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Hempoxies Variant CF-TAIA-125G80 (v.28-CF-Rev4.2-Opt)

Fully Ester-Converted, Zero-Percolation, Catalyst-Free Epoxidized Hemp Seed Oil Vitrimer Nanocomposite ($R_{\text{protic}} = 1.25$, $f_{\text{graft}} = 0.80$, 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: [THEORETICAL]** Pre-Experimental Specification (TRL 1–2). All molar masses, equivalent weights, protic conservation identities, gravimetric devolatilization bounds, Flory–Stockmayer percolation thresholds, and dynamic ester junction fractions are algebraically verified and kinetic bounds are anchored to peer-reviewed literature; however, this integrated formulation has not yet undergone wet-lab synthesis or physical ASTM characterization and must be treated strictly as a falsifiable experimental hypothesis until validated.

1. Observation of Anomalies in Prior Baselines & Technical Audit

Systematic chemical auditing of early Hempoxies baselines—including the Epoxidized Hemp Seed Oil (EHSO) / citric acid / zinc acetate / furfuryl glycidyl ether (FGE) system (v.17.2), sebacic acid / $Zn(acac)_2$ variants (H43), and multi-filler hybrid matrices (v.21–v.24)—previously resolved five foundational anomalies:

(i) solid carboxylic acid phase separation and dehydration micro-voids above 150 °C,
(ii) monofunctional FGE ($f = 1$) crosslink-density dilution and epichlorohydrin organochlorine waste [6, 12],
(iii) transition-metal catalyst chelation/leaching at oxidized carbon interfaces,
(iv) caustic KOH/HCl chemical activation waste in carbon nanosheets [5], and
(v) aerobic radical autoxidation of residual EHSO C = C bonds during hot-press reprocessing.

1.1 Seven Technical Defects Resolved & Equations-Driven Optimization (Rev4.2-Opt) [THEORETICAL]

An adversarial polymer-physics, rheological, and process audit of v.27-CF-Rev3.0 (CF-TAIA-105G55) exposed seven critical blind spots that would cause residual network locking, stabilizer precipitation, or solvent micro-voids in the wet lab. All seven are addressed in v.28-CF-Rev4.0/Rev4.1, and **v.28-CF-Rev4.2-Opt** closes the gravimetric devolatilization equations (Eqs. 19–21) and reconciles purity-dependent mass rounding:

1. **True Elimination of Static Aryl-Ether Percolation ($R_{\text{protic}} = 1.25$, $f_{\text{graft}} = 0.80$):** Under Flory–Stockmayer gelation theory ($p_A p_B = [(f_A - 1)(f_B - 1)]^{-1}$) [16], leaving 45% of tannic acid's $\bar{f}_{\text{TA}} \approx 25.0$ phenolic −OH groups ungrafted leaves $f_{\text{ArOH,free}} = 11.25$ permanent ether-forming sites per tannic acid core. Paired with EHSO ($\bar{f}_{\text{EHSO}} \approx 4.60$), the static percolation product in Rev3.0 ($R_{\text{protic}} = 1.05$, $f_{\text{graft}} = 0.55$) reached $p_A p_B = 0.3778$ (13.94× above $p_{c,\text{static}} = 0.0271$). Shifting to $R_{\text{protic}} = 1.25$, $f_{\text{graft}} = 0.80$ yields $\text{Eq}_{\text{COOH}} = 1.25 \times 0.80 = 1.0000$ eq carboxylic acid per 1.0000 eq epoxide ($\phi_{\text{ester}} = 100.0\%$, $0.00 \times p_{c,\text{static}}$) while preserving 0.2500 eq/eq epoxide free catalytic phenolic −OH (5.0 per TA molecule) plus 1.0000 eq secondary aliphatic −OH (1.2500 eq total catalytic −OH). Even at a 90% steric grafting ceiling ($f_{\text{graft,eff}} = 0.72$), static ethers yield $p_A p_B = 0.0286 < p_{c,\text{static}} = 0.0463$ ($0.62 \times p_c$).
2. **Split-Phase Antioxidant Delivery:** Dosing 50% of α-tocopherol and 100% of L-ascorbyl palmitate ($T_m \approx$ 115–118 °C) into Step 2's polar aprotic n-propyl acetate medium protects exocyclic vinylidene ($C = CH_2$) bonds during 90 °C pre-esterification and 60 °C Stage A stripping while avoiding L-ascorbyl palmitate micro-crystallinity in 70 °C EHSO. The remaining 50% α-tocopherol wets biochar micropores in EHSO.

3. **Three-Stage Devolatilization with 95 °C Dwell & Exact Gravimetric Gate:** Enforces dual mass-balance controls ($m_{\text{solv,res}} \leq 0.20$ g, i.e., $\geq 99.5\%$ initial solvent removal, AND sequential 5-min drift $\Delta m_{5\text{min}} < 0.03$ g) plus a 30 min 95 °C N_2-sweep pre-gelation dwell below *n*-propyl acetate's normal boiling point (101.6 °C).
4. **Exotherm-Safe Casting Thickness (≤ 3.0 mm) & Stepped Cure Ramp (105 °C/2.0 h + 125 °C/2.5 h + 150 °C/3.5 h):** Dissipates carboxylic–oxirane ring-opening enthalpy ($\Delta H \approx -80$ to -95 kJ/mol epoxide) in thin plaques below the 150 °C ceiling.
5. **In-Process Pre-Hardener ATR-FTIR Anhydride Gate ($\geq 90\%$ Conversion):** Verifies cyclic anhydride ring-opening (1780/1855 cm^{-1} attenuation $\geq 90\%$) at the end of Step 2 prior to EHSO addition.
6. **Long-Time Stress-Relaxation Completeness Gate ($G(3\tau^*)/G_0 \leq 0.08$):** Tests absence of a percolated permanent ether sub-network at $t = 3\tau^*$.
7. **Complete Equation, Purity-Rounding, Glyph, Header, and Table Integrity ($1 = 1 = 1$):** Adds gravimetric mass-balance equations (Eqs. 19–21), explicitly notes purity-dependent IA mass (53.38 g at $P_{\text{IA}} = 100.0\%$ yielding $M_{\text{matrix}} = 193.89$ g vs. 53.92 g at $P_{\text{IA}} = 99.0\%$ yielding $M_{\text{matrix}} = 194.43$ g), and restores all symbols and table layouts.

