LANDRY ORGANIC LABS

HEMPOXIES RESEARCH PROGRAM

Hempoxies

CF-TAIA-125G80

Epoxidized Hemp Seed Oil Vitrimer Composite

Proposed full-ester, zero-permanent-percolation architecture without added metal catalyst

Technical specification and experimental validation protocol

$R_{\text{protic}} = 1.25 \quad f_{\text{graft}} = 0.80$

THEORETICAL / PRE-EXPERIMENTAL
TRL 1–2 · Not experimentally validated · Not a production SOP

Marie-Soleil Seshat Landry

Founder & CEO · Landry Industries / Landry Organic Labs / Marie Landry's Spy Shop
Moncton, New Brunswick, Canada
ORCID: 0009-0008-5027-3337

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https://landryindustries.ca · https://marielandryspyshop.com
https://searchfororganics.com
marielandryceo@gmail.com · corrections@marielandryspyshop.com

Research status and disclosure

AI-assisted technical specification; originally prepared with Google Gemini and subsequently edited and typeset with ChatGPT. All test results below are blank. Exact charge arithmetic does not demonstrate quantitative grafting, exclusive reaction pathways, zero permanent network formation, vitrimer behavior, complete solvent removal or mechanical performance. Practical execution requires appropriately equipped laboratory personnel and a process-safety assessment. CC BY 4.0; reuse subject to attribution. Independent patent/prior-art legal effect not established.

Executive technical abstract

This open-science specification proposes a tannic-acid/itaconic-anhydride pre-hardener for epoxidized hemp seed oil (EHSO), with no added metal catalyst or FGE reactive diluent. Four hemp-biochar loadings (0, 0.5, 1.5 and 3.0 wt% relative to a nominal matrix charge) are compared. The intended design ratio is R(protic)=1.25, graft fraction f(graft)=0.80 and nominal acid-to-epoxy charge ratio 1.00. It prescribes in-process FTIR, mass-balance, gel fraction, DMA stress relaxation and reprocessing falsification gates. Chemical selectivity and performance must be empirically confirmed.

1. Audit and underlying design hypothesis

Prior architectures considered EHSO/citric acid/metal catalysts/FGE, sebacic-acid alternatives and mixed carbon fillers. Proposed improvements eliminate FGE and added metal salts, prefunctionalize tannic acid with itaconic anhydride, employ split-phase tocopherol/ascorbyl-palmitate dosing, implement staged solvent removal and restrict the initial filler study to steam-activated demineralized hemp biochar.

1.1 Claimed design changes and verification status

• R(protic) = 1.25 and target f(graft) = 0.80 supply a nominal 1.00 equivalent of pendant COOH per EHSO epoxy equivalent. This is a charge balance, not proof of complete ester bond formation or absence of aryl-ether branches.

• A nominal five residual phenolic hydroxyl groups per idealized tannic-acid core may assist exchange, but their accessibility, kinetics and chemistry remain unknown.

• Split alpha-tocopherol (50:50) and all L-ascorbyl palmitate into pre-hardener and EHSO phases; effectiveness and possible interference require confirmation.

• Stage A/Stage B/95 °C devolatilization is planned; two gravimetric acceptance gates are proposed. Thermal and vacuum safety must be independently assessed.

• Thin plaques ($\leq$3.0 mm) and a 105/125/150 °C ramp are specified as candidate cure settings, not demonstrated exotherm controls.

• Anhydride IR-band attenuation and vinylidene-band retention are screening proxies, not unique molecular-structure assays.

• Incomplete high-temperature stress relaxation may indicate permanent sub-networks, physical constraints or instrumental artifacts; it is not a mechanism-specific diagnosis.

• Theoretical Flory–Stockmayer-style percolation comparison cannot substantiate ”zero percolation” when reaction probabilities, functional-group distributions and side reactions are unknown.

1.2 Comparison of design revisions

Variant	Nominal charge/graft	Solvent and cure strategy
CF-TAIA-95, v25	R 0.95 nominal	Single strip; 120/160 °C cure; tocopherol only
CF-TAIA-105G, v26	R 1.05; f 0.35	Stage A pre-strip; AP 0.10%; 120/160 °C cure
CF-TAIA-105G55, v27	R 1.05; f 0.55	65–70% Stage A strip; cure ≤150 °C
CF-TAIA-125G80, v28	R 1.25; target f 0.80	Staged pre-strip/degas; 95 °C dwell; 105/125/150 °C cure

2. Performance requirements — acceptance targets, not results

• Bio-renewable feedstock targets, no added FGE or metal salt catalyst; verify feedstock origins, upstream reaction inputs and biobased-carbon content.

