OPEN-SOURCE PRIOR-ART SPECIFICATION | CREATIVE COMMONS CC BY 4.0 | TRL 1–2 PRE-EXPERIMENTAL

Hempoxies Variant CF-TAIA-125G80 (v.28-CF-Rev4.0)

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)

Author: Marie-Soleil Seshat Landry, Founder & CEO, Landry Industries / Landry Organic Labs / Marie Landry's Spy Shop
Location & Registry: Moncton, New Brunswick, Canada | ORCID: 0009-0008-5027-3337
Official Repositories: landryindustries.ca | marielandryspyshop.com | searchfororganics.com
Correspondence: marielandryceo@gmail.com | Public Corrections: corrections@marielandryspyshop.com
Date & Version: November 24, 2026 | Version: v.28-CF-Rev4.0 (Supersedes CF-TAIA-105G55 v.27-CF-Rev3.0)
License: Creative Commons Attribution 4.0 International (CC BY 4.0) — https://creativecommons.org/licenses/by/4.0/

Open-Source Prior-Art Notice (CC BY 4.0): Released openly as public prior art and an open-science benchmark. Anyone may replicate, test, modify, and redistribute this specification provided attribution is credited to Marie-Soleil Seshat Landry (ORCID: 0009-0008-5027-3337) and Landry Organic Labs.

Keywords: Hempoxies CF-TAIA-125G80, Hempoxies v.28-CF-Rev4.0, Flory–Stockmayer percolation-free vitrimer, 100% β-hydroxy ester bio-epoxy, catalyst-free transesterification, epoxidized hemp seed oil (EHSO), tannic acid galloyl-itaconate pre-hardener, split-phase lipophilic radical stabilization, demineralized hemp biochar, covalent adaptable networks (CANs), L-ascorbyl palmitate.

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, 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 & Rev3.0 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 in Rev3.0 (CF-TAIA-105G55) Resolved in Rev4.0 (CF-TAIA-125G80) [× 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 resolved in v.28-CF-Rev4.0 (CF-TAIA-125G80):

1. **True Elimination of Static Aryl-Ether Percolation ($R_{\text{protic}} = 1.05, f_{\text{graft}} = 0.55 \rightarrow R_{\text{protic}} = 1.25, f_{\text{graft}} = 0.80$):** Under Flory–Stockmayer gelation theory ($p_A p_B = [(f_A - 1)(f_B - 1)]^{-1}$) [16], leaving 45% of tannic acid's $\overline{f}_{\text{TA}} \approx 25.0$ phenolic −OH groups ungrafted leaves $\overline{f}_{\text{ArOH,free}} = 11.25$ permanent ether-forming sites per tannic acid core. Paired with EHSO ($\overline{f}_{\text{EHSO}} \approx 4.60$), the critical gelation product for the static aryl-ether sub-network alone is $p_{c,\text{static}} = 0.0271$. In Rev3.0 ($R_{\text{protic}} = 1.05, f_{\text{graft}} = 0.55$), 42.25% permanent aryl-ether junctions yielded $p_A p_B = 0.3778$ (13.94× above $p_{c,\text{static}}$), locking an unrelaxable static ether skeleton while requiring sluggish uncatalyzed phenolic etherification at 150°C. Shifting to $R_{\text{protic}} = 1.25, f_{\text{graft}} = 0.80$ yields $Eq_{\text{COOH}} = 1.25 \times 0.80 = 1.0000$ eq fast-reacting carboxylic acid per 1.0000 eq epoxide ($\phi_{\text{ester}} = 100.0\%$ exchangeable β-hydroxy ester linkages, $0.00 \times p_{c,\text{static}}$), while preserving 0.2500 eq/eq epoxide free catalytic phenolic −OH (5.0 per TA molecule) plus 1.0000 eq secondary aliphatic β-OH (1.2500 eq total catalytic −OH). Even if steric crowding caps grafting at 90% of target ($f_{\text{graft,eff}} = 0.72$), 90.0% of epoxides form dynamic esters and the remaining 10.0% static ethers yield

$p_A p_B = 0.0286 < p_{c,\text{static}} = 0.0463$ ($0.62 \times p_c$ — strictly non-percolating).

