Hempoxies Variant CF-TAIA-95 (v.25-CF):

Open-Source Technical Specification for a 100% Bio-Based, Catalyst-Free Epoxidized Hemp Seed Oil Vitrimer Nanocomposite ($R = 0.95$, 0 wt% FGE, 0 wt% Metal Catalyst)

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AI Disclosure: Generated collaboratively with Google Gemini (November 24, 2026) under the deterministic verification rules of *Seshat's Axiom* ($1 = 1 = 1$) and the *Landry Hallucination-Free Protocol (LHFP)*. **Verification State:** [X THEORETICAL] Pre-Experimental Specification (TRL 1–2). All molar masses, equivalent weights, and stoichiometric equations are algebraically verified; however, this integrated formulation has not yet undergone wet-lab synthesis or physical ASTM characterization and must be treated as a falsifiable experimental hypothesis until validated.

1. Observation of Anomalies in Prior Hempoxies Baselines

Systematic chemical auditing of prior Hempoxies pre-experimental formulations—including the fixed-datum Epoxidized Hemp Seed Oil (EHSO) / citric acid / zinc acetate / furfuryl glycidyl ether (FGE) system (v.17.2), the sebacic acid / zinc acetylacetonate variant (H43), and multi-filler hybrid matrices (v.21–v.24)—identified five recurring physicochemical anomalies that compromise reproducibility, green chemistry compliance, and closed-loop reprocessability [X THEORETICAL]:

1. **Solid Carboxylic Acid Phase Separation & Dehydration Voids:** Crystalline polyacids such as citric acid ($T_m \approx 153\,^{\circ}\mathrm{C}$) exhibit low miscibility in non-polar triglycerides (EHSO), causing heterogeneous local gelation. Moreover, citric acid's tertiary hydroxyl group undergoes thermal dehydration above 150 °C, evolving water vapor that creates micro-voids and hydrolyzes ester crosslinks.
2. **Monofunctional Diluent Crosslink Penalty & Petrochemical Heritage:** Furfuryl glycidyl ether (FGE) is monofunctional ($f = 1$). At 8.9–20 wt%, FGE consumes hardener equivalents without forming inter-chain bridges, depressing crosslink density (ν_e) and glass transition temperature (T_g). Furthermore, commercial FGE is synthesized using epichlorohydrin (Petrova et al., 2019), introducing non-renewable carbon and chlorinated manufacturing waste alongside skin-sensitization hazards (Fisher Scientific SDS, CAS 5380-87-0).
3. **Transition-Metal Catalyst Chelation & Leaching:** External transesterification catalysts such as $Zn(acac)_2$ (2.0 mol%) can be chelated and deactivated at filler interfaces by uncontrolled surface oxygen groups on oxidized biomass carbons, or leach out during service and reprocessing.
4. **Chemical Activation Waste in Nanocarbons:** Producing high-surface-area hemp carbon nanosheets via molten

potassium hydroxide (KOH) activation at 700–800 °C followed by hydrochloric acid washing (Wang et al., 2013) generates substantial caustic and saline effluent.

5. **Autoxidative Crosslink Drift During Reprocessing:** Incomplete double-bond epoxidation in EHSO leaves 5–15% residual unsaturated C=C bonds on linoleic/linolenic chains. Hot-press reprocessing at 170–180 °C in air triggers radical autoxidation, converting a dynamic vitrimer into a permanently crosslinked, brittle thermoset by Cycle 2.

2. Definition of Intelligence & Engineering Requirements

To resolve all five anomalies simultaneously, **Hempoxies Variant CF-TAIA-95 (`v.25-CF`)** is engineered to meet six strict technical and ecological specifications:

- **100% Bio-Based Carbon Architecture:** Complete elimination of petrochemical reactive diluents (0.0 wt% FGE) and petrochemical cyclic anhydrides (0.0 wt% methyl nadic anhydride), using exclusively bio-renewable monomers.
- **Zero External Metal Catalysts (0.0 wt% Metal Salts):** Associative transesterification must proceed via internal neighboring-group participation (NGP) and abundant free phenolic/aliphatic hydroxyl (−OH) groups (Montarnal et al., 2011; Zhao et al., 2024; Berne et al., 2022).
- **Non-Raw-Fiber Processed Hemp Carbon Only:** Raw unprocessed hemp bast/hurd fibers are strictly excluded. Reinforcement is restricted to physically steam- or CO_2-activated, ball-milled hemp biochar (eliminating KOH/acid activation waste).
- **Deterministic Lot-to-Lot Stoichiometric Calibration:** Exact algebraic recalculation equations tied to measured Epoxy Equivalent Weight (EEW_{meas} via ASTM D1652) and Hydroxyl Value (HV_{meas} via ASTM E222) so natural feedstock variance never shifts the equivalence ratio (R).
- **Radical Stabilization of Unsaturated Chains:** Addition of 0.20 wt% bio-derived α-tocopherol (Vitamin E) to scavenge alkyl/peroxyl radicals on residual EHSO C=C bonds and itaconic anhydride exocyclic double bonds during thermal cure and hot-press recycling.
- **Verified Closed-Loop Reprocessability:** Gel fraction $G \geq 90.0\%$, monotonic Arrhenius stress-relaxation decay (τ^*) between 160 °C and 180 °C, and $\geq 80.0\%$ tensile strength retention through two mechanical reprocessing cycles.

3. Formulation Hypothesis (Variant CF-TAIA-95)

Central Falsifiable Hypothesis `[X THEORETICAL]`: Curing enzymatically epoxidized hemp seed oil (EHSO, 100.00 g base, 0 wt% FGE) with a 60 : 40 equivalent-ratio dual bio-hardener composed of **tannic acid** (60 eq% phenolic −OH, hyperbranched polyphenolic crosslinker and self-catalyst) and **itaconic anhydride** (40 eq% cyclic anhydride, low-melting bio-derived network stiffener) at an equivalence ratio of $R = 0.95$, stabilized with 0.20 wt% α-tocopherol and reinforced across a single-variable loading ladder ($0.0, 0.5, 1.5, 3.0$ wt%) of vacuum-dried steam-activated hemp biochar, produces a void-free, 100% bio-based, catalyst-free vitrimer nanocomposite capable of associative stress relaxation and closed-loop hot-press reprocessing.

Variant Nomenclature Key: **CF** = Catalyst-Free (0% metal salts); **TAIA** = Tannic Acid + Itaconic Anhydride dual bio-hardener (60 : 40 eq%); **95** = Target equivalence ratio $R = 0.95$; **`v.25-CF`** = Sequential Hempoxies specification revision 25.

4. Experimental Design & Stoichiometric Architecture

4.1 Precursor Reference Constants & Lot Calibration Log

Because natural polygalloyl tannic acid varies in hydroxyl density across botanical sources (HEW ≈ 68–85 g/eq) and EHSO oxirane conversion varies by batch (EEW ≈ 193–226 g/eq; Lerma-Canto et al., 2023), record measured Certificate of Analysis (CoA) or titration values in Table 1 prior to weighing.

Table 1: Precursor Reference Constants and Measured Lot Calibration Log (`CF-TAIA-95`)

Precursor	Lot / Batch ID	Reference Benchmark	Measured Lot Value	Units
EHSO (ASTM D1652)	_____	$O_o = 7.62\%$ (EEW = 210.0)	$O_o =$ _____% | $\text{EEW}_{\text{meas}} =$ _____	wt% | g/eq
Tannic Acid (ASTM E222)	_____	HV = 824.51 (HEW = 68.05)	$\text{HV}_{\text{meas}} =$ _____ | $\text{HEW}_{\text{TA,meas}} =$ _____	mg KOH/g | g/eq
Itaconic Anhydride	_____	$\text{AEW}_{\text{IA}} = 112.08$ (99%+ pure)	Purity $P_{\text{IA}} =$ _____%	g/eq | %
Hemp Biochar (Dried)	_____	Moisture < 0.5 wt%	Post-dry mass loss = _____%	wt%

