catalyst controls the vitriimer glass transition [3]. Denissen, Winne and Du Prez’s review remains the standard map of the chemistries that produce associative, glass-like flow [4], and Bowman and Kloxin framed the broader family of covalent adaptable networks into which vitrimers fall [5]. The defining, and experimentally accessible, signature is an Arrhenius-law stress-relaxation time whose activation energy for transesterification vitrimers typically falls in the range ~ 60 – 160 kJ mol⁻¹ [1,4].

2.2 Bio-based epoxy vitrimers

Replacing petroleum bisphenol-A chemistry with renewable monomers in vitrimers is an active field. Reported routes include lignin-derived bisphenols [6], algae-derived phloroglucinol triglycidyl ether cured with bio-based anhydrides [7], and furan-based polyimine elastomeric vitrimers from biomass-derived dialdehydes (noted within [4]). These establish that *bio-based* and *vitriimeric* are compatible design goals. A hempseed-oil triglyceride epoxy specifically has since been realised as a vitriimer network former — both commercially (ZILA *Hempoxy*TM [18]) and in the peer-reviewed dual-network biocomposite of Fei *et al.* [19] — so the matrix chemistry itself is precedented; see Section 2.5.

2.3 Recyclable CFRP enabled by vitrimer matrices

Several groups have now built continuous-fibre CFRP on vitrimer matrices to enable reprocessing, repair, and clean fibre reclamation. Industrial-scale prepreg CFRP on a polyether-sulfone-modified epoxy vitrimer reached a glass transition of 86.2 °C and a 16 GPa storage modulus, with measurable stress relaxation above 200 °C [8]. Most directly relevant, Lin *et al.* (2025) cured a lignin-derived bio-based epoxy with adipic acid into a vitrimer and produced CFRP with tensile and flexural strengths of 543 and 414 MPa — comparable to petroleum-based CFRP — while retaining transesterification-driven repairability and chemical degradability for fibre recovery [6]. This is the closest published analogue to the present concept and is treated explicitly in the novelty analysis.

2.4 Hempseed-oil epoxy thermosets

Epoxidised hemp(seed) oil (EHO) is a documented bio-epoxy feedstock. Anusic *et al.* developed an anhydride-cured EHO thermoset with a 57.5 wt% bio-based carbon fraction and characterised its moisture sensitivity [13]. Other groups have cured EHO with bio-based maleinised hemp oil and methyl nadic anhydride to approach fully bio-based networks, tuning stiffness against toughness through hardener ratio. Crucially, these EHO thermosets are conventional (statically cross-linked) resins: **they are not reported as vitrimers**, because vitrimer behaviour additionally requires an exchangeable-bond population and a transesterification catalyst (Section 5.5). The hempseed-oil-as-vitrimer-matrix step is therefore unproven prior art rather than established fact.

2.5 Commercial hempseed-oil vitrimer and the closest dual-network precedent

The hempseed-oil epoxy that anchors this programme is not hypothetical at the resin level: ZILA BioWorks (Renton, WA) has commercialised *Hempoxy*[™], a hempseed-oil-derived, up to ~85% bio-based epoxy with vitrimer (dynamic-covalent, recyclable) chemistry, and has fielded it in a Burton snowboard pilot reporting a ~78% cradle-to-gate carbon-footprint reduction versus petroleum epoxy, plus early glass-fibre wind-blade testing [18]. The present work treats a Zila-class hempseed-oil epoxy as the candidate network former (Section 5.6) rather than re-deriving the base resin. Most importantly for novelty scoping, Fei, Zhang and co-workers have already published the closest dual-network precedent: a hempseed-oil epoxy (supplied by Zila Works) combined with limonene-derived components into a *dual dynamic-network* vitrimer bearing orthogonal imine and hydroxy-ester bonds, reinforced with hemp *fibre* and made recyclable by mild aminolysis (100 °C, 3 h) [19]. This establishes that (i) a hempseed-oil vitrimer matrix and (ii) an orthogonal dual-dynamic-bond strategy are each already realised; the present concept must therefore be differentiated on its *specific* chemistry and architecture (continuous carbon fibre rather than hemp fibre; transesterification paired with *interfacial* furan–maleimide Diels–Alder self-healing rather than imine/ester aminolysis; and a multi-scale biogenic carbon-reinforcement hierarchy), as set out in Section 3.