2. **Split-Phase Antioxidant Delivery for Pre-Hardener & Triglyceride Protection:** Dosing 50% of the α-tocopherol and 100% of the L-ascorbyl palmitate ($T_m \approx 115$–118°C) directly into Step 2's polar aprotic n-propyl acetate medium protects exocyclic vinylidene ($C=CH_2$) bonds during the 90°C pre-esterification and 60°C Stage A strip while eliminating L-ascorbyl palmitate micro-crystallinity in 70°C EHSO. The remaining 50% α-tocopherol pre-saturates biochar micropores in EHSO.
3. **Three-Stage Devolatilization with 95°C Pre-Gelation Dwell & Exact Gravimetric Endpoint:** Replaces Rev3.0's ambiguous $\Delta m < 0.05$ g criterion with explicit dual mass-balance controls ($m_{\text{solv,res}} \leq 0.20$ g, i.e., $\geq 99.5\%$ removal of initial solvent, 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 just below n-propyl acetate's normal boiling point (101.6°C) before ramping to gelation.
4. **Exotherm-Safe Casting Thickness (≤ 3.0 mm) & Stepped Gelation Ramp (105°C/2.0 h + 125°C/2.5 h + 150°C/3.5 h):** Dissipates the carboxylic–oxirane ring-opening enthalpy ($\Delta H \approx -80$ to -95 kJ/mol epoxide) in thin plaques so internal resin temperature never overshoots 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 ($G(3\tau^*)/G_0 \leq 0.08$):** Directly tests absence of a percolated permanent ether sub-network at $t = 3\tau^*$.
7. **Complete Rounding, Glyph, Header, and Table Structural Integrity ($1 = 1 = 1$):** Reconciles exact rounded and unrounded matrix sums ($M_{\text{matrix}} = 193.89$ g benchmark; 195.86 g sanity check), restores all Greek/math glyphs (β, τ^*, ν_e, $\sigma_{\max}$, ε_b, $\leq$, $\geq$, [× THEORETICAL]), fixes running headers, and places all captions cleanly above Tables 1–8.

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

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-Rev4.0)
Protic Stoichiometry & Graft Fraction	Nominal $R = 0.95$; post-graft $R_{\text{eff}} = 0.570$ (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; $13.9 \times p_{c,\text{static}}$)	$R_{\text{protic}} = 1.25$, $f_{\text{graft}} = 0.80$ (100.0% **dynamic β-hydroxy ester;** 0.0% **static ether;** $0.00 \times p_{c,\text{static}}$)
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 exact**)
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 **pre-gelation dwell**
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-Rev4.0`) 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 fully 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 ($D_{90} \leq 25\,\mu$m) 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 ≥ 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-Rev4.0)

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 ≥ 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-Rev4.0` = Sequential Hempoxies specification version 28, fourth catalyst-free optimization 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 ($HEW \approx 68$–85 g/eq) and EHSO oxirane conversion varies by batch ($EEW \approx 193$–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-Rev4.0)

Precursor	Lot / Batch ID	Reference Benchmark	Measured Lot Value	Units
EHSO (ASTM D1652) [20]		$O_o = 7.62$ wt% ($EEW = 209.97$)	$O_o =$ _____ ; $EEW_{\text{meas}} =$ _____	wt% | g/eq
Tannic Acid (ASTM E222) [21]		$HV = 824.51$ ($HEW = 68.05$)	$HV_{\text{meas}} =$ _____ ; $HEW_{\text{TA,meas}} =$ _____	mg KOH/g | g/eq
Itaconic Anhydride		$AEW_{\text{IA}} = 112.08$ (≥ 99.0% pure)	Purity $P_{\text{IA}} =$ _____	g/eq | wt%
Hemp Biochar (Demin.)		$D_{90} \leq 25\,\mu$m; Ash < 0.5%; H_2O < 0.5%	Ash = ___ ; H_2O_{dry} = ___ ; 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 Equations (for Chosen Base Mass m_{EHSO})

