

Hempoxies Master Technical SOP Suite (8 Protocols): A Complete Research & Development Framework for the Synthesis, Integration, and Characterization of a 7-Component, 100% Hemp-Derived, Reprocessable Vitrimeric Bionanocomposite Epoxy System for High-Performance Structural Applications

Document ID: MLSS-HEMPOXY-SOP-MASTER.

Date: November 12, 2025

Classification: OSINT

Author & Strategic Lead: Marie-Soleil Seshat Landry, CEO, Spymaster

ORCID iD: 0009-0008-5027-3337

Organization: Marie Landry Spy Shop / Landry Industries Conglomerate

Target Facility: Centre de développement des composites du Québec (CDCQ), Saint-Jérôme, QC

ABSTRACT

This document details the complete Research & Development (R&D) framework for the **Hempoxies** platform, a novel, 100% hemp-derived, carbon-negative, reprocessable thermoset (vitramer) bionanocomposite. The core innovation lies in a robust 7-component architecture that leverages a dual-cure mechanism, combining a primary, irreversible epoxy-amine network for structural integrity with a secondary, thermally reversible imine network for reprocessability and recyclability. The system uniquely utilizes Modified Hemp Lignin (MHL) as a triple-function agent: a dynamic cross-linker, a reinforcing filler, and a compatibilizer. This master document provides a suite of eight Standard Operating Protocols (SOPs) for the synthesis of each component—Epoxidized Hemp Seed Oil (EHSO), Modified Hemp Lignin (MHL), Hemp-Derived Amine (HDA), Furfuryl Glycidyl Ether (FGE), and a multi-scale reinforcement network of Hemp-Derived Carbon Nanosheets (HDCNS), Biochar (HDB), and Carbon Fibers (HDCF). Also included are a master integration protocol, a precise stoichiometric calculation framework, a comprehensive Design of Experiments (DoE) grid for system optimization, and rigorous quality control standards. The strategic mandate is to develop a high-performance, sustainable, drop-in replacement for petroleum-based epoxy systems, targeting applications in aerospace, automotive, marine, and renewable energy sectors, with target material properties including a tensile strength of ≥ 60 MPa and a glass transition temperature (T_g) of 80-120°C.

TABLE OF CONTENTS

- Executive Summary & Strategic Mandate
- System Architecture & Component Definitions
- Synthesis Protocols (SOP-01 through SOP-07)
- Master Integration Protocol (SOP-08)
- Stoichiometric Calculation Framework

6. Design of Experiments (DoE) Grid
 7. Quality Control & Validation Standards
 8. Risk Assessment & Mitigation Strategies
 9. Cost Modeling Framework
 10. Intellectual Property & Attribution
 11. References
 12. Appendices
 - A: Batch Record Template
 - B: Stoichiometry Calculation Spreadsheet
 - C: Testing Schedule Template
 - D: Equipment List & Specifications
 - E: Safety Data Summary
 - F: Grant Application Support Materials
-

1. EXECUTIVE SUMMARY & STRATEGIC MANDATE

1.1 Innovation Overview

The **Hempoxies** platform represents a paradigm shift in sustainable materials engineering: a 100% hemp-derived, carbon-negative, reprocessable thermoset (vitrimers) designed for the Organic Revolution of 2030. This document provides the complete technical framework for Phase 1A R&D execution at CDCQ.

1.2 Core Innovation

The system leverages a robust **7-component architecture** that guarantees the final material functions as a reprocessable thermoset through dynamic covalent chemistry. Unlike conventional epoxies that are permanently cross-linked, Hempoxies incorporates dynamic imine bonds that enable thermal reprocessing while maintaining excellent mechanical properties at service temperatures.

1.3 Strategic Positioning

Target Markets:

- Aerospace secondary structures (interior panels, fairings)
- Automotive body panels and structural components
- Wind turbine blades (replacement for glass/carbon fiber epoxy)
- Marine applications (boat hulls, deck structures)
- Sports equipment (bicycles, snowboards, protective gear)

Competitive Advantages:

- 100% bio-based content (highest in industry)
- Carbon-negative footprint (when accounting for hemp cultivation)
- Reprocessable/recyclable (addresses end-of-life waste problem)
- Drop-in replacement for conventional epoxy systems
- Premium positioning: \$12-18/kg resin system (vs. \$8-12/kg for petroleum epoxy)

1.4 Attribution of Novelty

Credited to Marie-Soleil Seshat Landry:

1. Conceptual design of the 7-component architecture
2. Definition of MHL (Modified Hemp Lignin) triple functionality: filler, dynamic cross-linker, and compatibilizer
3. Specification of HDA (Hemp-Derived Amine) as structural hardener distinct from MHL dynamic agent
4. Integration strategy for multi-scale carbon reinforcement network (HDCNS/HDB/HDCF)
5. Optimization framework balancing mechanical performance with reprocessability

2. SYSTEM ARCHITECTURE & COMPONENT DEFINITIONS

2.1 The 7-Component System

IP Name	Component Designation	Primary Role	SOP	Typical Loading Range
Hemp-EPOXY Matrix	Epoxidized Hemp Seed Oil (EHSO)	Primary Epoxy Matrix	SOP-01	50-65%
Hemp-VITRIMER Agent	Modified Hemp Lignin (MHL)	Dynamic Cross-linker/Filler	SOP-02	5-15%
Hemp-AMINE Hardener	Hemp-Derived Amine (HDA)	Primary Curing Agent	SOP-03	15-25%
Hemp-REACTIVE Diluent	Furfuryl Glycidyl Ether (FGE)	Viscosity Control	SOP-04	7% (fixed)
Hemp-CARBON Nanosheet	Hemp-Derived Carbon Nanosheets (HDCNS)	Nano-Reinforcement	SOP-05	0.1-0.5%
Hemp-MICRO Biochar	Hemp-Derived Biochar (HDB)	Micro-Reinforcement	SOP-06	5% (fixed)

IP Name	Component Designation	Primary Role	SOP	Typical Loading Range
Hemp-CARBON Fiber	Hemp-Derived Carbon Fibers (HDCF)	Macro-Reinforcement	SOP-07	3% (fixed)

2.2 System Chemistry Overview

Cure Mechanism:

The system employs a dual-cure architecture:

1. Primary Epoxy-Amine Cure (Irreversible):

- EHSO epoxide groups + HDA amine groups → β-hydroxyamine cross-links
- FGE epoxide groups (residual) + HDA amine groups → additional cross-links
- This provides the primary structural network

2. Secondary Imine Exchange (Reversible):

- MHL aldehyde groups + HDA amine groups → imine bonds (Schiff bases)
- These dynamic bonds enable reprocessing at T > 140°C
- Exchange reaction: $R-CH=N-R' + H_2N-R'' \rightleftharpoons R-CH=N-R'' + H_2N-R'$

Vitrimer Behavior:

- Service temperature: T < 100°C (material behaves as conventional thermoset)
- Reprocessing temperature: T > 140°C (imine exchange allows flow/reshaping)
- Glass transition temperature (Tg): Target 80-120°C depending on formulation

2.3 Material Property Targets

Property	Target Value	Test Method
Tensile Strength	≥60 MPa	ASTM D638
Young's Modulus	2.5-4.0 GPa	ASTM D638
Strain at Break	3-6%	ASTM D638
Glass Transition (Tg)	80-120°C	DSC/DMA
Stress Relaxation Time (τ) at 160°C	10-30 min	DMA stress relaxation
Reprocessing Cycles	≥3 without 20% property loss	Custom protocol
Water Absorption (24h)	<1.5%	ASTM D570
Carbon Footprint	<0 kg CO ₂ eq/kg	LCA (ISO 14040)

3. SYNTHESIS PROTOCOLS (SOP-01 THROUGH SOP-07)

SOP-01: Synthesis of Hemp-EPOXY Matrix (EHSO)

Primary Epoxy Matrix

Document ID: HEMP-SOP-01-EHSO-V2.0

Principle: In situ performic acid epoxidation of unsaturated fatty acids (C=C double bonds) in hemp seed oil.

Validation & QC: Oxirane Value (OV) ≥8% via titration (AOCS Method Cd 9-57); FTIR confirmation (epoxide ring stretch at 824 cm⁻¹).

3.1.1 Equipment Required

- 2L jacketed glass reactor with mechanical stirrer, reflux condenser, and temperature probe
- Dropping funnel (500 mL)
- Rotary evaporator
- pH meter

3.1.2 Materials

- Hemp Seed Oil (HSO): Cold-pressed, food-grade, iodine value (IV) = 155-165
- Formic Acid (HCOOH): 88-90% purity, ACS grade
- Hydrogen Peroxide (H₂O₂): 35% aqueous solution
- Toluene: Anhydrous, ≥99.5%
- Sodium Bicarbonate (NaHCO₃): Powder, ACS grade
- Magnesium Sulfate (MgSO₄): Anhydrous, ACS grade

3.1.3 Procedure

Step	Procedure	Critical Parameters	Safety Notes
1.1	Prepare Reactants: Charge 1000 g HSO, 150 g HCOOH, and 200 mL Toluene into the reactor.	HSO:HCOOH molar ratio = 1:1.5 (based on calculated C=C moles). Temperature = 20°C.	Formic acid is corrosive. Use PPE.
1.2	Add Oxidizer: Begin stirring (300 rpm). Slowly add 400 mL H ₂ O ₂ dropwise over 2 hours via the dropping funnel.	Maintain reactor temperature 40-60°C via jacket cooling. CRITICAL: Exotherm can cause runaway reaction.	H ₂ O ₂ is a strong oxidizer. Add slowly. Have ice bath ready.
1.3	Reaction Phase: Continue stirring for 6 hours. Monitor temperature and sample every 2 hours for OV/IV testing.	Reaction complete when OV plateaus (≥8%) or IV <10.	Monitor for excessive gas evolution.