Table 1: Side-by-Side Technical Evolution (CF-TAIA-95 Rev1.1 vs. Rev2.0 vs. Rev3.0 vs. Rev4.2-Opt)

Design Parameter	**CF-TAIA-95 (v.25-CF-Rev1.1)**	**CF-TAIA-105G (v.26-CF-Rev2.0)**	**CF-TAIA-105G55 (v.27-CF-Rev3.0)**	**CF-TAIA-125G80 (v.28-CF-Rev4.2-Opt)**
Protic Stoichiometry & Graft Fraction	Nominal $R = 0.95$; post-graft $R_{\text{eff}} \approx 0.570$ (43% oxirane deficit)	$R_{\text{protic}} = 1.05$, $f_{\text{graft}} = 0.35$ (36.8% dynamic ester; 63.2% static ether)	$R_{\text{protic}} = 1.05$, $f_{\text{graft}} = 0.55$ (57.8% dynamic ester; 42.2% static ether: $13.94 \times p_{c,\text{static}}$)	$R_{\text{protic}} = 1.25$, $f_{\text{graft}} = 0.80$ (100.0% dynamic β-hydroxy ester; $0.00 \times p_{c,\text{static}}$)
TA / IA Charge (100.00 g EHSO)	18.47 g TA + 20.28 g IA ($M_{\text{matrix}} = 138.75$ g)	34.02 g TA + 19.61 g IA ($M_{\text{matrix}} = 153.64$ g)	34.03 g TA + 30.83 g IA ($M_{\text{matrix}} = 164.85$ g)	40.51 g TA + 53.38 g IA ($M_{\text{matrix}} = 193.89$ g at $P_{\text{IA}} = 100\%$)
Solvent Strip & Devolatilization	Single 25 min strip post-EHSO mix (viscosity spike)	80–85% Stage A strip (glassy taffy phase-separation risk)	65–70% Stage A strip + 10 min mix + 15 min Stage B degas	65–70% Stage A strip + 15 min Stage B degas + 30 min 95 °C N_2 dwell ($\Delta m_{5\text{min}} < 0.03$ g)
Antioxidant Delivery	α-Tocopherol only ($0.20 + 0.04\, w_{\text{filler}}$ wt%) in EHSO	α-Tocopherol +0.10 wt% L-ascorbyl palmitate in EHSO	α-Tocopherol +0.10 wt% L-ascorbyl palmitate in EHSO	Split-phase: 50% Tocoph. +100% AP in Step 2 pre-hardener; 50% Tocoph. in EHSO/biochar
Cure & Reprocess Envelope	Cure 120/160 °C; Reprocess 165 °C, 15 MPa, 60–90 min	Cure 120/160 °C (4 + 4 h); Reprocess 160 °C, 15 MPa, 60 min	Cure 120/150 °C (3 + 4 h); Reprocess 155 °C, 15 MPa, 60 min	Stepped cure 105/125/150 °C (2.0 + 2.5 + 3.5 h); ≤ 3.0 mm plaque; Reprocess 155 °C, 15 MPa, 60 min

2. Definition of Intelligence & Engineering Requirements

Hempoxies Variant CF-TAIA-125G80 (v.28-CF-Rev4.2-Opt) is engineered to satisfy eight deterministic specifications:

1. **100% Bio-Based Carbon Architecture:** Zero petrochemical reactive diluents (0.0 wt% FGE) and zero petrochemical cyclic anhydrides (0.0 wt% methyl nadic anhydride), utilizing exclusively bio-renewable monomers and a stripped bio-derived ester processing aid (*n*-propyl acetate) [2, 15].
2. **Zero External Metal or Alkali-Ash Catalysts (0.0 wt%):** Associative transesterification proceeds strictly via internal neighboring-group participation (NGP) and abundant free phenolic/aliphatic −OH self-catalysis [1, 2, 3], verified by demineralized biochar (ash < 0.5 wt%).
3. **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, citric-acid demineralized, ball-milled, 500-mesh ($D_{90} \leq 25$ μm) hemp biochar.
4. **Zero-Percolation Full-Ester Stoichiometry ($R_{\text{protic}} = 1.25$, $f_{\text{graft}} = 0.80$):** Converts 80% of TA phenolic −OH groups into carboxylic acid arms in anhydrous *n*-propyl acetate (90 °C, 60 min), delivering 1.0000 eq carboxylic acid per 1.0000 eq epoxide ($\phi_{\text{ester}} = 100.0\%$, $0.00 \times p_{c,\text{static}}$) while retaining 0.2500 eq/eq epoxide free catalytic phenolic −OH [2, 16].
5. **Deterministic Lot-to-Lot Stoichiometric Calibration:** Exact algebraic recalculation tied to measured Epoxy Equivalent Weight (EEW_{meas} via ASTM D1652) [20] and Hydroxyl Value (HV_{meas} via ASTM E222) [21] so natural feedstock variance never shifts $R_{\text{protic}} = 1.25$ or $f_{\text{graft}} = 0.80$.
6. **Split-Phase Lipophilic Radical Stabilization & Vinylidene Conservation:** Biochar-compensated α-tocopherol ($(0.20+0.04\, w_{\text{filler}})$ wt%) paired synergistically with 0.10 wt% L-ascorbyl palmitate [13, 14] across both pre-hardener synthesis and EHSO compounding under a 150 °C thermal ceiling protects residual EHSO C = C and itaconate $C = CH_2$ bonds [11].
7. **Exotherm-Controlled, Void-Free Plaque Fabrication:** Three-stage devolatilization (60 °C Stage A strip, 60 °C Stage B degas, 95 °C N_2-sweep pre-gelation dwell) and ≤ 3.0 mm stepped curing (105/125/150 °C) prevent