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 using the protic-conserved equations below:

$$EEW_{\mathrm{meas}} = \frac{16.00 \times 100}{O_o(\%)} = \frac{1600}{O_o(\%)} \tag{1}$$

$$HEW_{\mathrm{TA,meas}} = \frac{56.106 \times 1000}{HV_{\mathrm{meas}}} = \frac{56106}{HV_{\mathrm{meas}}} \tag{2}$$

$$Eq_{\mathrm{epoxy}} = \frac{m_{\mathrm{EHSO}}}{EEW_{\mathrm{meas}}} \tag{3}$$

$$Eq_{\mathrm{TA},0} = R_{\mathrm{protic}} \times Eq_{\mathrm{epoxy}} = 1.2500 \times \left(\frac{m_{\mathrm{EHSO}}}{EEW_{\mathrm{meas}}}\right) \tag{4}$$

$$m_{\mathrm{TA}} = Eq_{\mathrm{TA},0} \times HEW_{\mathrm{TA,meas}} = 1.2500 \times \left(\frac{m_{\mathrm{EHSO}}}{EEW_{\mathrm{meas}}}\right) \times HEW_{\mathrm{TA,meas}} \tag{5}$$

$$Eq_{\mathrm{IA}} = f_{\mathrm{graft}} \times Eq_{\mathrm{TA},0} = 0.8000 \times 1.2500 \times Eq_{\mathrm{epoxy}} = 1.0000 \times \left(\frac{m_{\mathrm{EHSO}}}{EEW_{\mathrm{meas}}}\right) \tag{6}$$

$$m_{\mathrm{IA}} = Eq_{\mathrm{IA}} \times \frac{112.08}{P_{\mathrm{IA}}/100} = 112.0800 \times \left(\frac{m_{\mathrm{EHSO}}}{EEW_{\mathrm{meas}}}\right) \times \left(\frac{100}{P_{\mathrm{IA}}}\right) \tag{7}$$

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

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

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

- **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):

$$\mathrm{Ar-OH} + \mathrm{C_5H_4O_3\ (IA)} \longrightarrow \mathrm{Ar-O-C(=O)-C(=CH_2)\text{-}CH_2\text{-}COOH}$$

  After reacting Eq_{IA} moles of IA with $Eq_{\mathrm{TA},0}$ equivalents of TA phenolic −OH, the pre-hardener contains $Eq_{\mathrm{COOH}} = 1.0000\, Eq_{\mathrm{epoxy}}$ fast-reacting carboxylic acid equivalents (yielding 100.0% dynamic β-hydroxy ester junctions) and $Eq_{\mathrm{ArOH,free}} = (1.2500 - 1.0000)\, Eq_{\mathrm{epoxy}} = 0.2500\, Eq_{\mathrm{epoxy}}$ free catalytic phenolic −OH equivalents, summing to $1.0000 + 0.2500 = 1.2500\, Eq_{\mathrm{epoxy}}$ total protic equivalents ($R_{\mathrm{protic}} = 1.25$).
- **Worked Lot-Recalculation Sanity Check ($m_{\mathrm{EHSO}} = 100.00$ g; $w_{\mathrm{filler}} = 1.5$ wt%):** If a supplier CoA reports $O_o = 7.44$ wt% ($EEW_{\mathrm{meas}} = 215.05$ g/eq), $HV_{\mathrm{meas}} = 750.0$ mg KOH/g ($HEW_{\mathrm{TA,meas}} = 74.81$ g/eq), and $P_{\mathrm{IA}} = 99.5\%$, then $Eq_{\mathrm{epoxy}} = 0.4650$ eq, $Eq_{\mathrm{TA},0} = 0.5813$ eq, $m_{\mathrm{TA}} = 43.48$ g, $Eq_{\mathrm{IA}} = 0.4650$ eq, $m_{\mathrm{IA}} = 52.38$ g, $M_{\mathrm{matrix}} = 100.00 + 43.48 + 52.38 = 195.86$ g (exact unrounded sum = 195.86 g), $w_{\mathrm{tocopherol}} = 0.20 + 0.04(1.5) = 0.26$ wt%, $m_{\mathrm{tocopherol}} = 0.51$ g (0.255 g Step 2 + 0.255 g Step 4), $m_{\mathrm{AP}} = 0.20$ g, and $m_{\mathrm{biochar}} = 2.94$ g.

4.3 Master Batch Weighing Table ($R_{\mathrm{protic}} = 1.25$, $f_{\mathrm{graft}} = 0.80$; 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 C = CH_2 retention, and gelation before casting full 100.00 g EHSO plaques.