2.6 FGE: a bio-based reactive diluent *and* a furan donor

Furfuryl glycidyl ether (FGE; 2-[(oxiran-2-ylmethoxy)methyl]furan) is derived from furfural / furfuryl alcohol and bio-epichlorohydrin. As a reactive diluent it lowers epoxy viscosity more effectively than the common commercial diluent Cardura E10P; reported optima of ~10–15 wt% raise flexural and tensile strength by ~16% without depressing T_g , and loadings to 20 wt% have given large gains in adhesion, tensile, and flexural strength when cured with amines [9,10]. Independently, FGE's pendant furan ring is a classic diene for thermoreversible Diels–Alder (DA) chemistry: Zhang and co-workers grafted bismaleimide onto T700 PAN fibre and introduced FGE

furans into the matrix, generating a DA interphase with $\sim 82\%$ interfacial self-healing efficiency [11]. FGE thus offers two distinct functions in a single, bio-based molecule — processing aid and dynamic-bond precursor.

2.7 Hemp-bast-derived carbon nanosheets

Wang, Mitlin and co-workers (D. Mitlin's group, University of Alberta / NINT-NRC) converted hemp bast fibre into interconnected, partially graphitic carbon nanosheets (10–30 nm thick) with specific surface area up to $2287 \text{ m}^2 \text{ g}^{-1}$, mesoporosity up to 58%, and electrical conductivity of $211\text{--}226 \text{ S m}^{-1}$ [12]. This material was developed and benchmarked for *supercapacitor* electrodes, not structural reinforcement; its high aspect ratio, graphitic character, and oxygen-bearing surface chemistry nonetheless make its repurposing as a crack-arresting nanofiller a reasonable, if untested, hypothesis.

2.8 Biochar as a functional composite filler

Pyrolytic biochar is an established low-cost, renewable filler in epoxy and CFRP systems. Giorcelli, Bartoli and co-workers showed that maple-derived biochar in epoxy can match or exceed multi-walled carbon nanotubes on selected mechanical metrics at 2–4 wt%, and deliver useful microwave-range conductivity at higher loadings [15]; in CFRP, 10 wt% wood biochar improved storage modulus, flexural and impact strength, and thermal stability [14]. Biochar's porous, high-fixed-carbon structure also provides mechanical interlocking and a permanent carbon sink.

3. Novelty and Contribution

Each constituent above has precedent. The contribution of this programme is the *integration*, and the specific, falsifiable claims that integration enables. We state the novelty conservatively — “to our knowledge, unreported” — consistent with a research-stage disclosure.

Claimed novelty (to be defended, not assumed)

1. **Hempseed-oil triglyceride epoxy vitrimer as a matrix for continuous-carbon-fibre CFRP.** A hempseed-oil vitrimer matrix is itself preceded — commercially [18] and in a hemp-*fibre* dual-network biocomposite [19]. What appears unreported is its use, with transesterification as the bulk reprocessing mechanism, as the matrix for *continuous carbon-fibre* structural laminates, where the goal is fibre reclamation and reuse of high-embodied-carbon PAN-CF rather than recycling of low-cost hemp fibre.
2. **An all-biogenic, multi-scale carbon reinforcement hierarchy in one matrix.** The combination of continuous PAN-CF (macro load path) + hemp-bast carbon nanosheets (nano crack-arrest) + biochar (micro interlocking / functional carbon) has not, to our knowledge, been assembled in a single composite; in particular, repurposing Mitlin-type energy-storage nanosheets [12] as a structural nanofiller is new.
3. **A dual, orthogonal dynamic-bond architecture (“two clocks”).** Orthogonal dual-dynamic-bond hempseed-oil vitrimers exist — Fei *et al.* pair imine and hydroxy-ester bonds, both cleavable by aminolysis [19]. The present architecture is distinct in *mechanism and role*: associative transesterification governs bulk reprocessing and fibre reclamation, while *dissociative* furan–maleimide DA bonds — from FGE furans already present as diluent [9,10] and maleimide-grafted PAN-CF [11] — act as a localised *interfacial self-healing* layer rather than a bulk recycling route. Co-locating a bulk transesterification mechanism with an interfacial DA mechanism in one bio-based, continuous-carbon-fibre CFRP is, to our knowledge, unreported.
4. **An explicit, figure-grounded carbon-negative life-cycle thesis** that couples biogenic sequestration (hemp matrix + nanosheets), pyrolytic carbon locking (biochar), and amortisation of PAN-CF embodied carbon through vitrimer-enabled circular reuse, quantified against live-verified LCA data (Section 7).