Step	Procedure	Critical Parameters	Safety Notes
1.4	Quenching & Washing: Cool to 30°C. Transfer to separatory funnel. Wash with 500 mL saturated NaHCO ₃ solution. Repeat until pH = 7.0.	Perform 3–4 washes minimum. Final pH of oil layer must be 7.0 ±0.2.	Bicarbonate addition is exothermic. Add slowly.
1.5	Drying: Add 50 g anhydrous MgSO ₄ to oil phase. Stir 30 min. Filter. Rotary evaporate residual toluene under vacuum (60°C, 50 mbar).	Final H ₂ O content <0.05% (Karl Fischer titration).	Store under N ₂ gas blanket in amber bottles.

3.1.4 Quality Control

- **Oxirane Value (OV):** Target ≥8.0% (AOCS Cd 9-57)
- **Iodine Value (IV):** Target <10 (confirms complete epoxidation)
- **Viscosity:** 500–1200 mPa·s at 25°C (Brookfield viscometer)
- **Color:** Gardner scale <6
- **Shelf Life:** 6 months at 4°C under N₂

SOP-02: Synthesis of Hemp-VITRIMER Agent (MHL)

Triple-Function Dynamic Agent / Filler

Document ID: HEMP-SOP-02-MHL-V2.0

Principle: Aldehyde grafting onto hemp lignin’s phenolic OH groups to create sites for imine dynamic exchange with HDA.

Validation & QC: CRITICAL: TGA (ash content <5%); FTIR confirmation of aldehyde (R-CHO stretch at 1680 cm⁻¹); Aldehyde content via hydroxylamine titration.

3.2.1 Equipment Required

- 1L three-neck round-bottom flask with mechanical stirrer, N₂ inlet, reflux condenser
- Vacuum filtration setup with Büchner funnel
- Vacuum oven

3.2.2 Materials

- Hemp Lignin: Organosolv or Kraft lignin from hemp bast, particle size <100 µm
- p-Hydroxybenzaldehyde: ≥98% purity (aldehyde grafting agent)
- Dimethylformamide (DMF): Anhydrous, ≥99.8%
- Sodium Hydroxide (NaOH): Pellets, ACS grade
- Hydrochloric Acid (HCl): 1M solution
- Ethanol: Absolute, ≥99.5%

3.2.3 Procedure

Step	Procedure	Critical Parameters	Safety Notes
2.1	Prepare Lignin: Dry 200 g Hemp Lignin in vacuum oven (80°C, 12 hours, <1 mbar).	Particle size <100 µm (ball mill if needed). Moisture content <0.5%.	N/A
2.2	Solvation & Activation: Dissolve dried lignin in 600 mL DMF in the flask under N ₂ . Heat to 60°C with stirring. Add 12 g NaOH (dissolved in 50 mL DMF). Stir 1 hour.	Temperature = 60°C. Concentration = 10-15% w/v. NaOH deprotonates phenolic -OH.	DMF is toxic. Use fume hood. N ₂ prevents oxidation.
2.3	Aldehyde Grafting: Add 80 g p-hydroxybenzaldehyde (1.5 molar excess to estimated phenolic OH). Heat to 80°C. Stir 10 hours under continuous N ₂ flow.	Reaction time = 8-12 hours. Temperature = 80°C. Monitor by FTIR (aldehyde peak emergence).	p-Hydroxybenzaldehyde is an irritant.
2.4	Purification: Cool to RT. Slowly add reaction mixture to 2L acidified water/ethanol (50:50, pH = 3 with HCl). Precipitate forms. Filter under vacuum.	pH <4 for complete precipitation. Wash precipitate with acidified ethanol until filtrate pH = 7.	Exothermic precipitation. Add slowly.
2.5	Drying & Characterization: Dry MHL in vacuum oven (60°C, 24 hours, <1 mbar) to constant weight.	CRITICAL: Run TGA to determine inorganic ash content. Must be <5% for proper dispersion.	N/A

3.2.4 Quality Control

- **Aldehyde Content:** 2.5-4.0 mmol/g (hydroxylamine HCl titration)
- **TGA Ash Content:** <5%
- **FTIR:** Aldehyde C=O stretch at 1680 cm⁻¹; Aromatic C=C at 1510, 1600 cm⁻¹
- **Particle Size:** D₅₀ <50 µm (laser diffraction)
- **Moisture Content:** <0.5% (Karl Fischer)

3.2.5 Contingency: Sourcing Challenges

If p-hydroxybenzaldehyde is unavailable or cost-prohibitive:

- **Alternative 1:** Use vanillin (4-hydroxy-3-methoxybenzaldehyde) - more readily available from lignin
- **Alternative 2:** Use glutaraldehyde (5-carbon dialdehyde) - lower reactivity but dual-functional

SOP-03: Synthesis of Hemp-AMINE Hardener (HDA)

Primary Structural Curing Agent

Document ID: HEMP-SOP-03-HDA-V2.0

Principle: Polycondensation of Hemp Dimer Acid with a hemp-derived or bio-based diamine to yield a polyamide amine hardener.

Validation & QC: Total Amine Value (TAV) = 150-250 mg KOH/g; Viscosity = 1,000-10,000 mPa·s at 25°C.

3.3.1 Equipment Required

- 1L three-neck flask with mechanical stirrer, vacuum adapter, Dean-Stark trap
- Heating mantle with temperature controller
- Vacuum pump (capable of <10 mbar)

3.3.2 Materials

- **Hemp Dimer Acid (HDA):** Derived from hemp fatty acid dimerization, Acid Value (AV) = 190-200 mg KOH/g
- **Diamine:** Bio-based options (in order of preference):
 1. **Preferred:** 1,10-Diaminodecane from castor oil (bio-based, commercially available)
 2. **Alternative 1:** Ethylenediamine (EDA) - low MW, high reactivity
 3. **Alternative 2:** Hemp protein hydrolysate enriched in lysine (experimental, requires fermentation)
- **Catalyst:** Hypophosphorous acid (H₃PO₂), 1% w/w (prevents oxidation)

3.3.3 Procedure

Step	Procedure	Critical Parameters	Safety Notes
3.1	Prepare Hemp Dimer Acid: Verify AV of dimer acid stock. If AV <190, reject batch.	HDA Purity ≥90%. AV = 190-200 mg KOH/g.	N/A
3.2	Source/Prepare Amine: Calculate Amine Equivalent Weight (AEW) of diamine. Verify purity by GC or titration.	Diamine purity ≥98%. For 1,10-diaminodecane: AEW = 86 g/eq.	Diamines are corrosive bases. Use PPE.
3.3	Polycondensation: Charge 500 g HDA, 180 g diamine (1.05 eq. amine to 1.00 eq. acid), and 5 g H ₃ PO ₂ into flask. Heat gradually to 220°C under N ₂ with stirring. Once 220°C is reached, apply vacuum (<10 mbar). Continue 4-6 hours.	Stoichiometry: 1.05 Amine Eq. to 1.00 Acid Eq. Temperature = 220-250°C. Remove water continuously via Dean-Stark trap under vacuum.	High temperature and vacuum. Monitor for bumping.

Step	Procedure	Critical Parameters	Safety Notes
3.4	Termination & QC: Sample every hour and measure TAV. Stop when TAV = 150-250 mg KOH/g. Cool under N ₂ . Transfer to container.	TAV and Viscosity must be confirmed. Final product is viscous amber liquid/paste.	Allow to cool before sampling. Wear thermal gloves.

3.3.4 Quality Control

- **Total Amine Value (TAV):** 150-250 mg KOH/g (ASTM D2074)
- **Viscosity:** 1,000-10,000 mPa-s at 25°C (Brookfield, spindle #3, 50 rpm)
- **Amine Hydrogen Equivalent Weight (AHEW):** Calculate from TAV: $AHEW = 56100/TAV$
- **Color:** Gardner <8
- **Shelf Life:** 12 months at RT in sealed container under N₂

3.3.5 Contingency: Diamine Sourcing

If hemp protein-derived diamine is unavailable:

- Use 1,10-diaminodecane (bio-based from castor oil, commercially available from Evonik, Arkema)
- Acceptance criterion: This maintains ≥95% bio-based content (only amine source changes)
- Document sourcing in batch record for IP/marketing purposes

SOP-04: Synthesis of Hemp-REACTIVE Diluent (FGE)

Reactive Diluent / Viscosity Controller

Document ID: HEMP-SOP-04-FGE-V2.0

Principle: Glycidylation of Furfuryl Alcohol (derived from hemp hemicellulose) using Epichlorohydrin.

Validation & QC: Purity ≥99% via GC; Oxirane Value (OV) = 18-22%.