Table 3: Master Stoichiometric Batch Sheet for Hempoxies Variant CF-TAIA-125G80, v.28-CF-Rev4.0 (EEW = 209.97 g/eq, HEW_{TA} = 68.05 g/eq, AEW_{IA} = 112.08 g/eq, M_{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)	Actual Log (g)
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 ($D_{90} \leq 25\,\mu$m) 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; $\approx$ 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 $\geq$ 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 ($\sim$ 36–42 mL recovered), leaving a flowable syrup ($\sim$ 18–20 mL residual co-solvent, $\sim$ 82 wt% solids) that prevents glassy phase separation during EHSO blending.
4. **Pre-Wet Biochar in EHSO & Dissolve Remaining α-Tocopherol (70°C $\rightarrow$ 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 $\geq$ 99.5% total solvent removal ($m_{solv,res} \leq 0.20$ g above theoretical non-volatile mass $M_{matrix} + m_{tocopherol} + m_{AP} + m_{biochar}$, 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 $\leq$ 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 $\sim$ 168°C IA $\rightarrow$ 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-Rev4.0)

Process Stage	Setpoin
1. Demin. & Vac Dry	105–110
2. Aprotic Pre-Esterif.	90°C (N
3. Stage A Pre-Strip	60°C
4. Biochar / EHSO Mix	70°C →
5. Stage B + 95°C Dwell	60°C →
6. Stage 1A/1B Gelation	105°C -
7. Stage 2 Post-Cure	150°C (< 168°

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})	Broad
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: Solvent 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 seven standard polymer-physics, Flory–Stockmayer percolation, and dynamic-network relations:

$$G(\%) = \left(\frac{W_d}{W_0}\right) \times 100 \qquad \text{and} \qquad Q = \frac{W_s - W_d}{W_d} \tag{11}$$

$$\phi_{\text{ester}} = \min\left(1.0000,\ R_{\text{protic}} \times f_{\text{graft}}\right) = \min\left(1.0000,\ 1.25 \times 0.80\right) = 1.0000 \quad (100.0\%\ \text{dynamic } \beta\text{-hydroxy ester}) \tag{12}$$

$$p_{c,\text{static}} = \frac{1}{\left(\overline{f}_{\text{TA}}(1 - f_{\text{graft}}) - 1\right)\left(\overline{f}_{\text{EHSO}} - 1\right)} \qquad \text{(static aryl-ether percolation threshold)} \tag{13}$$

$$\nu_e = \frac{E'_r}{3R_{\text{gas}}T} \tag{14}$$

$$\frac{G(t)}{G_0} = \exp\left(-\frac{t}{\tau^*}\right) \qquad \text{with completeness gate } \frac{G(3\tau^*)}{G_0} \leq 0.08 \tag{15}$$

$$\ln(\tau^*) = \ln(\tau_0) + \frac{E_a}{R_{\text{gas}}T}, \qquad \text{Strength Retention } (\%) = \left(\frac{\sigma_{\text{Cycle}n}}{\sigma_{\text{Virgin}}}\right) \times 100 \tag{16}$$

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. (14), and τ^* in Eq. (15) 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].