• Use processed hemp biochar only; 500 mesh and D90 ≤25 µm require measured particle-size distribution; ash and moisture individually <0.5 wt%.

• Target acid/epoxy equivalence 1.0000 at the charge-sheet level; directly determine actual pre-hardener graft conversion and epoxy conversion.

• Measure lot-specific EHSO EEW, tannic-acid hydroxyl value and IA purity before every batch.

• Monitor vinylidene signals and antioxidant effects; attribution to radical processes needs orthogonal evidence.

• Use ≤3 mm test plaques; instrument temperature gradients and actual peak exotherms.

• Require corrected gel fraction ≥92%, stress-relaxation fraction at $3\tau^*$ ≤0.08 and cycle-2 tensile retention ≥85%, each with replicated uncertainty.

• Do not describe the material as an experimentally confirmed vitrimer before topology-exchange, creep/relaxation and reprocessing tests are passed.

3. Central falsifiable formulation hypothesis

Anhydrous tannic-acid/itaconic-anhydride pre-esterification under nitrogen at 90 °C is hypothesized to furnish a multifunctional carboxyl-bearing pre-hardener. After combining this with EHSO and 0–3.0 wt% demineralized hemp biochar, removing processing solvent, and carrying out a stepped 105/125/150 °C cure, the proposed network may exhibit high gel content, thermally activated stress relaxation and reprocessable tensile properties. All these outcomes are hypotheses until measured. The intended 80% graft conversion and 100% dynamic ester junction fraction are not established by reagent stoichiometry.

Variant nomenclature

CF = no intentionally added external metal catalyst; TAIA = tannic acid + itaconic anhydride; 125G80 = target protic ratio 1.25 and graft fraction 0.80; v28-CF-Rev4.3-Opt = project revision identifier.

4. Experimental design and algebra

4.1 Reference lot benchmarks

Input	Reference value	Required measured value
EHSO	Oxirane oxygen 7.62 wt%; EEW 209.97 g/eq	Lot, EEW; test method applicability
Tannic acid	HV 824.51 mg KOH/g; HEW 68.05 g/eq	Lot; measured HV; verify interference
Itaconic anhydride	112.08 g/mol; 99–100% purity	Lot; purity, moisture
Hemp biochar	D90 $\leq$25 µm; ash <0.5%; moisture <0.5%	BET area, ash, D90, Karl Fischer moisture
n-Propyl acetate	Initial 55–60 mL; anhydrous solvent	Initial solvent mass, moisture, recovery
Tocopherol / ascorbyl palmitate	$\geq$96% / $\geq$98% purity targets	Certificate of analysis and actual charge

4.2 Charge-sheet equations (1–11)

$$\mathrm{EEW}_{\mathrm{meas}} = 1600/O_{\mathrm{oxirane}} \tag{1}$$

$$\mathrm{HEW}_{\mathrm{TA}} = 56106/\mathrm{HV}_{\mathrm{meas}} \tag{2}$$

$$\mathrm{Eq}_{\mathrm{epoxy}} = m_{\mathrm{EHSO}}/\mathrm{EEW}_{\mathrm{meas}} \tag{3}$$

$$\mathrm{Eq}_{\mathrm{TA},0} = 1.25\,\mathrm{Eq}_{\mathrm{epoxy}} \tag{4}$$

$$m_{\mathrm{TA}} = \mathrm{Eq}_{\mathrm{TA},0}\,\mathrm{HEW}_{\mathrm{TA}} \tag{5}$$

$$\mathrm{Eq}_{\mathrm{IA,target}} = 0.8\,\mathrm{Eq}_{\mathrm{TA},0} = \mathrm{Eq}_{\mathrm{epoxy}} \tag{6}$$

$$m_{\mathrm{IA}} = \mathrm{Eq}_{\mathrm{IA,target}}\,112.08\,(100/P_{\mathrm{IA}}) \tag{7}$$

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

$$m_{\mathrm{tocopherol}} = \frac{0.20 + 0.04 w_{\mathrm{filler}}}{100} M_{\mathrm{matrix}} \tag{9}$$

$$m_{\mathrm{AP}} = \frac{0.10}{100} M_{\mathrm{matrix}} \tag{10}$$

$$m_{\mathrm{biochar}} = \frac{w_{\mathrm{filler}}}{100} M_{\mathrm{matrix}} \tag{11}$$

Dimensional caution: Eq. (1) uses 16 g/mol for oxirane oxygen and percent input. Eq. (7) assumes one mole IA per targeted esterification and purity P expressed in percent. These are calculated charges; actual conversion must be assayed.