3.4.1 Equipment Required

- 1L three-neck flask with mechanical stirrer, reflux condenser, thermometer
- Separatory funnel (1L)
- Vacuum distillation apparatus

3.4.2 Materials

- **Furfuryl Alcohol:** Derived from hemp hemicellulose via acid hydrolysis and dehydration, purity ≥98%

- **Epichlorohydrin:** ≥99% purity, ACS grade
- **Sodium Hydroxide (NaOH):** 50% aqueous solution
- **Toluene:** Anhydrous, for azeotropic water removal

3.4.3 Procedure

Step	Procedure	Critical Parameters	Safety Notes
4.1	Source Furfuryl Alcohol: Verify purity by GC. If purity <98%, redistill before use.	Purity ≥98%. Water content <0.2%.	Furfuryl alcohol has strong odor. Use fume hood.
4.2	Etherification: Charge 200 g Furfuryl Alcohol and 480 g Epichlorohydrin (2:1 molar ratio) into flask. Heat to 70°C with stirring. Slowly add 160 g NaOH (50% solution) dropwise over 2 hours.	Molar ratio Epichlorohydrin:Alcohol = 2:1. Temperature = 70°C. NaOH addition rate: ~1 drop/second.	HIGHLY EXOTHERMIC. Ice bath on standby. Epichlorohydrin is carcinogenic.
4.3	Dehydrochlorination: After NaOH addition, raise temperature to 90°C. Stir 2 hours. White NaCl precipitate forms.	pH ≥10 (test with pH paper). Salt byproduct visible.	N/A
4.4	Washing & Purification: Cool. Transfer to separatory funnel. Wash with 500 mL saturated NaCl solution. Repeat 3x. Dry organic layer over MgSO ₄ . Vacuum distill at 80-90°C/10 mbar.	Collect distillate at 80-90°C under 10 mbar. Discard forerun and tail fractions.	Unreacted epichlorohydrin is carcinogenic. Proper disposal required.

3.4.4 Quality Control

- **Purity:** ≥99% by GC (FID detector)
- **Oxirane Value:** 18-22% (AOCS Cd 9-57)
- **Viscosity:** 5-15 mPa·s at 25°C
- **Color:** <50 APHA
- **Odor:** Characteristic furfural note (acceptable for industrial use)

SOP-05: Synthesis of Hemp-CARBON Nanosheet (HDCNS)

Nano-Scale Reinforcement / Conductivity

Document ID: HEMP-SOP-05-HDCNS-V2.0

Principle: Hydrothermal carbonization followed by high-temperature KOH activation/templating to achieve 2D nanosheet morphology.

Validation & QC: Raman spectroscopy (I_D/I_G ratio = 0.85-1.10); TEM confirmation of sheet morphology; BET surface area 800-1200 m²/g.

3.5.1 Equipment Required

- Hydrothermal autoclave (500 mL, rated to 300°C, 10 MPa)
- Tube furnace with Ar gas flow (max temp 1100°C)
- Ball mill or planetary mill

3.5.2 Materials

- **Hemp Biomass Precursor:** Hemp hurd or shiv, pre-carbonized at 400°C (from SOP-06 Step 6.2)
- **Potassium Hydroxide (KOH):** Pellets, ACS grade (activating agent)
- **Hydrochloric Acid (HCl):** 1M solution (for washing)
- **Argon Gas:** Ultra-high purity (99.999%)

3.5.3 Procedure

Step	Procedure	Critical Parameters	Safety Notes
5.1	Precursor Prep: Ball mill pre-carbonized hemp biochar to <10 µm particle size. Mix 50 g biochar with 200 g KOH (mass ratio 1:4) and 50 mL water to form slurry.	Particle size <10 µm (SEM verification). Mass ratio Precursor:KOH = 1:4.	KOH is highly corrosive. Use PPE.
5.2	Hydrothermal Treatment: Transfer slurry to autoclave. Seal. Heat to 220°C for 12 hours.	Temperature = 200–250°C. Pressure will auto-generate (~2–3 MPa). Time = 12 hours.	Follow autoclave safety protocols. Pressure relief valve must be functional.
5.3	Activation: Transfer carbonized material to alumina crucible. Place in tube furnace under Ar flow (200 mL/min). Heat at 5°C/min to 900°C. Hold 3 hours. Cool to RT under Ar.	Temperature = 800–1000°C. Heating rate = 5°C/min. Hold time = 2–4 hours. Ar flow = 200 mL/min.	High temperature. Ensure furnace seals are intact. KOH vapors are corrosive.
5.4	Purification: Wash activated carbon repeatedly with 1M HCl (500 mL portions) until pH = 7, then wash with DI water until conductivity <10 µS/cm. Dry at 120°C overnight.	pH must be 6.5–7.5 final. Remove all K salts (verify by ICP-OES or AAS if available).	HCl washing generates H ₂ gas. Perform in fume hood.

3.5.4 Quality Control

- **Raman Spectroscopy:** I_D/I_G ratio = 0.85–1.10 (indicates graphitic ordering)
- **TEM:** Confirm sheet morphology; thickness <10 nm
- **BET Surface Area:** 800–1200 m²/g
- **Particle Size:** D₅₀ = 1–5 µm (lateral dimension)
- **Ash Content:** <2% (TGA)

SOP-06: Synthesis of Hemp-MICRO Biochar (HDB)

Micro-Scale Reinforcement / Cost Reduction

Document ID: HEMP-SOP-06-HDB-V2.0

Principle: Low-temperature pyrolysis of hemp hurd/shiv under N₂ to retain moderate surface activity while maintaining cost-effectiveness.

Validation & QC: Consistent particle size D₅₀ = 10 ±3 µm; Fixed carbon content 70-80%; Surface area 150-300 m²/g.

3.6.1 Equipment Required

- Rotary retort furnace or fixed-bed pyrolysis reactor (batch size 1-5 kg)
- N₂ gas supply with flow controller
- Ball mill or jet mill

3.6.2 Materials

- **Hemp Hurd/Shiv:** Agricultural waste stream, moisture <10%
- **Nitrogen Gas:** Industrial grade (≥99.9%)

3.6.3 Procedure

Step	Procedure	Critical Parameters	Safety Notes
6.1	Feedstock Prep: Air-dry hemp hurd to <5% moisture. Grind in hammer mill to 100-500 µm particle size.	Moisture <5%. Pre-milling size = 100-500 µm.	Dust explosion hazard during milling. Ground outside or with explosion-proof equipment.
6.2	Pyrolysis: Load 2 kg feedstock into retort furnace. Purge with N ₂ (10 min, 5 L/min). Heat rapidly to 550°C at 20°C/min under continuous N ₂ flow (2 L/min). Hold 60 min. Cool to <100°C under N ₂ before opening.	Target Temperature = 450-600°C. Heating rate = 20°C/min. Residence Time = 60 minutes. N ₂ flow prevents combustion.	Pyrolysis vapors are flammable. Ensure furnace is sealed. Have fire suppression ready.
6.3	Cooling & Milling: Cool biochar under N ₂ flow to <100°C. Transfer to sealed container. Mill to D ₅₀ = 10 µm using ball mill or jet mill.	Final particle size D ₅₀ = 10 ±3 µm for optimal dispersion.	Biochar dust can be a respiratory irritant. Use dust mask.

3.6.4 Quality Control

- **Particle Size:** D₅₀ = 10 ±3 µm (laser diffraction)
- **Fixed Carbon Content:** 70-80% (ASTM D3172)
- **Volatile Matter:** 15-25%
- **Ash Content:** <5% (ASTM D3174)

- **BET Surface Area:** 150-300 m²/g
- **pH:** 7-9 (in water slurry)

SOP-07: Synthesis of Hemp-CARBON Fiber (HDCF)

Macro-Scale Reinforcement / Structural Strength

Document ID: HEMP-SOP-07-HDCF-V2.0

Principle: Stabilization (pre-oxidation) of aligned hemp bast fibers followed by high-temperature carbonization under inert atmosphere.

Validation & QC: Tensile strength ≥1.0 GPa; Sizing application uniformity (visual + FTIR).

3.7.1 Equipment Required

- Convection oven for stabilization (max temp 350°C)
- Tube furnace for carbonization (max temp 1200°C)
- Fiber tensile testing apparatus (ASTM D3379)

3.7.2 Materials

- **Hemp Bast Fibers:** Decorticated, cleaned, aligned bundles (2-5 cm length)
- **Pre-treatment Solution:** 5% NaOH aqueous solution (removes pectin/wax)
- **Sizing Agent:** Maleated hemp seed oil (bio-based coupling agent)
- **Nitrogen Gas:** Ultra-high purity (99.999%)

3.7.3 Procedure

Step	Procedure	Critical Parameters	Safety Notes
7.1	Feedstock Prep: Soak hemp bast fibers in 5% NaOH solution at 80°C for 2 hours. Rinse thoroughly with DI water until pH = 7. Dry at 80°C overnight.	Fiber length = 2-5 cm. NaOH removes wax/pectin for better carbonization.	NaOH is corrosive. Use PPE.
7.2	Stabilization/Pre-Oxidation: Place aligned fibers in convection oven. Heat slowly from 150°C to 280°C over 8 hours (ramp rate = 2°C/min). Hold at 280°C for 2 hours in air.	Temperature ramp: 150°C → 280°C at <5°C/min. Hold time = 2 hours. Air atmosphere allows oxidative cross-linking.	Fire hazard at high temps. Monitor continuously.
7.3	Carbonization: Transfer stabilized fibers to tube furnace under N ₂ flow (500 mL/min). Heat to 1000°C at 10°C/min. Hold 30 minutes. Cool to <100°C under N ₂ .	Target Temperature = 900-1100°C. Heating rate = 10°C/min. N ₂ flow = 500 mL/min prevents oxidation.	High temperature carbonization. Ensure furnace integrity.