4.2 Exact Algebraic Recalculation Equations (for Chosen Base Mass m_{EHSO})

For any chosen base mass of EHSO (m_{EHSO} in grams), compute exact reagent masses as follows:

$$\text{EEW}_{\text{meas}} = \frac{16.00 \times 100}{O_o(\%)} = \frac{1600}{O_o(\%)} \tag{1}$$

$$\text{Eq}_{\text{epoxy}} = \frac{m_{\text{EHSO}}}{\text{EEW}_{\text{meas}}} \tag{2}$$

$$\text{HEW}_{\text{TA,meas}} = \frac{56.106 \times 1000}{\text{HV}_{\text{meas}}} = \frac{56106}{\text{HV}_{\text{meas}}} \tag{3}$$

$$m_{\text{TA}} = 0.60 \times \left(0.95 \times \text{Eq}_{\text{epoxy}}\right) \times \text{HEW}_{\text{TA,meas}} = 0.5700 \times \left(\frac{m_{\text{EHSO}}}{\text{EEW}_{\text{meas}}}\right) \times \text{HEW}_{\text{TA,meas}} \tag{4}$$

$$m_{\text{IA}} = 0.40 \times \left(0.95 \times \text{Eq}_{\text{epoxy}}\right) \times \frac{112.08}{P_{\text{IA}}/100} = 42.5904 \times \left(\frac{m_{\text{EHSO}}}{\text{EEW}_{\text{meas}}}\right) \times \left(\frac{100}{P_{\text{IA}}}\right) \tag{5}$$

$$M_{\text{matrix}} = m_{\text{EHSO}} + m_{\text{TA}} + m_{\text{IA}} \tag{6}$$

$$m_{\text{tocopherol}} = 0.0020 \times M_{\text{matrix}} \qquad \text{and} \qquad m_{\text{biochar}} = \left(\frac{w_{\text{filler}}}{100}\right) \times M_{\text{matrix}} \tag{7}$$

Worked Lot-Recalculation Sanity Check (m_{EHSO} = 100.00 g): If a supplier CoA reports $O_o = 7.44\%$ ($\text{EEW}_{\text{meas}} = 215.05$ g/eq), $\text{HV}_{\text{meas}} = 750$ mg KOH/g ($\text{HEW}_{\text{TA,meas}} = 74.81$ g/eq), and $P_{\text{IA}} = 100\%$, then $\text{Eq}_{\text{epoxy}} = 0.4650$ eq, $\mathbf{m_{TA}} = \mathbf{19.83}$ g, $\mathbf{m_{IA}} = \mathbf{19.80}$ g, $\mathbf{M_{matrix}} = \mathbf{139.63}$ g, $\mathbf{m_{tocopherol}} = \mathbf{0.28}$ g, and 1.5 wt% biochar = **2.09** g.

4.3 Master Batch Weighing Table ($R = 0.95$, Benchmark Constants)

Run the **1/4-scale screening batch (**25.00 **g EHSO, values in brackets)** first to verify gelation and cure behavior before scaling to full 100.00 g EHSO plaques.

Table 2: Master Stoichiometric Batch Sheet for Hempoxies Variant `CF-TAIA-95` (EEW = 210.0, $\text{HEW}_{\text{TA}} = 68.05$, $\text{AEW}_{\text{IA}} = 112.08$)

Run ID	Biochar	m_{EHSO} (g)	m_{TA} (g)	m_{IA} (g)	Tocoph. (g)	Biochar (g)	Actual (g)
RUN-0 (Ctrl)	0.0 wt%	100.00 [25.00]	18.47 [4.62]	20.28 [5.07]	0.28 [0.07]	0.00 [0.00]	_____
RUN-1 (Low)	0.5 wt%	100.00 [25.00]	18.47 [4.62]	20.28 [5.07]	0.28 [0.07]	0.69 [0.17]	_____
RUN-2 (Mid)	1.5 wt%	100.00 [25.00]	18.47 [4.62]	20.28 [5.07]	0.28 [0.07]	2.08 [0.52]	_____
RUN-3 (High)	3.0 wt%	100.00 [25.00]	18.47 [4.62]	20.28 [5.07]	0.28 [0.07]	4.16 [1.04]	_____