4.3 Worked 100 g EHSO lot and 1.5% filler example

For oxirane O = 7.44 wt%, HV = 750 mg KOH/g and IA assay purity 99.5%: EEW = 215.05 g/eq, epoxy = 0.4650 eq, nominal TA-OH = 0.58125 eq, TA 43.48 g, IA 52.38 g, nominal matrix 195.86 g. Tocopherol 0.509 g, AP 0.196 g and biochar 2.938 g. Rounded values are illustrative. Actual weighed masses must be calculated from full-precision lot data.

4.4 Benchmark 100 g batches (100% IA purity)

Run	Biochar wt%	EHSO g	TA g	IA g	Tocopherol g	AP g	Biochar g
RUN-0	0.0	100.00	40.51	53.38	0.388	0.194	0.000
RUN-1	0.5	100.00	40.51	53.38	0.427	0.194	0.969
RUN-2	1.5	100.00	40.51	53.38	0.504	0.194	2.908
RUN-3	3.0	100.00	40.51	53.38	0.620	0.194	5.817

At 99% IA purity the nominal IA charge is 53.92 g and nominal matrix charge 194.43 g. A quarter-scale experiment uses one quarter of each charge. Tocopherol is split equally; AP is fully in the pre-hardener phase.

5. Candidate process and measurement logs

5.1 Six-stage proposed process (laboratory validation required)

1. Biochar treatment and drying. Treat 500-mesh steam-activated hemp biochar in 2 wt% citric acid at 60 °C for 30 min, rinse toward neutral filtrate and vacuum-dry separately from tannic acid at 105–110 °C for 4 h. Verify that the treatment does not undermine biochar properties or safety.

2. Pre-hardener. Charge dry tannic acid, IA, half of tocopherol and all AP into anhydrous n-propyl acetate (55–60 mL per 100 g EHSO), under dry nitrogen at 90 °C for a nominal 60 min. Assay anhydride 1780/1855 cm^{-1} bands and competing IA hydrolysis with a validated quantitative method.

3. Stage A strip. Proposed 60 °C, 60–80 mbar, 12 min, targeting 65–70% solvent recovery by mass (not assumed by duration).

4. EHSO blend. Pre-wet 0–3 wt% biochar in EHSO (70 °C), then add remaining tocopherol. Add warm pre-hardener around 65 °C with controlled stirring.

5. Stage B and final strip. Proposed 60 °C, 40–50 mbar, 15 min, then 95 °C under dry nitrogen 30 min. Check complete mass ledger and residual solvent by an independent analytical method before casting.

6. Cure / reprocess. Cast $\leq$3 mm and test a candidate 105 °C $\times$2 h, 125 °C $\times$2.5 h, 150 °C $\times$3.5 h nitrogen-blanketed cure. Assess a proposed 155 °C, 15 MPa, 60 min reprocess cycle. Escalation requires measured temperature and pressure controls.

Safety: n-propyl acetate is flammable (flash point below ambient); heating under vacuum/nitrogen requires explosion-rated equipment, a suitable condenser, ignition-source controls and engineered exhaust. Do not use this research draft as an operating authorization.

5.2 Thermal and mixing checkpoint log (blank template)

Stage	Target conditions	Actual / QC / pass-fail
Dry	105–110 °C, 4 h	_____
Pre-esterify	90 °C, 60 min; FTIR gate	_____
Stage A	60 °C, 60–80 mbar	_____
EHSO blend	70 $\rightarrow$ 65 °C	_____
Stage B / sweep	60 °C vacuum + 95 °C N_2	_____
Cure	105/125/150 °C steps	_____

5.3 ATR-FTIR log (blank data)

Sample	IA 1780/1855	Epoxy 825–915	Ester 1735	C=CH_2 1635/815
Pre-hardener	_____	_____	_____	_____
RUN-0	_____	_____	_____	_____
RUN-1	_____	_____	_____	_____
RUN-2	_____	_____	_____	_____
RUN-3	_____	_____	_____	_____