Step	Procedure	Critical Parameters	Safety Notes
7.4	Sizing (Coupling Agent): Prepare 1% maleated hemp oil solution in ethanol. Dip carbonized fibers for 30 seconds. Dry at 120°C for 1 hour.	Sizing mass fraction ~1.0% (weigh before/after). Improves interfacial adhesion with EHSO matrix.	Ethanol is flammable. Perform in fume hood.

3.7.4 Quality Control

- **Tensile Strength:** ≥1.0 GPa (single fiber testing, ASTM D3379)
- **Fiber Diameter:** 8-15 μm (optical microscopy)
- **Carbon Content:** ≥90% (elemental analysis)
- **Sizing Coverage:** Uniform via SEM; FTIR confirms maleate ester peaks
- **Yield:** 25-35% by weight from original hemp bast

4. MASTER INTEGRATION PROTOCOL (SOP-08)

SOP-08: Master Compounding & Cure Protocol

Integration of All 7 Components into Final Hempoxies Composite

Document ID: HEMP-SOP-08-MASTER-V2.0

Pre-requisites: All 7 component synthesis SOPs (SOP-01 through SOP-07) must be complete with QC verification.

4.1 Fixed Baseline Components

To ensure statistical control and a complete 7-component system in every DoE run, the following components are held at constant weight percentages:

IP Name	Component	SOP	Fixed Loading (% wt.)	Rationale
Hemp-REACTIVE Diluent	FGE	SOP-04	7.0%	Viscosity reduction; enables processing at RT without heating
Hemp-MICRO Biochar	HDB	SOP-06	5.0%	Micro-scale reinforcement baseline; cost reduction filler
Hemp-CARBON Fiber	HDCF	SOP-07	3.0%	Macro-scale structural integrity; ensures minimum strength threshold

Total Fixed Components: 15.0% of final mass

4.2 Variable Components (DoE Optimization Variables)

The remaining 85% by weight consists of:

- Hemp-EPOXY Matrix (EHSO) - Variable, adjusted by stoichiometry
- Hemp-VITRIMER Agent (MHL) - **Variable: 5-15%** (DoE Factor)
- Hemp-AMINE Hardener (HDA) - Variable, determined by stoichiometric ratio (DoE Factor)
- Hemp-CARBON Nanosheet (HDCNS) - **Variable: 0.1-0.5%** (DoE Factor)

4.3 Compounding Procedure

4.3.1 Equipment Required

- High-shear mixer (overhead stirrer, 1000-2000 rpm capability)
- Ultrasonic probe (400W, 20 kHz)
- Analytical balance (± 0.001 g precision)
- Vacuum degassing chamber
- Heated molds (aluminum, PTFE-coated) per ASTM D638 Type I tensile bars
- Convection oven with programmable temperature control

4.3.2 Master Compounding Steps

Step	Procedure	Critical Parameters	Rationale
8.1	Prepare Fillers: Dry HDCNS, HDB, and HDCF in vacuum oven at 80°C for 4 hours. Store in desiccator until use.	Moisture content <0.1% (Karl Fischer verification).	Water interferes with epoxy-amine cure and causes voids.
8.2	Nano-Dispersion (Masterbatch A): Weigh HDCNS and FGE according to run specification. Add to 250 mL beaker. Sonicate with ultrasonic probe for 60 minutes (40% amplitude, pulse mode: 5s on/2s off).	FGE acts as dispersant. Sonication power = 400W. Bath temperature <40°C (use ice bath).	Ensures HDCNS exfoliation and prevents agglomeration. Critical for nano-reinforcement effectiveness.
8.3	Resin Compounding (Mix A): In a separate container, weigh EHSO and MHL according to DoE run specifications. Add Masterbatch A from Step 8.2. Add pre-weighed HDB and HDCF. Mix with high-shear mixer at 1500 rpm for 10 minutes. Scrape sides every 3 minutes.	Temperature <40°C (exotherm control). Visual inspection: uniform gray-black color, no agglomerates.	Incorporates all fillers into epoxy matrix. Ensures homogeneity before hardener addition.

Step	Procedure	Critical Parameters	Rationale
8.4	Hardener Dosing (Part B): Calculate required HDA mass using the Stoichiometry Framework (Section 5). Weigh HDA precisely. DO NOT ADD YET.	Weighing accuracy ± 0.001 g. CRITICAL: Use fresh stoichiometry calculation for each run accounting for actual component equivalents.	Accurate stoichiometry determines cure completeness and final properties.
8.5	Final Mixing & Degassing: Add HDA (Part B) to Mix A. Hand mix for 1 minute, then high-shear mix at 500 rpm for 3 minutes (avoid air entrainment). Transfer to vacuum chamber and degas at 50 mbar for 5 minutes until no bubbles emerge.	Mixing must be complete within pot life (typically 20-30 minutes at 25°C). Degassing time: 5 minutes.	Removes entrained air; ensures void-free castings.
8.6	Casting & Cure: Pour degassed resin into pre-warmed molds (40°C). Cure per DoE specification: Initial cure at 80°C for 2 hours, then post-cure at specified T_cure and time from DoE grid.	Use mold release agent (PTFE spray or paste wax). Cure profile per DoE grid (Section 6).	Multi-stage cure maximizes conversion of both epoxy-amine and imine reactions.
8.7	Demolding & Conditioning: Cool molds to RT. Demold specimens. Store in desiccator for 24 hours before testing.	Allow complete cooling before demolding to prevent warping.	Conditioning ensures consistent moisture content for testing.

5. STOICHIOMETRIC CALCULATION FRAMEWORK

5.1 Overview

The Hempoxies system contains **three reactive components** that must be precisely balanced:

- EHSO** - epoxide groups (from epoxidized hemp oil)
- FGE** - epoxide groups (from furfuryl glycidyl ether)
- MHL** - aldehyde groups (on modified lignin)

These react with:

- HDA** - amine groups (from hemp-derived polyamide hardener)

5.2 Key Definitions

Epoxide Equivalent Weight (EEW): Grams of resin containing 1 equivalent of epoxide groups

- For EHSO: $EEW = 100,000 / OV$ (where OV is Oxirane Value in %)
- Example: If EHSO has $OV = 8.5\%$, then $EEW_{EHSO} = 100,000 / 8.5 = 11,765 \text{ g/eq}$

Amine Hydrogen Equivalent Weight (AHEW): Grams of hardener containing 1 equivalent of active hydrogen

- For HDA: $AHEW = 56,100 / TAV$ (where TAV is Total Amine Value in mg KOH/g)
- Example: If HDA has $TAV = 200$, then $AHEW_{HDA} = 56,100 / 200 = 280.5 \text{ g/eq}$

Aldehyde Equivalent Weight (AldEW): Grams of MHL containing 1 equivalent of aldehyde groups

- For MHL: $AldEW = 1000 / \text{Aldehyde_Content}$ (where Aldehyde_Content in mmol/g)
- Example: If MHL has 3.0 mmol/g aldehydes, then $AldEW_{MHL} = 1000 / 3.0 = 333 \text{ g/eq}$

5.3 Stoichiometric Ratio (r)

The DoE grid specifies **r_Hardener/ReactiveGroups** which is defined as:

$$r = (\text{Total Amine Equivalents}) / (\text{Total Epoxide Equivalents} + \text{Total Aldehyde Equivalents})$$

Where:

- **Total Amine Equivalents** = $(\text{Mass}_{HDA}) / AHEW_{HDA}$
- **Total Epoxide Equivalents** = $(\text{Mass}_{EHSO} / EEW_{EHSO}) + (\text{Mass}_{FGE} / EEW_{FGE})$
- **Total Aldehyde Equivalents** = $(\text{Mass}_{MHL}) / AldEW_{MHL}$

5.4 Calculation Procedure for Each DoE Run

Given from DoE Grid:

- Total batch mass: M_{total} (typically 500 g for lab scale)
- $r_{Hardener/ReactiveGroups}$ (e.g., 0.95, 1.00, 1.05)
- L_{MHL} (% wt. of MHL, e.g., 5%, 10%, 15%)
- C_{HDCNS} (% wt. of HDCNS, e.g., 0.1%, 0.25%, 0.5%)
- Fixed components: $FGE = 7\%$, $HDB = 5\%$, $HDCF = 3\%$

Step-by-Step Calculation:

1. Calculate Fixed Component Masses:

- $M_{FGE} = M_{total} \times 0.07$
- $M_{HDB} = M_{total} \times 0.05$
- $M_{HDCF} = M_{total} \times 0.03$
- $M_{MHL} = M_{total} \times L_{MHL}$ (from DoE grid)
- $M_{HDCNS} = M_{total} \times C_{HDCNS}$ (from DoE grid)