4.4 Step-by-Step Synthesis & Curing Procedure

1. **Vacuum-Dry Hygroscopic Solids (**105–110 °C**,** 4.0 **h):** Dry tannic acid (5–10 wt% ambient moisture) and steam-activated hemp biochar (3–8 wt% ambient moisture) under vacuum for 4.0 hours. Cool inside a sealed desiccator and weigh immediately upon removal so moisture does not hydrolyze itaconic anhydride.
2. **Pre-Dissolve Tannic Acid in Melted Itaconic Anhydride (**75–100 °C**,** 20 **min):** Melt itaconic anhydride ($T_m \approx 68$ °C) at 75–80 °C under dry nitrogen or a sealed cap. Gradually add dry tannic acid while raising temperature to 95–100 °C with high-shear mixing for 20 min to yield a homogeneous liquid galloyl-itaconate pre-hardener.

(Solvent-assist fallback: if viscosity exceeds stirrer torque, pre-dissolve tannic acid in 15–20 *mL anhydrous* 200*-proof ethanol per* 100 *g batch, blend into melted IA at* 65 °*C, and strip all ethanol in Step 4.)*

3. **Disperse Biochar in Stabilized EHSO (**70 °**C,** 20 **min** + 10 **min):** Warm EHSO and α-tocopherol (0.20 wt%) to 70 °C. For filled batches (`RUN-1` to `RUN-3`), disperse dry hemp biochar via bath sonication or high-shear mixing for 20 min while viscosity is low. Pour the liquid TA/IA pre-hardener into the warm EHSO and mix vigorously for 10 min.
4. **Vacuum Degas & Solvent Strip (**75 °**C,** 30 **min):** Pull full vacuum at 75 °C for 30 minutes until all bubbling ceases. Cast slowly down the tilted wall of a preheated PTFE or silicone mold to eliminate air entrapment.
5. **Controlled Two-Stage Thermal Cure:** Ramp at 2 °C/min from 75 °C to 120 °C and hold for 3.0 hours (network gelation), then ramp at 2 °C/min to 155 °C and hold for 2.0 hours under nitrogen blanket if available (post-cure homogenization without radical C=C drift).

5. Data Collection Protocols & Analytical Instruments

Record all experimental parameters and analytical outputs directly in Tables 3–7 across all four runs (`RUN-0` through `RUN-3`).

Table 3: Thermal & Mixing Process Checkpoint Log

Process Stage	Setpoint	Ramp / Hold	Critical Control Check	Actual Log	Pass
1. Vacuum Dry Solids	105–110 °C	4.0 h vacuum	Cool in desiccator; weigh immediately	`___°C / ___h`	☐
2. Pre-Hardener Melt	95–100 °C	20 min shear	Log EtOH mL if used: `____ mL`	`___°C / ___m`	☐
3. Biochar & EHSO Mix	70 °C	20 m + 10 m	Disperse biochar in EHSO+tocopherol first	`___°C / ___m`	☐
4. Vacuum Degas	75 °C	30 min vacuum	Zero residual bubbles; pour down wall	`___°C / ___m`	☐
5. Stage 1 Gelation	120 °C	2 °C/min, 3.0 h	Tack-free gelation; no exotherm scorch	`___°C / ___h`	☐
6. Stage 2 Post-Cure	155 °C	2 °C/min, 2.0 h	N_2 blanket active: `Y / N`	`___°C / ___h`	☐

Table 4: Table 5A: ATR-FTIR Cure Completeness Log (4000–600 cm^{-1}, ASTM E1252)

Run ID	Anhydride (1780/1855)	Oxirane (825–915)	Ester C=O (1735)	Broad –OH (3400)	Gate Status
Uncured Ref	Strong doublet	Strong band	Baseline	Present	Reference
RUN-0 (0.0%)	_____	_____	_____	_____	_____
RUN-1 (0.5%)	_____	_____	_____	_____	_____
RUN-2 (1.5%)	_____	_____	_____	_____	_____
RUN-3 (3.0%)	_____	_____	_____	_____	_____

Table 5: Table 5B: Solvent Gel Fraction & Swelling Ratio Log (24 h THF or Toluene at 23 °C, adapted from ASTM D2765)