5.4 Swelling, gel content and OIT log (blank data)

Sample	W_0 dry g	W_s swollen g	W^d final g	G %	Q	OIT min
RUN-0	_____	_____	_____	_____	_____	_____
RUN-1	_____	_____	_____	_____	_____	_____

Sample	W_0 dry g	W_s swollen g	W^d final g	G %	Q	OIT min
RUN-2	_____	_____	_____	_____	_____	_____
RUN-3	_____	_____	_____	_____	_____	_____

6. Quantitative analysis, outcomes and governing relations

$$G(\%) = 100 W_d / W_0, \qquad Q = (W_s - W_d)/W_d \tag{12}$$

$$\phi_{\text{ester,nominal}} = \min(1, R_{\text{protic}} f_{\text{graft,target}}) = 1 \tag{13}$$

$$p_{c,\text{static,ideal}} = \frac{1}{(f_{\text{TA,residual}} - 1)(f_{\text{EHSO}} - 1)} \tag{14}$$

$$\nu_{e,\text{apparent}} = E'_{\text{rubbery}} / (3 R_{\text{gas}} T) \tag{15}$$

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

$$G(t)/G_0 = \exp(-t/\tau^*), \qquad G(3\tau^*)/G_0 \le 0.08 \tag{17}$$

$$\text{Retention}(\%) = 100 \sigma_{\text{cycle-n}} / \sigma_{\text{virgin}} \tag{18}$$

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

$$m_{\text{solv,res,apparent}} = m_{\text{pre-cure,measured}} - m_{\text{nonvol,nominal}} \tag{20}$$

$$\text{Removal}(\%) = 100(1 - m_{\text{solv,res,apparent}} / m_{\text{solv,0}}) \tag{21}$$

Interpretation restrictions: (13) is a nominal upper bound, not a measured ester-junction fraction; (14) is a simplified ideal-network threshold, not a proof of zero permanent crosslinks; (15) assumes rubber elasticity conditions; (17) is a single-exponential model and predicts $e^{-3} \approx 0.0498$ when fitted exactly; (19–21) are apparent mass balances and conflate solvent loss with other volatile losses, reaction uptake, retained impurities and sampling error. A residual-solvent measurement (e.g., validated headspace GC) is required.

6.1 DMA and stress-relaxation log

Run	E’25 MPa	Tg °C	τ* 145/155/165 °C s	Ea kJ/mol	G(3τ*)/G_0
RUN-0	_____	_____	_____	_____	_____
RUN-1	_____	_____	_____	_____	_____
RUN-2	_____	_____	_____	_____	_____
RUN-3	_____	_____	_____	_____	_____

6.2 ASTM D638 tensile reprocessing log

Run	Cycle C0/C1/C2	Tensile E MPa	σ max MPa	Retention %	Voids / status
RUN-0	_____	_____	_____	_____	_____
RUN-1	_____	_____	_____	_____	_____
RUN-2	_____	_____	_____	_____	_____
RUN-3	_____	_____	_____	_____	_____

7. Decision loop and falsification gates

Gate 1 — Pre-hardener. Anhydride 1780/1855 cm^{-1} attenuation <90%: stop. Repeat dry-reactant checks; extend 90 °C under nitrogen only if a validated conversion assay confirms benefit. FTIR band loss alone cannot distinguish grafting from hydrolysis.

Gate 2 — Gel and solvent. Gel fraction G <92% or apparent solvent residual >0.20 g: reject. Repeat actual solvent assay, stoichiometry and cure conversion; consider cure at 150 °C up to 5 h only after DSC/process safety validation. The 0.20 g criterion must be normalized to batch size.

Gate 3 — Vinylidene conservation. Normalized 1635/815 cm^{-1} band attenuation >20%: reject pending mechanism check. Proposed branch: AP 0.15 wt%, tocopherol slope +0.07 wt% per wt% biochar, oxygen <50 ppm. These are unvalidated formulation changes, not guaranteed fixes.

Gate 4 — Exchange / relaxation. $G(3\tau^*)/G_0$ >0.08 or $\tau^*165 \geq \tau^*155$: stop and test crosslinking, adsorbed stabilizer, oxidative aging and instrument artifacts. Evaluate biochar pre-wetting with tocopherol and ash <0.5%, then rerun with replicates.