2. Calculate Remaining Mass for EHSO + HDA:

$$M_{\text{remaining}} = M_{\text{total}} - M_{\text{FGE}} - M_{\text{HDB}} - M_{\text{HDCF}} - M_{\text{MHL}} - M_{\text{HDCNS}}$$

3. Set Up Stoichiometry Equations:

Let M_{HDA} = mass of hardener to determine

$$\text{Then } M_{\text{EHSO}} = M_{\text{remaining}} - M_{\text{HDA}}$$

From the stoichiometric ratio definition:

$$r = (M_{\text{HDA}} / A_{\text{HEW_HDA}}) / [(M_{\text{EHSO}} / E_{\text{EW_EHSO}}) + (M_{\text{FGE}} / E_{\text{EW_FGE}}) + (M_{\text{MHL}} / A_{\text{dEW_MHL}})]$$

4. Solve for M_{HDA} :

Rearranging and substituting for M_{EHSO} :

$$M_{\text{HDA}} = (r \times A_{\text{HEW_HDA}} \times [(M_{\text{remaining}} / E_{\text{EW_EHSO}}) + (M_{\text{FGE}} / E_{\text{EW_FGE}}) + (M_{\text{MHL}} / A_{\text{dEW_MHL}})]) / (1 + (r \times A_{\text{HEW_HDA}} / E_{\text{EW_EHSO}}))$$

5. Calculate M_{EHSO} :

$$M_{\text{EHSO}} = M_{\text{remaining}} - M_{\text{HDA}}$$

5.5 Worked Example

Given:

- $M_{\text{total}} = 500 \text{ g}$
- $r = 1.00$ (stoichiometric balance)
- $L_{\text{MHL}} = 10\%$ ($M_{\text{MHL}} = 50 \text{ g}$)
- $C_{\text{HDCNS}} = 0.25\%$ ($M_{\text{HDCNS}} = 1.25 \text{ g}$)
- Component properties:
 - EHSO: $OV = 8.5\% \rightarrow E_{\text{EW_EHSO}} = 11,765 \text{ g/eq}$
 - FGE: $OV = 20\% \rightarrow E_{\text{EW_FGE}} = 5,000 \text{ g/eq}$ (assuming for calculation)
 - HDA: $TAV = 200 \text{ mg KOH/g} \rightarrow A_{\text{HEW_HDA}} = 280.5 \text{ g/eq}$
 - MHL: Aldehyde content = $3.0 \text{ mmol/g} \rightarrow A_{\text{dEW_MHL}} = 333 \text{ g/eq}$

Calculations:

1. Fixed masses:

- $M_{\text{FGE}} = 500 \times 0.07 = 35 \text{ g}$
- $M_{\text{HDB}} = 500 \times 0.05 = 25 \text{ g}$
- $M_{\text{HDCF}} = 500 \times 0.03 = 15 \text{ g}$

- $M_{MHL} = 50 \text{ g}$
- $M_{HDCNS} = 1.25 \text{ g}$

2. $M_{\text{remaining}} = 500 - 35 - 25 - 15 - 50 - 1.25 = 373.75 \text{ g}$

3. Calculate equivalents from FGE and MHL:

- $Eq_{FGE} = 35 / 5000 = 0.007 \text{ eq}$
- $Eq_{MHL} = 50 / 333 = 0.150 \text{ eq}$

4. Solve for M_{HDA} using the formula from 5.4:

$$M_{HDA} = (1.0 \times 280.5 \times [(373.75 / 11,765) + 0.007 + 0.150]) / (1 + (1.0 \times 280.5 / 11,765))$$

$$M_{HDA} = (280.5 \times [0.03176 + 0.007 + 0.150]) / (1 + 0.02383)$$

$$M_{HDA} = (280.5 \times 0.18876) / 1.02383$$

$$M_{HDA} = 52.95 / 1.02383 = \mathbf{51.72 \text{ g}}$$

5. $M_{EHSO} = 373.75 - 51.72 = \mathbf{321.03 \text{ g}}$

Final Formulation for This DoE Run:

- EHSO: 321.03 g (64.2%)
 - MHL: 50.00 g (10.0%)
 - HDA: 51.72 g (10.3%)
 - FGE: 35.00 g (7.0%)
 - HDCNS: 1.25 g (0.25%)
 - HDB: 25.00 g (5.0%)
 - HDCF: 15.00 g (3.0%)
 - **Total: 500.00 g**
-

6. DESIGN OF EXPERIMENTS (DoE) GRID

6.1 Experimental Variables

Independent Variables (Factors):

1. **r_Hardener/ReactiveGroups:** Stoichiometric ratio (0.95, 1.00, 1.05)
 - Controls cure completeness and cross-link density
2. **L_MHL (% wt.):** Modified Hemp Lignin loading (5%, 10%, 15%)
 - Controls dynamic bond density and reprocessability
3. **C_HDCNS (% wt.):** Carbon Nanosheet loading (0.1%, 0.25%, 0.5%)

- Controls nano-reinforcement and stiffness
4. **T_{cure} (°C):** Post-cure temperature (100°C, 120°C, 140°C)
- Controls reaction completion and final T_g

Fixed Variables:

- FGE: 7.0%, HDB: 5.0%, HDCF: 3.0%
- Initial cure: 80°C for 2 hours (all runs)

Dependent Variables (Responses):

1. **T_g (°C):** Glass transition temperature (DSC/DMA)
2. **σ_{tensile} (MPa):** Tensile strength at break (ASTM D638)
3. **E (GPa):** Young’s modulus (ASTM D638)
4. **ε_{break} (%):** Strain at break (ASTM D638)
5. **τ (min):** Stress relaxation time at 160°C (DMA)
6. **Reprocessability:** Property retention after 3 heat/press cycles

6.2 DoE Matrix (20 Runs + 3 Validation)

Run #	r	L_MHL (%)	C_HDCNS (%)	T_cure (°C)	Time (h)	Notes
1	1.05	5.0	0.25	100	4	Low MHL, balanced ratio
2	1.05	15.0	0.25	100	4	High MHL, balanced ratio
3	0.95	5.0	0.25	100	8	Low MHL, amine deficient
4	0.95	15.0	0.25	100	8	High MHL, amine deficient
5	1.00	10.0	0.10	120	4	Low HDCNS
6	1.00	10.0	0.50	120	4	High HDCNS
7	1.05	10.0	0.25	120	6	Center point variant
8	0.95	10.0	0.25	120	6	Center point variant
9	1.00	5.0	0.10	140	2	High temp, short time
10	1.00	15.0	0.50	140	2	High temp, all high
11	1.05	15.0	0.10	140	4	High temp, mixed
12	0.95	5.0	0.50	140	4	High temp, mixed
13	1.00	5.0	0.25	100	6	Low temp, low MHL
14	1.00	15.0	0.25	100	6	Low temp, high MHL

Run #	r	L_MHL (%)	C_HDCNS (%)	T_cure (°C)	Time (h)	Notes
15	1.05	10.0	0.50	140	8	Long cure, high HDCNS
16	0.95	10.0	0.10	140	8	Long cure, low HDCNS
17	1.00	10.0	0.25	120	4	CENTER POINT 1
18	1.00	10.0	0.25	120	6	CENTER POINT 2
19	1.05	5.0	0.50	120	6	Edge condition
20	0.95	15.0	0.10	120	6	Edge condition
21	1.00	10.0	0.25	120	5	CENTER POINT 3(validation)
22	TBD	TBD	TBD	TBD	TBD	Optimal formulation (post-analysis)
23	TBD	TBD	TBD	TBD	TBD	Optimal validation replicate

6.3 Statistical Analysis Plan

Software: JMP Pro, Minitab, or Design-Expert

Analysis Methods:

- Response Surface Methodology (RSM):** Fit quadratic models for each response.
- ANOVA:** Determine significant factors and interactions ($\alpha = 0.05$).
- Multi-objective Optimization:** Maximize σ_{tensile} and minimize τ simultaneously.

Success Criteria for Optimization:

- T_g: 80-120°C
- σ_{tensile} : ≥60 MPa
- τ at 160°C: 10-30 minutes
- Property retention after 3 cycles: ≥80%

Here is the continuation of the finalized document.

7. QUALITY CONTROL & VALIDATION STANDARDS

7.1 Component-Level QC (Summary)

Component	Key QC Parameter	Method	Acceptance Criterion
-----------	------------------	--------	----------------------

Component	Key QC Parameter	Method	Acceptance Criterion
EHSO	Oxirane Value	AOCS Cd 9-57	≥8.0%
MHL	Aldehyde Content	Hydroxylamine titration	2.5-4.0 mmol/g
HDA	Total Amine Value	ASTM D2074	150-250 mg KOH/g
FGE	Purity	GC-FID	≥99%
HDCNS	BET Surface Area	N ₂ adsorption	800-1200 m ² /g
HDB	Particle Size D ₅₀	Laser diffraction	10 ±3 μm
HDCF	Tensile Strength	ASTM D3379	≥1.0 GPa

7.2 Final Composite Testing Protocol

For Each DoE Run:

1. Mechanical Testing (ASTM D638):

- Cast 5 replicates of Type I tensile bars.
- Test at 25°C, 5 mm/min crosshead speed.
- Record: σ_{tensile} , E, ϵ_{break} .
- Report: Mean ±SD.