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

Run ID	$E'_{25°C}$ (MPa)
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	$\sigma_{\max}$ (MPa)	E (MPa)	ε_b (%)	ΔT_g (°C)	Retention (%)	≥ 85% Gate
RUN-0 (0.0%)	Virgin (C0)				0.0 (Ref)	100.0%	Reference
	Cycle 1 (C1)						
	Cycle 2 (C2)						
RUN-1 (0.5%)	Virgin (C0)				0.0 (Ref)	100.0%	Reference
	Cycle 1 (C1)						
	Cycle 2 (C2)						
RUN-2 (1.5%)	Virgin (C0)				0.0 (Ref)	100.0%	Reference
	Cycle 1 (C1)						
	Cycle 2 (C2)						
RUN-3 (3.0%)	Virgin (C0)				0.0 (Ref)	100.0%	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-Rev4.0`) 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 $f_{\text{graft,eff}} \geq 0.72$. **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 (**$G < 92.0\%$**):** 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 $m_{\text{solv,res}} \leq 0.20$ 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 (**$G(3\tau^*)/G_0 > 0.08$**) or Non-Arrhenius Decay (**$\tau^*_{165^\circ\text{C}} \geq \tau^*_{155^\circ\text{C}}$**):** 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 (**$\text{Retention}_{C2} < 85.0\%$ **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 (**$\sigma_{\max,\text{RUN}-n} < \sigma_{\max,\text{RUN}-0}$**):** 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 $Eq_{\text{COOH}} = 1.0000\, Eq_{\text{epoxy}}$ via $R_{\text{protic}} = 1.25, f_{\text{graft}} = 0.80$ eliminates static aryl-ether percolation ($0.00 \times p_{c,\text{static}}$) while preserving $0.2500\, Eq_{\text{epoxy}}$ free catalytic phenolic −OH. Equations (1)–(16) are exact algebraic identities.
- **Pre-Experimental Status [× THEORETICAL]:** The specific multi-component integration of EHSO, full-ester galloyl-itaconate pre-hardener ($R_{\text{protic}} = 1.25, f_{\text{graft}} = 0.80$), 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-Rev4.0`) 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 (EEW_{meas}, HV_{meas}, P_{IA}, n-propyl acetate moisture, Stage A/B/95°C strip mass balance, 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-Rev4.0)`.

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 ($R_{\text{protic}} = 1.05, f_{\text{graft}} = 0.35$), 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 C = CH_2 gate.
- **`v.28-CF-Rev4.0` (November 24, 2026 — Current Specification):** Variant `CF-TAIA-125G80`. Eliminates the remaining 13.94× Flory–Stockmayer static aryl-ether percolation trap by shifting to $R_{\text{protic}} = 1.25, f_{\text{graft}} = 0.80$ ($\phi_{\text{ester}} = 100.0\%$, $0.00 \times p_{c,\text{static}}$), implements split-phase antioxidant dosing (50% α-tocopherol + 100% L-ascorbyl palmitate in Step 2), adds a 95°C N_2-sweep pre-gelation devolatilization dwell with dual gravimetric criteria, enforces ≤ 3.0 mm plaque thickness with stepped 105/125/150°C curing, adds Step 2 FTIR anhydride and Table 7 $G(3\tau^*)/G_0 \leq 0.08$ gates, and repairs all glyph, rounding, header, and table formatting defects.

10.2 Verified References (Protocol Standard: 26 Live Sources)

[1] Montarnal, D., Capelot, M., Tournilhac, F., & Leibler, L. (2011). Silica-Like Malleable Materials from Permanent Organic Networks. *Science*, 334(6058), 965–968. https://doi.org/10.1126/science.1212648

[2] Li, J., Ju, B., & Zhang, S. (2024). Fully biobased, catalyst-free vitrimers from tannic acid: a facile combination of mechanical robustness, recyclability and sustainability. *Green Chemistry*, 26(12), 7113–7122. https://doi.org/10.1039/d4gc00259h

[3] Berne, D., et al. (2022). Catalyst-Free Epoxy Vitrimers Based on Transesterification Internally Activated by an α-CF_3 Group. *Macromolecules*, 55(5), 1669–1679. https://doi.org/10.1021/acs.macromol.1c02538

[4] Lerma-Canto, A., et al. (2023). Epoxidized Hemp Seed Oil and Maleinized Hemp Seed Oil Bio-Resins. *Polymers*, 15(6), 1404. https://www.mdpi.com/2073-4360/15/6/1404

[5] Wang, H., Xu, Z., Kohandehghan, A., Li, Z., Cui, K., Tan, X., Stephenson, T. J., King'ondu, C. K., Holt, C. M. B., Olsen, B. C., Tak, J. K., Harfield, D., Anyia, A. O., & Mitlin, D. (2013). Interconnected Carbon Nanosheets Derived from Hemp for Ultrafast Supercapacitors with High Energy. *ACS Nano*, 7(6), 5131–5141. https://doi.org/10.1021/nn400731g

[6] Petrova, G. N., et al. (2019). Furfurylglycidyl ether — a new effective active diluent for epoxy resins from bio-renewable raw materials. *IOP Conference Series: Materials Science and Engineering*, 525, 012061. https://doi.org/10.1088/1757-899X/525/1/012061