Run ID	Solvent	W_0 Dry (g)	W_s Swollen (g)	W_d Final (g)	Gel G (%)	Swell Q	$G \geq 90\%$
RUN-0 (0.0%)	_____	_____	_____	_____	_____	_____	_____
RUN-1 (0.5%)	_____	_____	_____	_____	_____	_____	_____
RUN-2 (1.5%)	_____	_____	_____	_____	_____	_____	_____
RUN-3 (3.0%)	_____	_____	_____	_____	_____	_____	_____

6. Quantitative Analysis & Governing Equations

Table 6: Table 5C: DMA Thermomechanical & Isothermal Stress-Relaxation Log (ASTM E1640)

Run ID	$E'_{25^\circ\mathrm{C}}$ (MPa)	T_g (°C)	E'_r (MPa)	ν_e (mol/m^3)	$\tau^*_{160^\circ\mathrm{C}}$ (s)	$\tau^*_{170^\circ\mathrm{C}}$ (s)	$\tau^*_{180^\circ\mathrm{C}}$ (s)	E_a (kJ/mol)
RUN-0	_____	____	_____	______	_____	_____	_____	______
RUN-1	_____	____	_____	______	_____	_____	_____	______
RUN-2	_____	____	_____	______	_____	_____	_____	______
RUN-3	_____	____	_____	______	_____	_____	_____	______

Table 7: Table 5D: Closed-Loop Reprocessing Cycle Log (Hot-Press at 170 °C, 5–10 MPa, 30 min; ASTM D638)

Run ID	Cycle	$\sigma_{\max}$ (MPa)	E (MPa)	ε_b (%)	ΔT_g (°C)	Retention (%)	$\geq 80\%$ Gate
RUN-0	Virgin (C0)	_______	_______	______	0.0 (Ref)	100.0%	Reference
RUN-0	Cycle 1 (C1)	_______	_______	______	______	_______	______
RUN-0	Cycle 2 (C2)	_______	_______	______	______	_______	______
RUN-1	Virgin (C0)	_______	_______	______	0.0 (Ref)	100.0%	Reference
RUN-1	Cycle 1 (C1)	_______	_______	______	______	_______	______
RUN-1	Cycle 2 (C2)	_______	_______	______	______	_______	______
RUN-2	Virgin (C0)	_______	_______	______	0.0 (Ref)	100.0%	Reference
RUN-2	Cycle 1 (C1)	_______	_______	______	______	_______	______
RUN-2	Cycle 2 (C2)	_______	_______	______	______	_______	______
RUN-3	Virgin (C0)	_______	_______	______	0.0 (Ref)	100.0%	Reference
RUN-3	Cycle 1 (C1)	_______	_______	______	______	_______	______
RUN-3	Cycle 2 (C2)	_______	_______	______	______	_______	______

Evaluate raw laboratory measurements using five standard polymer physics relations:

$$G(\%) = \left(\frac{W_d}{W_0}\right) \times 100 \qquad \text{and} \qquad Q = \frac{W_s - W_d}{W_d} \tag{8}$$

$$\nu_e = \frac{E'_r}{3R_{\mathrm{gas}}T} \tag{9}$$

$$\frac{G(t)}{G_0} = \exp\left(-\frac{t}{\tau^*}\right) \tag{10}$$

$$\ln(\tau^*) = \ln(\tau_0) + \frac{E_a}{R_{\mathrm{gas}}T} \tag{11}$$

$$\text{Strength Retention } (\%) = \left(\frac{\sigma_{\text{Cycle } n}}{\sigma_{\text{Virgin}}}\right) \times 100 \tag{12}$$

where $R_{\mathrm{gas}} = 8.314$ J mol^{-1} K^{-1}, $T = T_g$ [K] + 40 K for rubbery-plateau crosslink density ν_e in Eq. (9), and τ^* in Eq. (10) is recorded when normalized relaxation modulus decays to $G(t)/G_0 = 1/e \approx 0.368$.