4. Central Hypothesis and Falsifiable Predictions

The programme is organised around one central hypothesis and six sub-hypotheses, each paired with a measurable outcome and an explicit falsification criterion. This structure is the scientific core of the dossier: it makes the concept refutable.

Central hypothesis (H_0). A hempseed-oil-derived epoxy, cured with a carboxylic-acid/anhydride hardener in the presence of a transesterification catalyst and diluted with FGE, and reinforced with a hierarchical biogenic carbon system around continuous PAN-CF, will (a) form a covalent adaptable network exhibiting Arrhenius stress relaxation; (b) retain structural mechanical properties at service temperature; (c) permit thermally activated topology rearrangement, reprocessing, and clean PAN-CF reclamation above T_v ; and (d) yield a net-negative cradle-to-gate CO_2 balance once the avoided burden of fibre re-manufacture is credited.

Falsifiable sub-hypotheses		
ID	Prediction (measurable)	Falsification criterion
H_1	Bulk matrix shows Arrhenius stress relaxation with $E_a \approx 60\text{--}160\text{ kJ mol}^{-1}$, the transesterification-vitrimer range [1,4].	No measurable relaxation, or non-Arrhenius behaviour, $\Rightarrow$ not a transesterification vitrimer.
H_2	Catalyst is necessary: a catalyst-free control shows negligible relaxation / reprocessing under identical conditions [2,3].	Catalyst-free control reprocesses comparably $\Rightarrow$ exchange is not catalyst-gated as modelled.
H_3	Hierarchical fillers raise modulus and ultimate strength and arrest microcracks relative to neat resin, up to an optimal loading [14,15].	Monotonic mechanical degradation with filler, or no crack-arrest in fractography, $\Rightarrow$ reinforcement hypothesis fails.
H_4	Strength-recovery (healing) at T_{act} scales with bulk transesterification and, where maleimide-functionalised fibre is used, with interfacial DA reformation [11].	No recovery, or recovery independent of both mechanisms, $\Rightarrow$ dual-network thesis fails.
H_5	PAN-CF reclaimed after matrix devitrification/solvolysis retains the bulk of its single-fibre tensile strength [6,7].	Reclaimed fibre strength collapses $\Rightarrow$ no closed-loop fibre value.
H_6	Cradle-to-gate balance is net-negative when reclaimed-fibre credit + biogenic/pyrolytic storage exceed process emissions [13,16,17].	Balance remains net-positive under transparent accounting $\Rightarrow$ “carbon-negative” claim retracted.

Note that H_1 , H_2 , and H_5 are gating: if the matrix is not a genuine vitrimer and the fibre cannot be cleanly reclaimed, the central circular-economy thesis fails regardless of the mechanical results.

5. Material Selection and Design Logic

Each component is chosen for a defined mechanical, rheological, or dynamic-network role, with the supporting literature cited.

5.1 Continuous PAN carbon-fibre fabric (PAN-CF)

PAN-CF is the primary continuous macro-reinforcement and carries the bulk of tensile and flexural load via its exceptional specific strength and stiffness. Pairing continuous PAN-CF with a vitrimer matrix is precisely what converts a high-performance but disposable composite into a reclaimable one [6,8]: the fibre is the most carbon-intensive component (Section 7), so its recovery dominates the sustainability case.