solvent boiling voids and adiabatic exotherm runaway.

8. **Verified Closed-Loop Reprocessability:** Gel fraction $G \geq 92.0\%$ [25], monotonic Arrhenius stress-relaxation decay (τ^*) between 145 °C and 165 °C with long-time residual modulus $G(3\tau^*)/G_0 \leq 0.08$, and $\geq 85.0\%$ tensile strength retention across two mechanical hot-press cycles at 155 °C [2, 22].

3. Formulation Hypothesis (Variant CF-TAIA-125G80, v.28-CF-Rev4.2-Opt)

Central Falsifiable Hypothesis [THEORETICAL]: Pre-esterifying vacuum-dried tannic acid ($R_{\text{protic}} = 1.25$ phenolic −OH equivalents per epoxy equivalent) with itaconic anhydride at a galloyl grafting fraction of $f_{\text{graft}} = 0.80$ in anhydrous aprotic *n*-propyl acetate (90 °C, 60 min) stabilized with 50% of the target α-tocopherol dose plus 100% of the L-ascorbyl palmitate (0.10 wt% of M_{matrix}), followed by a moderate Stage A vacuum pre-strip (60 °C, 60–80 mbar, 12 min; 65–70% solvent removal), blending with enzymatically epoxidized hemp seed oil (EHSO, 100.00 g base, 0 wt% FGE, 0 wt% metal catalyst) pre-dispersed with the remaining 50% α-tocopherol and a single-variable loading ladder ($0.0, 0.5, 1.5, 3.0$ wt%) of citric-acid demineralized 500-mesh steam-activated hemp biochar, devolatilizing via Stage B vacuum (60 °C, 40–50 mbar, 15 min) and a 95 °C N_2-sweep dwell (30 min), and curing in ≤ 3.0 mm plaques via a three-stage N_2-blanketed cycle (105 °C/2.0 h + 125 °C/2.5 h + 150 °C/3.5 h) yields a void-free, 100% bio-based, catalyst-free vitrimer nanocomposite achieving $G \geq 92.0\%$, complete stress relaxation ($G(3\tau^*)/G_0 \leq 0.08$), and $\geq 85.0\%$ tensile strength retention across two closed-loop hot-press reprocessing cycles at 155 °C.

Variant Nomenclature Key: CF = Catalyst-Free (0.0 wt% metal salts, < 0.5 wt% biochar ash); TAIA = Tannic Acid + Itaconic Anhydride galloyl-itaconate pre-hardener; 125G80 = Protic-to-epoxy equivalence ratio $R_{\text{protic}} = 1.25$ with zero-percolation Graft-balanced anhydride ratio $f_{\text{graft}} = 0.80$; v.28-CF-Rev4.2-Opt = Sequential Hempoxies specification version 28, equation-closed fourth catalyst-free revision.

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) [4], record measured Certificate of Analysis (CoA) or titration values in Table 2 prior to weighing.

Table 2: Precursor Reference Constants and Measured Lot Calibration Log (CF-TAIA-125G80, v.28-CF-Rev4.2-Opt)

Precursor	**Lot/Batch ID**	**Reference Benchmark**	**Measured Lot Value**	**Units**
EHSO (ASTM D1652) [20]	_____	O_o = 7.62 wt% (EEW = 209.97)	O_o = ____; EEW_{meas} = ____	wt%; g/eq
Tannic Acid (ASTM E222) [21]	_____	HV = 824.51 (HEW = 68.05)	HV_{meas} = ____; $\text{HEW}_{\text{TA,meas}}$ = ____	mg KOH/g; g/eq
Itaconic Anhydride	_____	AEW_{IA} = 112.08 (≥ 99.0% pure)	P_{IA} = ____; H_2O_{dry} = ____	g/eq; wt%
Hemp Biochar (Demin.)	_____	$D_{90} \leq 25$ μm; Ash < 0.5%; H_2O < 0.5%	Ash = ___; H_2O = ___; S_{BET} = ___	wt%; wt%; m^2/g
n-Propyl Acetate	_____	Anhydrous (≥ 99.5%, H_2O < 0.05%)	Karl Fischer / CoA H_2O = _____	wt%
α-Tocopherol	_____	Bio-derived, ≥ 96.0% purity	CoA Purity = _____	wt%
L-Ascorbyl Palmitate	_____	Bio-derived, ≥ 98.0% purity	CoA Purity = _____	wt%

4.2 Exact Algebraic Recalculation & Gravimetric Devolatilization Equations

For any chosen base mass of EHSO (m_{EHSO} in grams) and target biochar filler loading w_{filler} (wt% relative to M_{matrix}), compute exact reagent masses and solvent/devolatilization bounds using Eqs. (1)–(11) and Eqs. (19)–(21):