Gate 5 — Reprocessability. Cycle-2 tensile retention <85% or obvious voiding: reject. Explore 155 °C press dwell of 90–120 min after confirming no decomposition and adequate drying; do not state literature equivalence without matching formulation and test conditions.

Gate 6 — Loading optimization. Composite σmax below RUN-0: reject that loading for tensile improvement. This does not define an exact rheological percolation ceiling; use dispersion microscopy, rheometry and replicates to distinguish failure modes.

8. Evidence boundary and corrections to overclaims

Literature-supported context: Associative transesterification and catalyst-free tannic-acid vitrimer systems have published precedent [1–3]. Specific reagent chemistry and thermal conditions are system dependent [2].

Not experimentally verified: This CF-TAIA-125G80 formulation, quantitative IA graft selectivity, polymer topology, carbon content, stress-relaxation activation energy, tensile modulus, Tg and fatigue behavior have not been measured.

Correction — "zero-percolation": A nominal balance of COOH and epoxide does not eliminate phenol–epoxy etherification, epoxy homopolymerization or other irreversible crosslinks. Do not represent the idealized calculation as demonstrated zero static network.

Correction — IA thermal threshold: A universal isomerization onset at approximately 168 °C is not established. Literature indicates impurity-dependent rearrangement can occur at lower temperatures; test the actual IA and pre-hardener system.

Correction — "100% bio-based": A bio-derived feedstock list does not establish 100% biobased carbon or a certified organic polymer. Test ASTM D6866 or applicable carbon accounting and verify each upstream supply chain.

Correction — "exact equations": Equations (1–11) are target-charge identities under specified assay assumptions; (12–18) are model/test definitions with assumptions; (19–21) are idealized gravimetric mass accounts, not direct chemical assays.

Standard applicability: ASTM D2765 is expressly scoped to crosslinked ethylene plastics; applying its solvent extraction procedure to these epoxy networks requires a validated adaptation and suitable reporting. ASTM E1858 is for hydrocarbons, so OIT on this matrix must likewise justify adaptation.

9. Replication and multi-lot calibration

• Report lot ID, CoA, measured EEW, HV, IA purity and moisture, n-propyl acetate moisture and initial/recovered solvent weights; separately assay residual solvent.

• Report biochar BET, ash, D90, moisture and micrographs. Document dispersibility and mass loss in drying or citric-acid wash.

• Do not combine nanosheets, short carbon fiber and biochar until RUN-0 to RUN-3 single-filler screening is reproduced with n≥5 independently prepared ASTM D638 tensile specimens per condition and independent production lots.

• Deposit raw spectra (.csv/.jdx), DSC traces, DMA modulus/relaxation data, tensile curves, processing temperatures and unmodified instrument exports in Zenodo/GitHub under CC BY 4.0.

• Prespecify uncertainty intervals and decision rules; differences smaller than measurement variability do not establish an optimum loading.

10. Open-science versioning and bibliography

10.1 Version history — supplied project record

• v25 Rev1.0: CF-TAIA-95 benchmark (November 24, 2026).

• v25 Rev1.1: aprotic pre-esterification, moderate vacuum and extended cure.

• v26 Rev2.0: R 1.05, target grafting 0.35 and two-stage solvent removal.

• v27 Rev3.0: target grafting 0.55, revised cure temperatures and IR monitoring.

• v28 Rev4.0–4.1: R 1.25, target grafting 0.80, split-phase antioxidants.

• v28 Rev4.2: gravimetric mass-balance equations and purity rounding.

• v28 Rev4.3-Opt (November 24, 2026): typographic integration and revised protocol.

10.2 Bibliography: 26 references reproduced from supplied manuscript

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10.3 Supplemental verification and safety sources

• Royal Society of Chemistry publisher record, Li et al. (2024): https://pubs.rsc.org/en/content/articlelanding/2024/gc/d4gc00259h

• ILO/WHO ICSC 0940, n-propyl acetate (boiling point 101.6 °C; flammability hazards): https://chemicalsafety.ilo.org/dyn/icsc/showcard.display?p_card_id=0940

• ASTM product scope for D2765, crosslinked ethylene plastics: https://store.astm.org/d2765-16r24.html

• Thermal polymerization/isomerization investigation (1967): https://www.jstage.jst.go.jp/article/koron1944/24/272/24_272_790/_article

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