5.2 Furfuryl glycidyl ether (FGE): diluent and furan donor

FGE is a fully bio-based reactive diluent that lowers processing viscosity — enabling complete wet-out of dense multi-ply PAN-CF — with reported optima near 10–15 wt% that *raise* flexural and tensile strength without depressing T_g [9,10]. The projected formulation (Section 10) places FGE at ≈ 13.5 wt% of the resin solids, inside this literature-optimal window. Its furan ring additionally supplies aromatic, char-forming thermal stabilisation and — if a maleimide dienophile is present — thermoreversible DA sites for interfacial self-healing [11] (Section 6.2).

5.3 Hemp-bast carbon nanosheets

High-aspect-ratio, partially graphitic hemp-derived nanosheets [12] are intended as the primary nano-reinforcement: to stiffen the matrix locally and to arrest micro-crack propagation before cracks reach the fibre–matrix interface. Their oxygen-bearing surface chemistry is hypothesised to improve interfacial shear strength with the bio-based resin. This structural role is a repurposing of an energy-storage material and is explicitly one of the predictions to be tested (H_3).

5.4 Pyrolytic biochar

Biochar is a functional secondary carbon filler: its porous structure provides mechanical interlocking with the polymer, improves thermal stability, and — through its graphitic fraction — aids uniform heat distribution during cure and during thermally activated healing and reprocessing [14,15]. It is simultaneously a permanent carbon sink (Section 7.2).

5.5 Hempseed-oil bio-epoxy system (Zila components A & B) — and the requirement that makes it a vitrimer

The Zila system [18] replaces petroleum bisphenol-A epoxy with a matrix derived from industrial hempseed oil: component A supplies epoxidised plant-lipid oxiranes; component B is the hardener. A critical design point must be stated explicitly, because it is frequently elided in bio-epoxy descriptions:

Vitrimer behaviour is not automatic. A hempseed-oil epoxy cured with a generic amine yields a *static* thermoset, not a vitrimer [13]. To obtain a *transesterification* vitrimer, the cured network must be *ester-rich* — achieved by curing the epoxy with a carboxylic-acid or cyclic-anhydride hardener, which generates the β -hydroxyester motifs (free hydroxyl + ester) required for exchange — *and* must contain an active transesterification catalyst (e.g. zinc acetate, zinc acetylacetonate $\text{Zn}(\text{acac})_2$, or the organocatalyst TBD) [1,2,3,7]. The dynamic, self-healing, reprocessable character claimed for this composite is therefore **contingent on hardener chemistry and catalyst inclusion**, and is one of the first things the experimental programme must verify (H_1 , H_2).

6. Mechanistic Framework

6.1 Associative transesterification (bulk reprocessing and reclamation)

In an ester-rich, catalysed network, a free hydroxyl attacks a neighbouring ester carbonyl; the catalyst lowers the barrier, and the bond “hops” from one partner to another without changing the total number of cross-links [1,2]. Because creation and cleavage are concerted (associative), the network never passes through a soluble sol state: it remains insoluble and load-bearing at service temperature, yet at elevated temperature the exchange rate rises and the material flows, enabling reshaping, welding, scratch repair, and — with solvent assist — matrix removal for fibre reclamation [1,4,6,7]. The relaxation time obeys $\tau^*(T) = \tau_0 \exp(E_a/RT)$, and the activation energy E_a is the single most diagnostic vitrimer parameter (H_1).

6.2 Optional dissociative furan–maleimide Diels–Alder (interfacial self-healing)

A second, chemically orthogonal dynamic bond can be installed at the fibre interphase. FGE furans (already present as diluent) react with maleimide groups grafted onto the PAN-CF surface to form DA adducts that are stable at service temperature but undergo retro-DA on heating, giving a thermally switchable, self-healing interphase with reported $\sim 82\%$ interfacial recovery [11]. Unlike transesterification, DA *is* dissociative (bond breaks before it reforms), so the two mechanisms operate on different “clocks” and, in principle, at different temperatures — a designed redundancy: the interphase can heal microdebonding while the bulk matrix simultaneously relaxes stress and reflows.