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

$$\text{HEW}_{\text{TA,meas}} = \frac{56.106 \times 1000}{\text{HV}_{\text{meas}}} = \frac{56106}{\text{HV}_{\text{meas}}} \tag{2}$$

$$\text{Eq}_{\text{epoxy}} = \frac{m_{\text{EHSO}}}{\text{EEW}_{\text{meas}}} \tag{3}$$

$$\text{Eq}_{\text{TA},0} = R_{\text{protic}} \times \text{Eq}_{\text{epoxy}} = 1.2500 \times \left(\frac{m_{\text{EHSO}}}{\text{EEW}_{\text{meas}}}\right) \tag{4}$$

$$m_{\text{TA}} = \text{Eq}_{\text{TA},0} \times \text{HEW}_{\text{TA,meas}} = 1.2500 \times \left(\frac{m_{\text{EHSO}}}{\text{EEW}_{\text{meas}}}\right) \times \text{HEW}_{\text{TA,meas}} \tag{5}$$

$$\text{Eq}_{\text{IA}} = f_{\text{graft}} \times \text{Eq}_{\text{TA},0} = 0.8000 \times 1.2500 \times \text{Eq}_{\text{epoxy}} = 1.0000 \times \left(\frac{m_{\text{EHSO}}}{\text{EEW}_{\text{meas}}}\right) \tag{6}$$

$$m_{\text{IA}} = \text{Eq}_{\text{IA}} \times \frac{112.08}{P_{\text{IA}}/100} = 112.0800 \times \left(\frac{m_{\text{EHSO}}}{\text{EEW}_{\text{meas}}}\right) \times \left(\frac{100}{P_{\text{IA}}}\right) \tag{7}$$

$$M_{\text{matrix}} = m_{\text{EHSO}} + m_{\text{TA}} + m_{\text{IA}} \tag{8}$$

$$m_{\text{tocopherol}} = \left(\frac{0.20 + 0.04 \times w_{\text{filler}}}{100}\right) M_{\text{matrix}} \quad \text{(split 50\% Step 2 / 50\% Step 4)} \tag{9}$$

$$m_{\text{AP}} = \left(\frac{0.10}{100}\right) M_{\text{matrix}} \quad \text{(100\% in Step 2)} \tag{10}$$

$$m_{\text{biochar}} = \left(\frac{w_{\text{filler}}}{100}\right) M_{\text{matrix}} \tag{11}$$

Post-Pre-Esterification Functional Group Conservation Check: During aprotic pre-esterification (90 °C, 60 min), each mole of IA ring-opens by consuming 1 eq of TA phenolic −OH to yield 1 eq of carboxylic acid (−COOH):

$$\text{Ar-OH} + \text{C}_5\text{H}_4\text{O}_3 \text{ (IA)} \longrightarrow \text{Ar-O-C(=O)-C(=CH}_2\text{)-CH}_2\text{-COOH}$$

After reacting Eq_{IA} moles of IA with $\text{Eq}_{\text{TA},0}$ equivalents of TA phenolic −OH, the pre-hardener contains $\text{Eq}_{\text{COOH}} = 1.0000\,\text{Eq}_{\text{epoxy}}$ fast-reacting carboxylic acid equivalents (yielding 100.0% dynamic β-hydroxy ester junctions) and $\text{Eq}_{\text{ArOH,free}} = (1.2500 - 1.0000)\,\text{Eq}_{\text{epoxy}} = 0.2500\,\text{Eq}_{\text{epoxy}}$ free catalytic phenolic −OH equivalents, summing to $1.0000 + 0.2500 = 1.2500\,\text{Eq}_{\text{epoxy}}$ total protic equivalents ($R_{\text{protic}} = 1.25$).

Worked Lot-Recalculation Sanity Check ($m_{\text{EHSO}} = 100.00$ g; $w_{\text{filler}} = 1.5$ wt%): If a supplier CoA reports $O_o = 7.44$ wt% ($\text{EEW}_{\text{meas}} = 215.05$ g/eq), $\text{HV}_{\text{meas}} = 750.0$ mg KOH/g ($\text{HEW}_{\text{TA,meas}} = 74.81$ g/eq), and $P_{\text{IA}} = 99.5\%$, then $\text{Eq}_{\text{epoxy}} = 0.4650$ eq, $\text{Eq}_{\text{TA},0} = 0.5813$ eq, $m_{\text{TA}} = 43.48$ g, $\text{Eq}_{\text{IA}} = 0.4650$ eq, $m_{\text{IA}} = 52.38$ g, $M_{\text{matrix}} = 100.00 + 43.48 + 52.38 = 195.86$ g, $w_{\text{tocopherol}} = 0.20 + 0.04(1.5) = 0.26$ wt%, $m_{\text{tocopherol}} = 0.51$ g (0.255 g Step 2 +0.255 g Step 4), $m_{\text{AP}} = 0.20$ g, and $m_{\text{biochar}} = 2.94$ g.

4.3 Master Batch Weighing Table ($R_{\text{protic}} = 1.25$, $f_{\text{graft}} = 0.80$; Benchmark Constants)

Execute the 1/4-scale screening batch (25.00 g EHSO, values in brackets) first to verify pre-hardener anhydride conversion, cured FTIR $\text{C}=\text{CH}_2$ retention, and gelation before casting full 100.00 g EHSO plaques. Note that at reference constants $O_o = 7.62$ wt% and HV = 824.51, $m_{\text{TA}} = 40.51$ g and $m_{\text{IA}} = 53.38$ g at $P_{\text{IA}} = 100.0\%$ ($M_{\text{matrix}} = 193.89$ g; for $P_{\text{IA}} = 99.0\%$, Eq. (7) gives $m_{\text{IA}} = 53.92$ g and $M_{\text{matrix}} = 194.43$ g).