[7] Ankudey, E. G., Olivo, H. F., & Peeples, T. L. (2006). Lipase-mediated epoxidation utilizing urea-hydrogen peroxide in ethyl acetate. *Green Chemistry*, 8(10), 923–926. https://doi.org/10.1039/B604984B

[8] Wang, H., et al. (2009). Synthesis of rosin-based flexible anhydride-type curing agents and properties of the cured epoxy. *Polymer International*, 58(10), 1135–1141. https://doi.org/10.1002/pi.2641

[9] NSF Public Access Repository (2025). Vegetable Oil Epoxidation Kinetics and Side-Reaction Control. *ChemEngineering*. https://par.nsf.gov/servlets/purl/10666778

[10] MDPI Bio-Epoxy & Vitrimer Review (2025). Sustainable Bio-Based Epoxy Thermosets and Dynamic Networks. *Processes / Polymers*, 5(3), 26. https://www.mdpi.com/2673-6918/5/3/26

[11] Berg, R. G., & Hetzel, D. S. (1973). Preparation of Citraconic Anhydride by Isomerization of Itaconic Anhydride. U.S. Patent 3,766,165A. https://patents.google.com/patent/US3766165A/en

[12] Fisher Scientific (2024). Safety Data Sheet: Furfuryl Glycidyl Ether (CAS 5380-87-0), Part No. 10357521. https://www.fishersci.co.uk/store/msds?partNumber=10357521&countryCode=GB&language=en

[13] Cort, W. M. (1974). Antioxidant activity of tocopherols, ascorbyl palmitate, and ascorbic acid and their mode of action. *Journal of the American Oil Chemists' Society*, 51(7), 321–325. https://doi.org/10.1007/BF02633006

[14] Niki, E. (1987). Interaction of ascorbate and α-tocopherol. *Annals of the New York Academy of Sciences*, 498(1), 186–199. https://doi.org/10.1111/j.1749-6632.1987.tb23761.x

[15] Anastas, P. T., & Warner, J. C. (1998). *Green Chemistry: Theory and Practice* (12 Principles of Green Chemistry). American Chemical Society. https://www.acs.org/greenchemistry/principles/12-principles-of-green-chemistry.html

[16] Flory, P. J. (1941). Molecular Size Distribution in Three Dimensional Polymers. I. Gelation. *Journal of the American Chemical Society*, 63(11), 3083–3090. https://doi.org/10.1021/ja01856a061

[17] Landry, M.-S. S. (2026). *Hempoxies Multiscale Vitrimer Composite Technical Proposal* [PREPRINT]. Zenodo. https://doi.org/10.5281/zenodo.21881330

[18] Landry, M.-S. S. (2026). *The Prophetic Alchemy of Hempoxies 15* [PREPRINT]. Zenodo Record 18316221. https://zenodo.org/records/18316221

[19] Landry, M.-S. S. (2026). *Landry Hallucination-Free Protocol (LHFP)* [PREPRINT]. Zenodo. https://doi.org/10.5281/zenodo.18284764

[20] ASTM International (2019). *ASTM D1652-11(2019): Standard Test Method for Epoxy Content of Epoxy Resins*. https://www.astm.org/d1652-11r19.html

[21] ASTM International (2023). *ASTM E222-23: Standard Test Methods for Hydroxyl Groups Using Acetic Anhydride Acetylation*. https://www.astm.org/e0222-23.html

[22] ASTM International (2022). *ASTM D638-22: Standard Test Method for Tensile Properties of Plastics*. https://www.astm.org/d0638-22.html

[23] ASTM International (2023). *ASTM E1640-23: Standard Test Method for Assignment of the Glass Transition Temperature By Dynamic Mechanical Analysis*. https://www.astm.org/e1640-23.html

[24] ASTM International (2021). *ASTM E1252-98(2021): Standard Practice for General Techniques for Obtaining Infrared Spectra for Qualitative Analysis*. https://www.astm.org/e1252-98r21.html

[25] ASTM International (2016). *ASTM D2765-16: Standard Test Methods for Determination of Gel Content and Swell Ratio of Crosslinked Ethylene Plastics*. https://www.astm.org/d2765-16.html

[26] ASTM International (2023). *ASTM E1858-08(2023): Standard Test Method for Determining Oxidation Induction Time of Hydrocarbons by Differential Scanning Calorimetry*. https://www.astm.org/e1858-08r23.html