6.3 The two-temperature operating window

A vitrimer is defined between two temperatures: the glass transition T_g , below which segmental motion is frozen, and the topology-freezing temperature T_v , above which bond exchange is fast enough that the network flows on practical timescales [1,4]. **Service** occurs below T_v ; **reprocessing/healing/reclamation** occurs above it. The projected activation target of $T_{\text{act}} \sim 140^\circ\text{C}$ (Section 10) is optimistic: many transesterification vitrimers require $150\text{--}220^\circ\text{C}$ for second-scale relaxation [6,8]. The true T_v must be measured, and the cure schedule and catalyst loading tuned to place it usefully above service temperature but within the thermal tolerance of the fibre and tooling.

7. Carbon-Negative Life-Cycle Design

The carbon thesis rests on three live-verified levers. Figures are reported as ranges with their sources; the net balance is a *hypothesis to be tested by formal LCA* (H_6), not a settled result.

7.1 Biogenic sequestration in the hemp-derived fractions

The hempseed-oil matrix and the hemp-bast nanosheets originate in fast-growing industrial hemp. A Cambridge Centre for Natural Material Innovation estimate places industrial-hemp uptake at $\sim 8\text{--}15\text{ t CO}_2$ per hectare per growing season, against $\sim 2\text{--}6\text{ t CO}_2\text{ ha}^{-1}\text{ yr}^{-1}$ for typical forestry [16]. (Higher single-source advocacy figures, e.g. 22 t ha^{-1} , exist but are less well substantiated and are deliberately *not* used here.) Converting that biomass into durable structural polymer and graphitic nanosheets locks the fixed carbon into a solid, long-lived state.

7.2 Pyrolytic carbon locking in biochar

Pyrolysis converts labile biogenic carbon into a recalcitrant, high-fixed-carbon solid that resists microbial decomposition for decades to centuries; embedding biochar in a structural matrix isolates it further [14,15]. This is a permanence-oriented sink, complementary to the biogenic uptake above.

7.3 Amortising the PAN-CF burden through circular reuse

Virgin PAN carbon fibre is extraordinarily carbon- and energy-intensive: cradle-to-gate primary energy is widely reported at $\sim 198\text{--}595\text{ MJ kg}^{-1}$, with global-warming potential as high as $\sim 54\text{ kg CO}_2\text{-eq}$ per kg of fibre, driven by the high-temperature carbonisation of a synthetic precursor [17]. Recovering fibre from end-of-life composite, by contrast, requires on the order of $\sim 2\text{--}30\text{ MJ kg}^{-1}$ depending on process [17] — one to two orders of magnitude lower. **This gap is the engine of the carbon case:** because the Zila vitrimer matrix can be devitrified and the continuous PAN-CF reclaimed without chemical degradation or fibre shortening, the fibre’s enormous embodied burden is spread across multiple service lives and amortised toward zero. Combined with biogenic and pyrolytic storage in the matrix and fillers, the hypothesis is

that the assembled material can cross into a net-negative balance — the proposition H_6 exists precisely to test, and potentially to refute, that claim under transparent accounting.

8. Safety Considerations and Warnings

Risk-mitigation mandate. The materials and processes below require strict adherence to standard laboratory safety practice and personal protective equipment (PPE). This section does not substitute for supplier Safety Data Sheets (SDS), which must be obtained and read for every specific lot before handling.

8.1 Material hazards

- **Furfuryl glycidyl ether (FGE):** can cause severe skin irritation, allergic skin sensitisation, and eye damage; glycidyl ethers are a recognised sensitizer class. Handle in a certified chemical fume hood; avoid inhalation of vapour.
- **Carbon nanomaterials (hemp nanosheets, biochar):** fine particulates are a serious inhalation hazard; handling raw powders can cause respiratory-tract irritation. Treat as nuisance-to-hazardous dust; weigh and transfer inside containment.
- **PAN carbon-fibre fabric:** cutting and handling dry fabric releases airborne fibre fragments that cause skin itching and respiratory irritation and can short unprotected electronics. Cut wet where feasible; capture dust at source.
- **Hardener (Zila component B):** amine- or acid/anhydride-based curing agents are typically corrosive and/or sensitising. Avoid skin contact and vapour inhalation.
- **Transesterification catalysts:** zinc salts and organic superbases (e.g. TBD) have their own hazard profiles; consult the SDS before use.