Table 3: Master Stoichiometric Batch Sheet for Hempoxies Variant CF-TAIA-125G80, v.28-CF-Rev4.2-Opt (EEW = 209.97 g/eq, $\text{HEW}_{\text{TA}} = 68.05$ g/eq, $\text{AEW}_{\text{IA}} = 112.08$ g/eq, $M_{\text{matrix}} = 193.89$ g [48.47 g])

Run ID	Biochar	m_{EHSO} (g)	m_{TA} (g)	m_{IA} (g)	Tocoph. (g)*	Asc. Palm. (g)†	Biochar (g)	Actua
RUN-0 (Ctrl)	0.0 wt%	100.00 [25.00]	40.51 [10.13]	53.38 [13.34]	0.39 [0.10]	0.19 [0.05]	0.00 [0.00]	_
RUN-1 (Low)	0.5 wt%	100.00 [25.00]	40.51 [10.13]	53.38 [13.34]	0.43 [0.11]	0.19 [0.05]	0.97 [0.24]	_
RUN-2 (Mid)	1.5 wt%	100.00 [25.00]	40.51 [10.13]	53.38 [13.34]	0.50 [0.13]	0.19 [0.05]	2.91 [0.73]	_
RUN-3 (High)	3.0 wt%	100.00 [25.00]	40.51 [10.13]	53.38 [13.34]	0.62 [0.16]	0.19 [0.05]	5.82 [1.45]	_

*Split-phase α-tocopherol: charge 50% in Step 2 (pre-esterification) and 50% in Step 4 (EHSO/biochar). †Charge 100% L-ascorbyl palmitate in Step 2.

4.4 Literature-Optimized Step-by-Step Synthesis & Curing Procedure

1. **Biochar Citric-Acid Demineralization & Vacuum Drying (105–110 °C, 4.0 h):** Wash ball-milled, 500-mesh ($D_{90} \leq 25$ μm) steam-activated hemp biochar in 2.0 wt% aqueous bio-fermented citric acid (60 °C, 30 min) to

remove native K_2CO_3/$CaCO_3$ alkali ash, rinse with deionized water to neutral filtrate (pH 6.5–7.5), and vacuum-dry together with raw tannic acid (5–10 wt% ambient moisture) at 105–110 °C for 4.0 h. Cool inside a sealed P_2O_5 or freshly regenerated silica-gel desiccator and weigh immediately upon removal so ambient moisture cannot hydrolyze itaconic anhydride.

2. **Stabilized Aprotic Pre-Esterification & In-Process FTIR Check (90 °C, 60 min):** Charge dry tannic acid (40.51 g), dry itaconic anhydride (53.38 g), 50% of the batch α-tocopherol dose (0.20–0.31 g), and 100% of the L-ascorbyl palmitate (0.19 g) into a dry round-bottom flask fitted with a reflux condenser and dry N_2 inlet, followed by 55–60 mL anhydrous *n*-propyl acetate per 100 g EHSO batch (14–15 mL at 1/4 scale; ≈ 49–53 g at $\rho = 0.888$ g/mL). Stir magnetically at 90 °C for 60 min (adapted from Li, Ju & Zhang, 2024) [2] to graft IA onto 80% of TA phenolic −OH sites, forming a homogeneous, amber liquid galloyl-itaconate polycarboxylic pre-hardener. Withdraw a 10 μL aliquot and verify ≥ 90% attenuation of cyclic anhydride bands (1780/1855 cm^{-1}) via ATR-FTIR before proceeding. **Critical Prohibition:** Never use ethanol, methanol, or any protic alcohol, which causes irreversible anhydride alcoholysis into monofunctional alkyl itaconates.
3. **Moderate Stage A Solvent Pre-Strip from Isolated Pre-Hardener (60 °C, 60–80 mbar, 12 min):** While the stabilized galloyl-itaconate pre-hardener is still isolated from EHSO, apply moderate vacuum (60–80 mbar) at 60 °C for 12 min to strip 65–70% of the *n*-propyl acetate (≈ 36–42 mL recovered), leaving a flowable syrup (≈ 18–20 mL residual co-solvent, ≈ 82 wt% solids) that prevents glassy phase separation during EHSO blending.
4. **Pre-Wet Biochar in EHSO & Dissolve Remaining α-Tocopherol (70 °C → 65 °C, 15 + 10 + 10 min):** Warm EHSO (100.00 g) to 70 °C. For filled batches (RUN-1 to RUN-3), disperse dry demineralized hemp biochar directly into pure EHSO via bath sonication or high-shear mixing for 15 min first so triglycerides wet biochar micropores before stabilizer addition. Add the remaining 50% α-tocopherol dose (0.20–0.31 g) and stir at 70 °C for 10 min until optically uniform. Cool to 65 °C, pour in the warm flowable galloyl-itaconate pre-hardener syrup, and homogenize vigorously for 10 min.
5. **Stage B Vacuum Degas & 95 °C N_2-Sweep Pre-Gelation Devolatilization (60 °C/15 min+95 °C/30 min):** Strip the remaining 18–20 mL co-solvent under vacuum (40–50 mbar) at 60 °C for 15 min until gross bubbling ceases, then vent with dry N_2 and hold at 95 °C (6.6 °C below *n*-propyl acetate's 101.6 °C boiling point) under a gentle dry N_2 sweep for 30 min until gravimetric mass confirms ≥ 99.5% total solvent removal ($m_{solv,res} \leq 0.20$ g above theoretical non-volatile mass m_{nonvol} AND sequential 5-min mass drift $\Delta m_{5min} < 0.03$ g). Pour slowly down the tilted wall of a preheated PTFE or silicone mold with cavity depth ≤ 3.0 mm.
6. **Exotherm-Controlled Three-Stage Thermal Cure (105 °C/2.0 h+125 °C/2.5 h+150 °C/3.5 h under N_2):** Ramp at 2 °C/min to 105 °C and hold for 2.0 h (controlled carboxylic–oxirane ring-opening without adiabatic exotherm spike), ramp at 2 °C/min to 125 °C and hold for 2.5 h (network gelation), then ramp at 2 °C/min to 150 °C and hold for 3.5 h under a continuous dry N_2 blanket to drive esterification to completion while remaining 18 °C below the ≈ 168 °C IA → citraconic anhydride isomerization threshold [11] and suppressing thermal vinylidene radical self-polymerization.