2. Thermal Analysis:

◦ DSC (Differential Scanning Calorimetry):

- Sample: 10-15 mg.
- Method: Heat 0°C → 200°C at 10°C/min under N₂.
- Measure: T_g (midpoint method).

◦ DMA (Dynamic Mechanical Analysis):

- Sample: 35×10×3 mm bar.
- Method: Temperature sweep -50°C → 180°C, 3°C/min, 1 Hz.
- Measure: T_g (tan δ peak), E' (storage modulus).

3. Stress Relaxation Test (Vitrimer Validation):

◦ DMA Stress Relaxation Mode:

- Sample: 35×10×3 mm bar.
- Pre-load to 1% strain at test temperature (160°C).
- Hold strain constant, record stress decay over time.
- Calculate τ : time for stress to decay to 1/e (37%) of its initial value.

4. Reprocessability Test:

- Cut a failed tensile bar into small pieces.
- Compression mold at 160°C, 10 MPa pressure, for 15 minutes.
- Cool under pressure and re-machine into new tensile bars.
- Repeat tensile testing.

- **Perform 3 cycles total.**
- Calculate property retention: $(\sigma_{\text{cycle3}} / \sigma_{\text{original}}) \times 100\%$.

5. Water Absorption (ASTM D570):

- Sample: 3 replicates, 50×50×3 mm plaques.
- Dry to constant weight (W_0), then immerse in DI water at 25°C for 24 hours.
- Blot dry and measure the final weight (W_{24}).
- Calculate: % Absorption = $[(W_{24} - W_0) / W_0] \times 100\%$.

7.3 Documentation & Traceability

For Each Batch:

- A Batch Record Sheet must document all component lot numbers.
- A signed stoichiometry calculation worksheet.
- QC test results for all components.
- Cure cycle time/temperature chart from the oven's data logger.
- Final property test reports.
- A deviation log for any out-of-spec conditions.

8. RISK ASSESSMENT & MITIGATION STRATEGIES

8.1 Technical Risks

Risk ID	Risk Description	Probability	Impact	Mitigation Strategy
T-01	EHSO epoxidation yields low OV (<8%)	Medium	High	Pre-qualify hemp oil suppliers; verify IV before epoxidation; optimize reaction time.
T-02	MHL contains excessive ash/inorganics	Medium	High	Source high-purity organosolv lignin; implement TGA screening; wash thoroughly.
T-03	HDA amine value drifts out of spec	Low	High	Implement real-time TAV monitoring during synthesis; adjust stoichiometry per batch.
T-04	HDCNS agglomerates during mixing	High	Medium	Pre-disperse in FGE with extended sonication; verify by TEM before compounding.
T-05	Incomplete cure (low Tg, weak properties)	Medium	High	Verify stoichiometry calculations; extend post-cure time; run DSC to confirm cure completion.

Risk ID	Risk Description	Probability	Impact	Mitigation Strategy
T-06	Excessive porosity in castings	Medium	Medium	Extend degassing time; reduce mixing speed; consider vacuum-assisted mold filling.

8.2 Safety Risks

Risk ID	Hazard	Severity	Control Measures
S-01	H ₂ O ₂ exotherm during epoxidation	High	Dropwise addition; ice bath on standby; continuous temperature monitoring; emergency shutdown procedure.
S-02	Epichlorohydrin carcinogenicity	High	Mandatory fume hood use; appropriate nitrile gloves; proper disposal via a licensed waste contractor.
S-03	Pyrolysis flammable vapor release	High	Sealed retort furnace; N ₂ purge; fire suppression system; operator training.
S-04	High-temperature burns (carbonization)	Medium	Thermal gloves; face shield; allow cooling before handling; mandatory SOP training.
S-05	NaOH/HCl chemical burns	Medium	PPE (goggles, gloves, lab coat); eyewash station; spill kit; neutralization agents readily available.

8.3 Supply Chain Risks

Component	Primary Supplier	Backup Strategy	Lead Time
Hemp Seed Oil	Local Quebec hemp farmers co-op	Alberta hemp processors	2-4 weeks
Hemp Lignin	Canadian biorefinery (pilot scale)	Import from EU (e.g., Lignin Technologies)	4-8 weeks
Hemp Dimer Acid	Custom synthesis	Purchase commercial dimer acid (Croda, Oleon)	6-12 weeks
Diamine	Bio-based: Evonik VESTAMIN®	Fallback: Huntsman Jeffamine®	2-6 weeks

9. COST MODELING FRAMEWORK

9.1 Component Cost Estimates (2025 CAD, Quebec)

Component	SOP	Yield (%)	Raw Material Cost (\$/kg)	Processing Cost (\$/kg)	Total Cost (\$/kg)
EHSO	01	85	Hemp oil: \$4.50; Chemicals: \$2.00	Energy + Labor: \$1.50	\$9.41
MHL	02	70	Lignin: \$2.00; Aldehyde: \$15.00	Energy + Labor: \$8.00	\$28.57
HDA	03	75	Dimer acid: \$8.00; Diamine: \$12.00	Energy + Labor: \$6.00	\$35.00
FGE	04	60	Furfuryl alcohol: \$6.00; Epi: \$3.00	Energy + Labor: \$5.00	\$23.33
HDCNS	05	50	Biochar: \$1.00; KOH: \$2.00	Energy + Labor: \$12.00	\$30.00
HDB	06	35	Hemp hurd: \$0.50	Energy + Labor: \$2.00	\$7.14
HDCF	07	30	Hemp bast: \$1.50	Energy + Labor: \$8.00	\$31.67

9.2 Hempoxies Resin System Cost (Example Formulation – Center Point)

- **EHSO:** 64.2% × \$9.41/kg = \$6.04/kg
- **MHL:** 10.0% × \$28.57/kg = \$2.86/kg
- **HDA:** 10.3% × \$35.00/kg = \$3.61/kg
- **FGE:** 7.0% × \$23.33/kg = \$1.63/kg
- **HDCNS:** 0.25% × \$30.00/kg = \$0.08/kg
- **HDB:** 5.0% × \$7.14/kg = \$0.36/kg
- **HDCF:** 3.0% × \$31.67/kg = \$0.95/kg

Total Material Cost: \$15.53/kg

Additional Costs:

- Compounding/Mixing: \$1.50/kg
- Quality Control: \$0.75/kg
- Overhead (20%): \$3.56/kg

Total Manufacturing Cost: \$21.34/kg

Target Selling Price: \$28–35/kg (premium bio-epoxy market)

9.3 Cost Reduction Opportunities

Strategy	Potential Savings	Timeline	Feasibility
Bulk lignin sourcing (>1 ton)	-\$8/kg on MHL	6 months	High
In-house dimer acid production	-\$12/kg on HDA	12 months	Medium
Scale up to 100 kg batches	-\$2/kg overall	12 months	High
Total Potential Reduction	~\$8-10/kg	18 months	Target: \$12-14/kg manufacturing cost

10. INTELLECTUAL PROPERTY & ATTRIBUTION

10.1 Novelty Attribution

All intellectual property rights and novelty claims arising from this work are attributed to **Marie-Soleil Seshat Landry** (ORCID ID: 0009-0008-5027-3337). The specific credited contributions include the 7-component system architecture, the definition of MHL's triple functionality, the multi-scale reinforcement strategy, and the overall vitrimer design principle.

10.2 Prior Art Analysis (Preliminary)

A preliminary search (November 2025) indicates strong novelty for the complete 7-component system and the MHL/HDA dual-cure architecture. While prior art exists for individual components (e.g., lignin-epoxy composites, imine-based vitrimers), their integration into a 100% hemp-derived, reprocessable system appears unique.

Recommendation: File a provisional patent application before any public disclosure.

10.3 IP Protection Strategy

- **Phase 1 (Months 0-6):** Maintain as a confidential trade secret under NDAs with all partners.
- **Phase 2 (Months 6-12):** File a provisional patent covering composition of matter, method of manufacture, and applications.
- **Phase 3 (Months 12-24):** Convert to a full utility patent and begin academic publications and conference presentations.

10.4 AI Assistance Disclaimer

This technical documentation was generated with assistance from large language models (Google Gemini, Anthropic Claude), which acted as strategic technical advisors for enforcing chemical engineering rigor, organizing documentation, applying statistical DoE methodology, and ensuring compliance with testing protocols. All conceptual innovation, system design, and strategic direction originated from Marie-Soleil Seshat Landry.

Here is the regenerated reference list and the complete, fully generated appendices for the document.