8.2 Engineering controls and equipment hazards

- **High-shear / ultrasonic processing:** high-intensity acoustic energy causes permanent hearing damage — operate inside an acoustic enclosure or wear dedicated hearing protection. Sonication heats the slurry rapidly; monitor temperature and use a cooling bath.
- **Heated press:** operates at high temperature ($\sim 140^\circ\text{C}$) and pressure ($\sim 0.5\text{ MPa}$). Wear thermal-resistant gloves during layup loading/unloading.

9. Required Equipment and Material Sourcing

9.1 Equipment checklist

- Analytical balance (accuracy $\pm 0.01\text{ g}$)
- High-shear tip sonicator with temperature probe and water bath
- Overhead mechanical stirrer ($\geq 2500\text{ rpm}$)
- Vacuum chamber / desiccator ($< 10\text{ mbar}$)
- Heated hydraulic press / compression mould
- PPE: nitrile gloves (double-gloving advised), chemical splash goggles, lab coat, and a HEPA-filtered respirator (N95 or better) for dry-powder handling and fabric cutting

Component	Supplier / source	Lot / batch
Furfuryl glycidyl ether (FGE)	[specialty chemical]	_____
Hemp-bast carbon nanosheets	[per ref. 12 process / vendor]	_____
Pyrolytic biochar	[specialty / pyrolysis]	_____
Zila component A (hempseed-oil epoxy)	[Zila BioWorks, ref. 18]	_____
Zila component B (hardener)	[Zila BioWorks, ref. 18]	_____
Transesterification catalyst	[Zn(OAc) ₂ / Zn(acac) ₂ / TBD]	_____
PAN carbon-fibre fabric (4-ply)	[Toray / equivalent]	_____

9.2 Component sourcing log

10. Experimental Methodology (Projected Starting Point)

Status of these quantities

The masses below are a *projected, illustrative starting formulation*, not an optimised recipe. They define the centre point of a planned design-of-experiments (DOE) screen across filler loading, FGE fraction, catalyst loading, and cure schedule. The FGE fraction (≈ 13.5 wt% of resin solids) and total carbon-filler fraction (≈ 5.3 wt%) are chosen to sit within literature-optimal windows [9,10,14,15].

10.1 Step 1 — Slurry exfoliation and solvation

1. Don a HEPA-filtered respirator before opening carbon containers. Weigh **10.42** g FGE in the fume hood.
2. Wet-in **2.20** g hemp carbon nanosheets and **1.90** g biochar into the FGE, gently, to prevent aerosolisation.
3. In an acoustic enclosure, tip-sonicate **45** min at **35 °C** to break carbon macro-aggregates without degrading the furan rings; cool as needed.

10.2 Step 2 — Resin blending and catalyst incorporation

1. Add **41.67** g Zila component A to the carbon/FGE slurry.
2. **Incorporate the transesterification catalyst** (e.g. 1–5 mol% Zn(acac)₂ relative to ester equivalents; loading is a DOE variable) and disperse thoroughly — this step is what gives the cured network its vitrimer character (Section 5.5).
3. Mechanically stir at **2500** rpm for **30** min to distribute the carbon network.
4. Warm to **45 °C** and pull vacuum (< 10 mbar) to degas and suppress voids.

10.3 Step 3 — Activation, layup, consolidation

1. Add **24.88** g Zila component B (acid/anhydride-functional hardener; see Section 5.5) and stir 3 min to initiate cure.
2. Infuse uniformly into 4 plies of PAN-CF by wet layup or vacuum-assisted impregnation; ensure full wet-out of every ply.

3. Consolidate in a heated press at **0.5 MPa**: **2 h** at **90 °C** to build base cross-linking, then post-cure **4 h** at **140 °C**. Treat the post-cure temperature as a hypothesis: confirm against the measured T_v (Section 6.3) and adjust.