5. Data Collection Protocols & Analytical Instruments

Record all experimental process checkpoints and analytical measurements directly in Tables 4–8 across all four runs (RUN-0 through RUN-3).

Table 4: Thermal & Mixing Process Checkpoint Log (CF-TAIA-125G80, v.28-CF-Rev4.2-Opt)

Process Stage	Setpoint / Target	Actual Temp / Vac	Duration / Δm_{5min}	Operator Notes / Gate Status
1. Demin. & Vac Dry	105–110 °C	_____	4.0 h	_____
2. Aprotic Pre-Esterif.	90 °C (N_2)	_____	60 min	FTIR ≥ 90% gate: _____
3. Stage A Pre-Strip	60 °C; 60–80 mbar	_____	12 min (65–70% strip)	_____
4. Biochar / EHSO Mix	70 → 65 °C	_____	15 + 10 + 10 min	_____
5. Stage B + 95 °C Dwell	60 °C vac + 95 °C N_2	_____	15 min + 30 min	$m_{solv,res} \leq 0.20$ g; $\Delta m_{5min} < 0.03$ g
6. Cure Stage 1A / 1B	105 °C / 125 °C (N_2)	_____	2.0 h + 2.5 h	Plaque ≤ 3.0 mm: _____
7. Stage 2 Post-Cure	150 °C (N_2, < 168 °C)	_____	3.5 h	_____

Table 5: ATR-FTIR Cure Completeness & Vinylidene Retention Log (4000–600 cm^{-1}, ASTM E1252) [24]

Run ID	**Anhydride** (1780/1855 cm^{-1})	**Oxirane** (825–915 cm^{-1})	**Ester** C = O (1735 cm^{-1})	**Vinylidene** C = CH_2 (1635/815 cm^{-1})
Uncured Ref	Strong doublet	Strong band	Baseline	Strong intact (100%)
RUN-0 (0.0%)	_____	_____	_____	_____
RUN-1 (0.5%)	_____	_____	_____	_____
RUN-2 (1.5%)	_____	_____	_____	_____
RUN-3 (3.0%)	_____	_____	_____	_____

Table 6: Gel Fraction, Swelling Ratio (24 h THF / Toluene, ASTM D2765) [25] & DSC OIT (180 °C, ASTM E1858) [26]

Run ID	**Solvent**	W_0 **Dry (g)**	W_s **Swell (g)**	W_d **Final (g)**	**Gel** G **(%) / Swell** Q	$OIT_{180\,°C}$ **(min) / Gate**
RUN-0 (0.0%)	_____	_____	_____	_____	_____	_____
RUN-1 (0.5%)	_____	_____	_____	_____	_____	_____
RUN-2 (1.5%)	_____	_____	_____	_____	_____	_____
RUN-3 (3.0%)	_____	_____	_____	_____	_____	_____

6. Quantitative Analysis & Governing Equations

Evaluate raw laboratory measurements using polymer-physics, Flory–Stockmayer percolation, dynamic-network, and gravimetric mass-balance relations:

$$G(\%) = \left(\frac{W_d}{W_0}\right) \times 100 \quad \text{and} \quad Q = \frac{W_s - W_d}{W_d} \tag{12}$$

$$\phi_{\text{ester}} = \min(1.0000,\ R_{\text{protic}} f_{\text{graft}}) = \min(1.0000,\ 1.25 \times 0.80) = 1.0000 \text{ (100.0\% dynamic } \beta\text{-hydroxy ester)} \tag{13}$$

$$p_{c,\text{static}} = \frac{1}{(\bar{f}_{\text{TA}}(1 - f_{\text{graft}}) - 1)(\bar{f}_{\text{EHSO}} - 1)} \quad \text{(static aryl-ether percolation threshold)} \tag{14}$$

$$\nu_e = \frac{E'_r}{3R_{\text{gas}}T} \tag{15}$$

$$\ln(\tau^*) = \ln(\tau_0) + \frac{E_a}{R_{\text{gas}}T} \tag{16}$$

$$\frac{G(t)}{G_0} = \exp\left(-\frac{t}{\tau^*}\right) \quad \text{with completeness gate } \frac{G(3\tau^*)}{G_0} \leq 0.08 \tag{17}$$

$$\text{Strength Retention (\%)} = \left(\frac{\sigma_{\text{Cycle n}}}{\sigma_{\text{Virgin}}}\right) \times 100 \tag{18}$$

$$m_{\text{nonvol}} = M_{\text{matrix}} + m_{\text{tocopherol}} + m_{\text{AP}} + m_{\text{biochar}} \tag{19}$$

$$m_{\text{solv,res}} = m_{\text{measured,pre-cure}} - m_{\text{nonvol}} \leq 0.20 \text{ g} \tag{20}$$

$$\text{Solvent Removal (\%)} = \left(1 - \frac{m_{\text{solv,res}}}{m_{\text{solv,0}}}\right) \times 100 \geq 99.5\% \tag{21}$$

where $R_{\text{gas}} = 8.314$ J mol^{-1} K^{-1}, $T = T_g$[K] + 40 K for rubbery-plateau crosslink density ν_e in Eq. (15), τ^* in Eq. (16) is recorded when normalized relaxation modulus decays to $G(t)/G_0 = 1/e \approx 0.368$ across isothermal temperatures 145 °C, 155 °C, and 165 °C [1, 2, 16], and $m_{\text{solv,0}}$ in Eq. (21) is the initial charged *n*-propyl acetate mass.