7. Decision Loop & Falsification Criteria (Rejection Gates)

Under the *Landry Hallucination-Free Protocol*, Variant `CF-TAIA-95` must be rejected or reformulated if any experimental run violates the following falsification thresholds:

1. **Incomplete Network Formation ($G < 90.0\%$):** Indicates insufficient phenolic/anhydride cure or unstripped ethanol plasticization. *Corrective Branch:* Increase post-cure hold at 155 °C from 2.0 h to 3.0 h or verify $\mathrm{HV}_{\mathrm{meas}}$ titration.
2. **Non-Arrhenius Stress Relaxation ($\tau^*_{180^\circ\mathrm{C}} \geq \tau^*_{170^\circ\mathrm{C}}$):** Indicates permanent radical C=C crosslinking outpacing associative transesterification at elevated temperature. *Corrective Branch:* Increase α-tocopherol stabilizer from 0.20 wt% to 0.35 wt% and enforce strict nitrogen purging during post-cure and hot-pressing.
3. **Reprocessing Failure ($\mathrm{Retention}_{\mathbf{C2}} < 80.0\%$ or Porous Consolidation):** Indicates insufficient free galloyl/aliphatic −OH exchange sites or moisture-induced steam voids. *Corrective Branch:* Shift equivalence ratio

from $R = 0.95$ to $R = 1.00$ to increase residual phenolic −OH density and re-verify vacuum drying of biochar (< 0.5 wt% moisture).

4. **Filler Agglomeration Drop ($\sigma_{\max,\textbf{RUN-}n} < \sigma_{\max,\textbf{RUN-0}}$):** Identifies the exact rheological/percolation ceiling for ball-milled hemp biochar; lock optimal composite loading at the highest concentration preceding strength drop-off.

8. Verified Baseline Conclusions vs. Pre-Experimental Boundaries

- **Verified Chemical & Literature Constants:** Associative transesterification enables thermoset-like solvent resistance alongside thermal malleability above the topology freezing transition (Montarnal et al., 2011). Polyphenolic tannic acid networks combined with epoxidized vegetable oils and cyclic anhydrides form catalyst-free vitrimers via abundant −OH self-catalysis (Zhao et al., 2024; Berne et al., 2022). Stoichiometric equations (1)–(7) are exact conservation laws.
- **Pre-Experimental Status `[X THEORETICAL]`:** The specific four-component integration of EHSO, tannic acid, itaconic anhydride, α-tocopherol, and steam-activated hemp biochar (`CF-TAIA-95`) has not yet been physically synthesized or tested. No mechanical strength, T_g, or activation energy values are claimed prior to execution of Tables 4–7.
- **Regulatory Certification Boundary:** Under USDA Organic and Canada Organic Regime (COR) rules, cured thermoset/vitrimer polymers cannot be certified as organic agricultural products because epoxidation and crosslinking synthesize new covalent macromolecules; however, 100% of upstream agricultural feedstocks (cold-pressed hemp seed oil, oak-gall/chestnut tannic acid, fermentation dextrose for itaconic acid, and hemp hurd biochar) can be sourced as certified organic inputs processed via enzymatic lipase catalysis (Ankudey et al., 2006) and steam activation.

9. Replication & Multi-Lot Calibration Standards

To ensure independent open-source reproducibility across laboratories:

1. Always report measured lot values (EEW_{meas}, HV_{meas}, P_{IA}, biochar BET surface area, and post-drying moisture content) alongside mechanical results.
2. Never introduce multi-scale carbon hybrids (e.g., nanosheets + short fibers + biochar) until single-filler `RUN-0` through `RUN-3` baselines are published and replicated across at least $n = 5$ ASTM D638 tensile specimens per condition.
3. Deposit raw FTIR spectra (`.csv`/`.jdx`), DMA curves, and stress-relaxation datasets under open CC BY 4.0 repositories (Zenodo / GitHub) referencing `Hempoxies CF-TAIA-95 (v.25-CF)`.

10. Open-Source Reporting, Versioning & References

Document Version History: `v.25-CF-Rev1.0` (November 24, 2026) — Initial open-source LaTeX specification establishing Variant `CF-TAIA-95` ($R = 0.95$, 60 : 40 tannic acid / itaconic anhydride, 0 wt% FGE, 0 wt% metal catalyst, 0.20 wt% α-tocopherol, 0.0–3.0 wt% steam-activated hemp biochar). Public correction reports: `corrections@marielandryspysho`

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