11. REFERENCES

- Goud, V. V., et al. (2007). "Epoxidation of karanja (*Pongamia glabra*) oil by H₂O₂." *Journal of the American Oil Chemists' Society*, 84(8), 781-789. <https://doi.org/10.1007/s11746-007-1096-7>
- Winne, J. M., et al. (2019). "Dynamic covalent polymer networks: a new paradigm in polymer chemistry." *Progress in Polymer Science*, 94, 1-33. <https://doi.org/10.1016/j.progpolymsci.2019.03.001>
- Karoki, P. K., et al. (2024). "Lignin-Based Vitrimers: A Review of Their Chemistry, Properties and Applications." *Materials Advances*, 5(3), 842-865. <https://doi.org/10.1039/D3MA00789H>
- Liu, Z., et al. (2017). "Synthesis and properties of bio-based amine curing agents from vegetable oil for epoxy coatings." *Progress in Organic Coatings*, 112, 117-126. <https://doi.org/10.1016/j.porgcoat.2017.07.013>
- Auvergne, R., et al. (2014). "Biobased thermosetting epoxy: present and future." *Chemical Reviews*, 114(2), 1082-1115. <https://doi.org/10.1021/cr3001274>
- Zhang, C., et al. (2019). "Synthesis of bio-based curing agents from plant oil derivatives and their application in epoxy resins." *Green Chemistry*, 21(12), 3308-3319. <https://doi.org/10.1039/C9GC00968J>
- Foyer, G., et al. (2016). "New method for the synthesis of formaldehyde-free phenolic resins from lignin." *European Polymer Journal*, 74, 296-309. <https://doi.org/10.1016/j.eurpolymj.2015.11.036>
- Vilela, C., et al. (2019). "The quest for sustainable polyesters – insights into the future." *Polymers*, 11(1), 114. <https://doi.org/10.3390/polym11010114>
- Wang, G., et al. (2014). "From biomass-derived nanoporous carbon to carbon-supported platinum nanoparticles." *Advanced Functional Materials*, 24(27), 4324-4331. <https://doi.org/10.1002/adfm.201400178>
- Li, Y., et al. (2017). "Synthesis of 2D carbon nanosheets from biomass via a low-temperature activation method." *Chemical Society Reviews*, 46(5), 1124-1164. <https://doi.org/10.1039/C6CS00424E>
- Chen, S., et al. (2017). "Functionalization of technical lignins for advanced polymer applications." *ACS Sustainable Chemistry & Engineering*, 5(12), 11325-11334.

<https://doi.org/10.1021/acssuschemeng.7b02821>

Duval, A., & Lawoko, M. (2014). "A review on lignin-based polymeric, micro- and nano-structured materials." *Reactive and Functional Polymers*, 85, 78–96.

<https://doi.org/10.1016/j.reactfunctpolym.2014.09.017>

Mariscal, R., et al. (2016). "Furfural: a renewable and versatile platform molecule for the synthesis of chemicals and fuels." *Energy & Environmental Science*, 9(4), 1144–1189.

<https://doi.org/10.1039/C5EE02666K>

Guo, C. (2011). "Synthesis and characterization of furfuryl glycidyl ether and its epoxy resins." *Journal of Applied Polymer Science*, 122(3), 1654–1660. <https://doi.org/10.1002/app.34275>

Bartoli, M., et al. (2020). "Bio-based carbon materials from hemp waste: From synthesis to characterization." *Journal of Analytical and Applied Pyrolysis*, 151, 104917.

<https://doi.org/10.1016/j.jaap.2020.104917>

Gacru, M., et al. (2019). "Hemp fiber reinforced polypropylene composites: Fiber treatments and mechanical properties." *Composites Part A: Applied Science and Manufacturing*, 120, 95–105.

<https://doi.org/10.1016/j.compositesa.2019.02.023>

Zhang, Z., et al. (2021). "Hemp-derived carbon fibers: A review on preparation, properties and applications." *Composites Part B: Engineering*, 217, 108879.

<https://doi.org/10.1016/j.compositesb.2021.108879>

Montgomery, D. C. (2017). *Design and Analysis of Experiments* (10th ed.). Wiley. ISBN: 978-1119492443

ASTM D638-14. (2014). *Standard Test Method for Tensile Properties of Plastics*. ASTM International.

<https://www.astm.org/d0638-14.html>

Samanta, S., et al. (2016). "Vegetable oil based epoxy resins and their composites with natural fibers." *Polymer-Plastics Technology and Engineering*, 55(1), 38–66.

<https://doi.org/10.1080/03602559.2015.1070871>

Pickering, K. L., et al. (2016). "A review of recent developments in natural fibre composites and their mechanical performance." *Composites Part A: Applied Science and Manufacturing*, 83, 98–112.

<https://doi.org/10.1016/j.compositesa.2015.08.038>

Wang, S., et al. (2019). "Lignin-based epoxy vitrimers with excellent reprocessability and adhesive properties." *ACS Sustainable Chemistry & Engineering*, 7(17), 15020–15028.

<https://doi.org/10.1021/acssuschemeng.9b03922>

Ma, S., et al. (2019). "Bio-based epoxy vitrimers: Reprocessability and their application in composites." *Polymer*, 178, 121604. <https://doi.org/10.1016/j.polymer.2019.121604>

Zhu, Y., et al. (2020). "Lignin-derived polyurethanes with enhanced mechanical and self-healing properties via dynamic disulfide bonds." *ChemSusChem*, 13(9), 2376-2385.

<https://doi.org/10.1002/cssc.202000301>

Tchoupa, A., et al. (2017). "Functionalization of biochar for use as filler in polymer composites." *Journal of Industrial and Engineering Chemistry*, 49, 184-193. <https://doi.org/10.1016/j.jiec.2017.01.026>

AOCS Official Method Cd 9-57. (Reapproved 2017). *Oxirane Oxygen*. American Oil Chemists' Society.

ASTM D2074-07. (Reapproved 2015). *Standard Test Methods for Total, Primary, Secondary, and Tertiary Amine Values of Fatty Amines by Potentiometric Method*. ASTM International.

Titirici, M. M., et al. (2007). "A direct synthesis of mesoporous carbons with ordered circuitry from new, sustainable precursors." *New Journal of Chemistry*, 31(5), 797-799. <https://doi.org/10.1039/B616045G>

Petrović, Z. S. (2008). "Polyurethanes from vegetable oils." *Polymer Reviews*, 48(1), 109-155.

<https://doi.org/10.1080/15583720701834224>

Culebras, M., et al. (2019). "Lignin-Derived Carbon Materials with Multi-Scale Porosity for High-Performance Supercapacitors." *ACS Applied Energy Materials*, 2(6), 4464-4473.

<https://doi.org/10.1021/acsaem.9b00694>

12. APPENDICES

APPENDIX A: Batch Record Template

Hempoxies Batch Record Sheet

Field	Value
Batch Number	HEMPOXY-P1A-
Date	
Operator	
DoE Run Number	
Target Total Mass (g)	500.00

Component Lot Numbers & Masses:

Component	Lot #	QC Status	Equivalent Weight (g/eq)	Target Mass (g)	Actual Mass (g)
EHSO		☐ Pass	EEW =		

Component	Lot #	QC Status	Equivalent Weight (g/eq)	Target Mass (g)	Actual Mass (g)
MHL		☐ Pass	AldEW =		
HDA		☐ Pass	AHEW =		
FGE		☐ Pass	EEW =	35.00	
HDCNS		☐ Pass	N/A		
HDB		☐ Pass	N/A	25.00	
HDCF		☐ Pass	N/A	15.00	
Total				500.00	

Stoichiometry Calculation:

- **r_target:** _____
- **M_remaining (EHSO+HDA):** _____ g
- **Calculated M_HDA:** _____ g
- **Calculated M_EHSO:** _____ g
- **Calculated by:** _____ Date: _____
- **Verified by:** _____ Date: _____

Processing Log:

- **Sonication Time (HDCNS in FGE):** _____ minutes
- **High-Shear Mixing Time (Fillers):** _____ minutes at _____ rpm
- **Final Mixing Time (with HDA):** _____ minutes at _____ rpm
- **Degassing Time & Vacuum Level:** _____ minutes at _____ mbar
- **Cure Profile:** 80°C / 2h → _____°C / _____h
- **Deviations / Observations:** _____

Sign-offs:

- **Compounder:** _____ Date: _____
- **QC Inspector:** _____ Date: _____

APPENDIX B: Stoichiometry Calculation Spreadsheet

Excel/Google Sheets Template Structure:

INPUTS SECTION (Yellow Cells):

Parameter	Value	Units
-----------	-------	-------

Parameter	Value	Units
M_total	500	g
r_Hardener/ReactiveGroups	1.00	ratio
L_MHL	10.0	% wt.
C_HDCNS	0.25	% wt.
EHSO_OV	8.5	%
FGE_OV	20.0	%
HDA_TAV	200	mg KOH/g
MHL_Aldehyde_Content	3.0	mmol/g

CALCULATIONS SECTION (Grey Cells):

Parameter	Formula	Result	Units
Equivalent Weights			
EEW_EHSO	=100000 / EHSO_OV	11764.7	g/eq
EEW_FGE	=100000 / FGE_OV	5000.0	g/eq
AHEW_HDA	=56100 / HDA_TAV	280.5	g/eq
AldEW_MHL	=1000 / MHL_Aldehyde_Content	333.3	g/eq
Component Masses			
M_FGE	= M_total * 0.07	35.00	g
M_HDB	= M_total * 0.05	25.00	g
M_HDCF	= M_total * 0.03	15.00	g
M_MHL	= M_total * (L_MHL/100)	50.00	g
M_HDCNS	= M_total * (C_HDCNS/100)	1.25	g
M_remaining	= M_total - SUM(Fixed Masses)	373.75	g
Equivalents (Fixed)			
Eq_FGE	= M_FGE / EEW_FGE	0.007	eq
Eq_MHL	= M_MHL / AldEW_MHL	0.150	eq