Table 7: DMA Thermomechanical & Isothermal Stress-Relaxation Log (ASTM E1640) [23]

Run ID	$E'_{25\,°C}$ **(MPa)**	T_g **(°C)**	$\tau^*_{145\,°C}$ / $\tau^*_{155\,°C}$ / $\tau^*_{165\,°C}$ **(s)**	E_a **(kJ/mol)**	$G(3\tau^*)/G_0$ **(≤ 0.08)**
RUN-0 (0.0%)	_____	_____	_____	_____	_____
RUN-1 (0.5%)	_____	_____	_____	_____	_____
RUN-2 (1.5%)	_____	_____	_____	_____	_____
RUN-3 (3.0%)	_____	_____	_____	_____	_____

Table 8: Closed-Loop Reprocessing Cycle Log (Hot-Press at 155 °C, 15 MPa, 60 min under N_2; ASTM D638) [22]

Run ID	Cycle	E (MPa)	σ_{max} (MPa)	Retention (%)	$\geq 85\%$ Gate / Void Check
RUN-0 (0.0%)	Virgin (C0)	_____	_____	100.0% (Ref)	Reference
	Cycle 1 (C1)	_____	_____	_____	_____
	Cycle 2 (C2)	_____	_____	_____	_____
RUN-1 (0.5%)	Virgin (C0)	_____	_____	100.0% (Ref)	Reference
	Cycle 1 (C1)	_____	_____	_____	_____
	Cycle 2 (C2)	_____	_____	_____	_____
RUN-2 (1.5%)	Virgin (C0)	_____	_____	100.0% (Ref)	Reference
	Cycle 1 (C1)	_____	_____	_____	_____
	Cycle 2 (C2)	_____	_____	_____	_____
RUN-3 (3.0%)	Virgin (C0)	_____	_____	100.0% (Ref)	Reference
	Cycle 1 (C1)	_____	_____	_____	_____
	Cycle 2 (C2)	_____	_____	_____	_____

7. Decision Loop & Falsification Criteria (Rejection Gates)

Under the Landry Hallucination-Free Protocol [19], Variant CF-TAIA-125G80 (v.28-CF-Rev4.2-Opt) must be rejected or reformulated if any experimental run violates the following falsification thresholds:

1. **Incomplete Pre-Hardener Grafting (Step 2 FTIR** 1780/1855 cm^{-1} **Attenuation** $< 90\%$**):** Indicates moisture hydrolysis of IA or steric arrest before $f_{graft,eff} \geq 0.72$. *Corrective Branch:* Extend Step 2 aprotic pre-esterification at 90 °C from 60 min to 90 min under dry N_2 and re-verify desiccator moisture seals.
2. **Incomplete Network Formation (**$G < 92.0\%$**) or Excess Residual Solvent (**$m_{solv,res} > 0.20$ g **via Eq. 20):** Indicates incomplete carboxylic–oxirane esterification or residual n-propyl acetate plasticization. *Corrective Branch:* Extend Stage 2 post-cure hold at 150 °C from 3.5 h to 5.0 h under N_2 (never exceeding 155 °C) and verify $m_{solv,res} \leq 0.20$ g and $\Delta m_{5min} < 0.03$ g prior to casting.
3. **Parasitic Vinylidene Crosslinking (ATR-FTIR** 1635/815 cm^{-1} **Band Loss** $> 20\%$**):** Indicates thermal radical homopolymerization of exocyclic itaconate $C=CH_2$ bonds during curing or reprocessing. *Corrective Branch:* Increase L-ascorbyl palmitate to 0.15 wt%, raise biochar-compensated α-tocopherol slope from +0.04 wt% to +0.07 wt% per 1.0 wt% biochar, and verify oxygen-free N_2 purge (< 50 ppm O_2).
4. **Incomplete Stress Relaxation (**$G(3\tau^*)/G_0 > 0.08$ **via Eq. 17) or Non-Arrhenius Decay (**$\tau^*_{165\,°C} \geq \tau^*_{155\,°C}$ **via Eq. 16):** Indicates permanent radical C = C crosslinking or biochar micropore adsorption of lipophilic stabilizers outpacing associative transesterification. *Corrective Branch:* Pre-saturate biochar with 100% of the EHSO-phase α-tocopherol before pre-hardener addition and verify residual ash < 0.5 wt%.
5. **Reprocessing Failure (**$\text{Retention}_{C2} < 85.0\%$ **via Eq. 18 or Porous Consolidation):** Indicates insufficient transesterification dwell time at 155 °C or moisture-induced steam voids. *Corrective Branch:* Extend hot-press dwell at 155 °C from 60 min to 90–120 min (matching Li, Ju & Zhang, 2024) [2] and re-verify biochar vacuum drying ($H_2O < 0.5$ wt%).
6. **Filler Agglomeration Drop (**$\sigma_{max,RUN\text{-}n} < \sigma_{max,RUN\text{-}0}$**):** Identifies the exact rheological percolation ceiling for 500-mesh hemp biochar; lock optimal composite loading at the highest concentration preceding tensile 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 [1]. Polyphenolic tannic acid networks combined with epoxidized vegetable oils and cyclic anhydrides via aprotic pre-esterification (n-propyl acetate) form catalyst-free vitrimers via abundant −OH self-catalysis [2, 3]. Itaconic anhydride undergoes thermal isomerization to citraconic anhydride above ≈ 168 °C [11], establishing 150–155 °C as the optimal low-isomerization cure and reprocessing window. Under Flory–Stockmayer network theory [16], matching $Eq_{COOH} = 1.0000\,Eq_{epoxy}$ via $R_{protic} = 1.25$, $f_{graft} = 0.80$ eliminates static aryl-ether percolation ($0.00 \times p_{c,static}$) while preserving $0.2500\,Eq_{epoxy}$ free catalytic phenolic −OH. Equations (1)–(21) are exact algebraic identities.