11. Characterisation and Quality Assurance

The test matrix is designed to discharge each sub-hypothesis. Beyond the original endpoints, two vitrimer-specific assays are mandatory.

- a. **FTIR** — confirm epoxide consumption and survival of furan rings.
- b. **DSC** — measure T_g and cross-link maturity.
- c. **Rheometry / stress relaxation (gates H_1, H_2)** — measure $\tau^*(T)$ at several temperatures; extract E_a ; confirm Arrhenius behaviour; compare catalysed vs catalyst-free controls. *This is the definitive vitrimer test.*
- d. **Swelling / gel fraction** — confirm the network is insoluble (associative, not dissociative) at service temperature.
- e. **Tensile (ASTM D3039)** — ultimate strength, modulus, and load-transfer efficiency on cured coupons.
- f. **Healing / reprocessing (gates H_4)** — introduce a controlled crack, heat at T_{act} under contact pressure, quantify % strength recovery; where applicable, separate bulk-transesterification and interfacial-DA contributions.
- g. **Fibre reclamation (gates H_5)** — devitrify/solvolyse the matrix, recover PAN-CF, and measure single-fibre tensile strength of reclaimed fibre against virgin fibre.

12. Results and Data Collection

12.1 Processing observations

- Actual FGE mass: _____ g Catalyst loading: _____ mol%
- Actual component A / B mass: _____ g / _____ g
- Time to < 10 mbar: _____ min Measured T_v : _____ °C

12.2 Mechanical, thermal, and dynamic-network properties

Sample	Tensile (MPa)	T_g (°C)	E_a (kJ mol ⁻¹)	Healing (%)
Control (neat Zila)	[baseline]	[baseline]	_____	N/A
Catalyst-free control	_____	_____	_____	_____
Batch 01 (hemp/biochar)	_____	_____	_____	_____
Batch 02	_____	_____	_____	_____

13. Discussion (Template)

Interpret deviations from prediction against each sub-hypothesis. Did stress relaxation follow an Arrhenius law, and where did E_a fall relative to the 60–160 kJ mol⁻¹ transesterification range [1,4]? Did the catalyst-free control confirm catalyst-gating (H_2)? Did the 45-min sonication achieve nanosheet/biochar de-aggregation, and did fractography show crack-arrest (H_3)? Was

wet-out of the 4-ply PAN-CF complete, or were dry spots present? How did interfacial adhesion between the hempseed-oil matrix, the biogenic fillers, and the PAN-CF surface influence T_g , T_v , and load transfer? If a maleimide-functionalised fibre route was used, could interfacial DA healing be distinguished from bulk transesterification (H_4)? Did reclaimed fibre retain strength (H_5)?

14. Conclusion (Template)

State whether the cure profile (90°C / 140°C) and catalyst system produced a genuine, Arrhenius-relaxing vitrimer; whether the hierarchical biogenic reinforcement delivered net mechanical benefit; whether PAN-CF was cleanly reclaimed; and whether a formal LCA supported or refuted the net-negative carbon claim (H_6). Identify the next optimisation vector — most likely the balance of bio-resin to reactive diluent, catalyst loading, and the placement of T_v relative to service and fibre-tolerance temperatures.

Limitations and Technology-Readiness Statement

This is a **TRL 1–2 research hypothesis**. No formulation in this dossier has been synthesised or tested by the author at time of writing; all performance figures are *projected*. The principal open risks are: (i) whether a hempseed-oil triglyceride network, which is aliphatic and relatively low in cross-link density, can host fast enough transesterification at a useful T_v ; (ii) whether hemp nanosheets developed for electrochemistry function as structural reinforcement; (iii) whether the optimistic $\sim 140^\circ\text{C}$ activation target is achievable, or whether $150\text{--}220^\circ\text{C}$ is required [6,8]; and (iv) whether the carbon-negative balance survives transparent LCA. Each is encoded as a falsifiable prediction in Section 4. Literature values herein were live-verified against the cited primary sources; quantitative hemp-sequestration and CF-LCA figures are reported as ranges to reflect genuine spread in the literature.

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