Parameter	Formula	Result	Units
Solver for M_HDA	= (r*AHEW_HDA* ((M_rem/EEW_EHSO)+Eq_FGE+Eq_MHL)) / (1+(r*AHEW_HDA/EEW_EHSO))	51.72	g
Solver for M_EHSO	=M_remaining - M_HDA	322.03	g

OUTPUT SECTION (Green Cells):

Component	Final Mass (g)	Weight %
EHSO	322.03	64.41%
MHL	50.00	10.00%
HDA	51.72	10.34%
FGE	35.00	7.00%
HDCNS	1.25	0.25%
HDB	25.00	5.00%
HDCF	15.00	3.00%
VERIFICATION SUM	500.00 g	100.00%

APPENDIX C: Testing Schedule Template

Phase 1A Testing Timeline (Per DoE Run)

Day	Activity	Samples Required	Equipment Used
0	Component QC verification & Stoichiometry calculation	All 7 components	FTIR, Titrator, GC, Spreadsheet
1	Compounding, casting, and start of initial cure	5 tensile bars + 3 plaques + 2 DMA bars	High-shear mixer, Sonicator, Molds, Oven
2	Post-cure continues	All samples	Oven
3	Demolding & 24h conditioning	All samples	Desiccator, Calipers
4	Mechanical Testing & start of Water Absorption	5 tensile bars, 3 plaques	UTM, Scale, Water bath
5	Thermal Analysis (DSC & DMA)	1 tensile bar scrap, 1 DMA bar	DSC, DMA

Day	Activity	Samples Required	Equipment Used
6	Stress Relaxation Test & 24h Water Absorption measurement	1 DMA bar, 3 plaques	DMA, Scale
7	Reprocessing Cycle 1 (grind, press, machine)	Failed tensile bars	Hot press, Mill
8	Tensile testing (Cycle 1)	3 reprocessed bars	UTM
9	Reprocessing Cycle 2	Failed cycle 1 bars	Hot press, Mill
10	Tensile testing (Cycle 2)	3 reprocessed bars	UTM
11	Reprocessing Cycle 3	Failed cycle 2 bars	Hot press, Mill
12	Tensile testing (Cycle 3)	3 reprocessed bars	UTM
13	Final data compilation & analysis	All results	Statistical software

- **Total Timeline per Single Run:** ~13 days
- **Estimated Timeline for 20 Runs (with parallel processing):** 8-10 weeks

APPENDIX D: Equipment List & Specifications

Minimum Equipment Required for Phase 1A:

1. Synthesis Equipment:

- **2L Jacketed Reactor:** Glass, with overhead stirrer (50-2000 rpm), reflux condenser, and temperature controller ($\pm 1^\circ\text{C}$).
- **Rotary Evaporator:** Vacuum capability to <1 mbar, with temperature-controlled bath.
- **Tube Furnace:** Max temperature $\geq 1200^\circ\text{C}$, with quartz tube and inert gas (Ar/N_2) mass flow controller.
- **Hydrothermal Autoclave:** 500 mL, stainless steel or Hastelloy, rated to $\geq 300^\circ\text{C}$ and ≥ 10 MPa.
- **Vacuum Oven:** Max temperature $\geq 200^\circ\text{C}$, vacuum capability <1 mbar.

2. Characterization Equipment:

- **FTIR Spectrometer:** Range $4000\text{--}400\text{ cm}^{-1}$, with ATR accessory.
- **Gas Chromatograph (GC):** With FID detector for purity analysis.
- **Karl Fischer Titrator:** For precise moisture content measurement.
- **Brookfield Viscometer:** With multiple spindles for a range of viscosities.
- **Laser Diffraction Particle Size Analyzer:** For HDB and MHL particle size distribution.
- **Thermogravimetric Analyzer (TGA):** Range $25\text{--}1000^\circ\text{C}$, N_2 and air atmosphere.
- **Raman Spectrometer:** 532 nm or 785 nm laser for carbon analysis.
- **TEM/SEM:** For HDCNS morphology and composite fracture surface analysis.
- **BET Surface Area Analyzer:** N_2 adsorption/desorption isotherms.

3. Compounding Equipment:

- **High-Shear Mixer:** Overhead "Cowles" type disperser, ≥ 2000 rpm.
- **Ultrasonic Probe (Sonicator):** ≥ 400 W, 20 kHz, with temperature monitoring.
- **Analytical Balance:** ± 0.001 g precision.
- **Vacuum Degassing Chamber:** Capable of maintaining < 50 mbar.

4. **Testing Equipment:**

- **Universal Testing Machine (UTM):** ≥ 10 kN load cell, with extensometer for accurate modulus measurement.
- **Differential Scanning Calorimeter (DSC):** Temperature range -80 to 300°C .
- **Dynamic Mechanical Analyzer (DMA):** Temperature range -100 to 400°C , with tension and 3-point bend clamps, including stress relaxation mode.
- **Hot Press:** Platen temperature up to 300°C , pressure capability up to 50 MPa.

- **Estimated Capital Equipment Cost:** \$800,000 – \$1,200,000 CAD
- **Annual Operating Cost (Consumables, Maintenance):** \$150,000 – \$200,000 CAD

APPENDIX E: Safety Data Summary

Hazardous Materials Used in Hempoxies Synthesis:

Chemical	Hazard Class	PPE Required	Storage	Disposal
Hydrogen Peroxide (35%)	Oxidizer, Corrosive	Goggles, nitrile gloves, lab coat	Cool, dark, vented cabinet	Dilute to $< 3\%$, then neutralize with sodium bisulfite.
Formic Acid (88%)	Corrosive, Flammable	Face shield, neoprene/butyl gloves	Corrosives cabinet, away from bases	Neutralize with sodium bicarbonate.
Epichlorohydrin	Carcinogen, Flammable, Toxic	Fume hood, double nitrile gloves	Flammables cabinet, secondary containment	Licensed hazwaste contractor only.
Sodium Hydroxide (50%)	Corrosive	Goggles, nitrile gloves, apron	Corrosives cabinet, away from acids	Neutralize with dilute HCl.
Hydrochloric Acid (1M)	Corrosive	Goggles, nitrile gloves	Corrosives cabinet, away from bases	Neutralize with dilute NaOH or NaHCO_3 .
Potassium Hydroxide	Corrosive	Goggles, nitrile gloves, dust mask (solid)	Dry storage, away from moisture/acids	Neutralize with dilute HCl.
DMF (Dimethylformamide)	Toxic, Reproductive Hazard	Fume hood, nitrile gloves	Flammables cabinet	Licensed hazwaste contractor only.

Emergency Contacts:

- **CDCQ Safety Officer:** [Insert Name & Contact]
 - **Quebec Poison Control:** 1-800-463-5060
 - **Fire/Medical Emergency:** 911
 - **Hazmat Cleanup Contractor:** [Insert Contractor Name & Contact]
-

APPENDIX F: Grant Application Support Materials

Suggested Funding Sources for Phase 1A R&D:

1. NSERC Alliance Grants

- **Program:** Supports collaborative R&D projects between university researchers and industry partners.
- **Funding:** Up to \$1,000,000 per year.
- **Fit:** Excellent for partnering Landry Industries with a Quebec university to leverage CDCQ facilities. The project's high degree of scientific novelty and clear commercial goals align perfectly.

2. NRC-IRAP (Industrial Research Assistance Program)

- **Program:** Provides financial assistance and advisory services to Canadian SMEs to develop and commercialize technologies.
- **Funding:** Covers a significant portion of R&D salary and contractor costs.
- **Fit:** Ideal for the SME-led (Landry Industries) nature of this project. Funds can support hiring technicians and paying for services at CDCQ.

3. Sustainable Development Technology Canada (SDTC) - Seed Fund

- **Program:** Supports early-stage Canadian cleantech entrepreneurs.
- **Funding:** Up to \$100,000.
- **Fit:** Perfect for validating the core technology. The carbon-negative and recyclability claims are central to SDTC's mandate of reducing GHG emissions and improving environmental sustainability.

4. Quebec Research Fund (FRQNT) - Partnership Research Projects

- **Program:** Fosters collaboration between Quebec-based academic researchers and public/private sector partners.
- **Funding:** Varies, typically in the range of \$50,000-\$200,000 per year.
- **Fit:** Strong, as the project is targeted for a Quebec facility (CDCQ) and leverages a key provincial resource (hemp). Emphasizes local innovation and supply chains.

Recommended Phased Application Strategy:

- **Year 1 (Phase 1A - This Document):** Secure initial R&D funding through a combined **NRC-IRAP** (for Landry Industries) and **NSERC Alliance** (for a university partner) application. Use the **SDTC Seed Fund** to cover initial validation and prototyping costs.

- **Year 2 (Phase 1B - Scale-up & Demonstration):** Following successful Phase 1A results, apply for a larger **SDTC Ecosystem Fund** project to demonstrate the technology at a pilot scale (100 kg batches) and produce prototype parts for target industries (aerospace, automotive).
- **Year 3+ (Phase 2 - Commercialization):** Leverage validated technology to secure strategic investment and partnerships with OEMs, supported by further federal and provincial business growth programs.