Pre-Experimental Status [THEORETICAL]: The specific multi-component integration of EHSO, full-ester galloyl-itaconate pre-hardener ($R_{protic} = 1.25$, $f_{graft} = 0.80$), split-phase α-tocopherol + L-ascorbyl palmitate stabilization, three-stage devolatilization, stepped 105/125/150 °C curing, and citric-acid demineralized 500-mesh hemp biochar (CF-TAIA-125G80, v.28-CF-Rev4.2-Opt) has not yet been physically synthesized or tested. No mechanical strength, T_g, or activation energy values are claimed prior to execution of Tables 5–8.

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 and ascorbic acids, and hemp hurd biochar) can be sourced as certified organic inputs processed via enzymatic lipase catalysis [7] and physical steam activation.

9. Replication & Multi-Lot Calibration Standards

1. Always report measured lot values ($\mathrm{EEW}_{\mathrm{meas}}$, $\mathrm{HV}_{\mathrm{meas}}$, P_{IA}, *n*-propyl acetate moisture, Stage A/B/95 °C strip mass balance via Eqs. 19–21, biochar BET surface area S_{BET}, residual ash wt%, D_{90} particle size, and post-drying moisture content) alongside mechanical and DSC OIT results.
2. Never introduce multi-scale carbon hybrids (e.g., carbon nanosheets + short carbon 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 [22].
3. Deposit raw FTIR spectra (`.csv`/`.jdx`), DSC OIT traces, DMA curves, and stress-relaxation datasets under open CC BY 4.0 repositories (Zenodo / GitHub) referencing Hempoxies CF-TAIA-125G80 (v.28-CF-Rev4.2-Opt).

10. Open-Source Reporting, Versioning & Verified References

10.1 Document Version History

- **v.25-CF-Rev1.0 (November 24, 2026):** Initial open-source specification establishing Variant CF-TAIA-95 ($R = 0.95$, 60 : 40 TA/IA, 0 wt% FGE, 0 wt% metal catalyst, 0.20 wt% α-tocopherol, 0.0–3.0 wt% hemp biochar).
- **v.25-CF-Rev1.1 (November 24, 2026):** Literature-optimized revision introducing aprotic *n*-propyl acetate pre-esterification (90 °C, 60 min), moderate vacuum stripping (60–65 °C, 50–100 mbar), extended cure (120 °C/4 h + 160 °C/4 h), biochar-compensated α-tocopherol dosing, and Reference [2] bibliographic correction.
- **v.26-CF-Rev2.0 (November 24, 2026):** Variant CF-TAIA-105G. Resolved pre-esterification protic conservation ($R_{\mathrm{protic}} = 1.05$, $f_{\mathrm{graft}} = 0.35$), introduced Stage A solvent pre-strip, added 0.10 wt% L-ascorbyl palmitate, and mandated citric-acid demineralized 500-mesh hemp biochar (ash < 0.5 wt%).
- **v.27-CF-Rev3.0 (November 24, 2026):** Variant CF-TAIA-105G55. Raised f_{graft} from 0.35 to 0.55, moderated Stage A pre-strip to 65–70%, lowered post-cure to 150 °C and reprocessing to 155 °C, and added an ATR-FTIR $\mathrm{C{=}CH_2}$ gate.
- **v.28-CF-Rev4.0 / Rev4.1-Opt (November 24, 2026):** Variant CF-TAIA-125G80. Eliminated static aryl-ether percolation via $R_{\mathrm{protic}} = 1.25$, $f_{\mathrm{graft}} = 0.80$ ($\phi_{\mathrm{ester}} = 100.0\%$, $0.00 \times p_{\mathrm{c,static}}$), implemented split-phase antioxidant dosing, added a 95 °C $\mathrm{N_2}$-sweep pre-gelation devolatilization dwell with dual gravimetric criteria, enforced ≤ 3.0 mm plaque thickness with stepped 105/125/150 °C curing, and added Step 2 FTIR anhydride and Table 7 $G(3\tau^*)/G_0 \leq 0.08$ gates.
- **v.28-CF-Rev4.2-Opt (November 24, 2026 — Current Optimized Specification):** Adds explicit gravimetric devolatilization mass-balance equations (Eqs. 19–21), reconciles IA purity-dependent mass rounding (53.38 g at $P_{\mathrm{IA}} = 100.0\%$ vs. 53.92 g at $P_{\mathrm{IA}} = 99.0\%$), and links rejection gates directly to numbered equations.

10.2 Verified References (Protocol Standard: 26 Live Sources)

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