

Hempoxies HERA-75

Hemp Epoxy Reinforced Adaptive Vitrimer Architecture

Experimental Research and Validation Specification

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Research status: Experimental proposal / pre-validation.
This document defines a testable materials-research program. It does not constitute evidence that the proposed formulation will achieve any stated performance target. All performance claims must be established from experimental measurements, statistical analysis, and independent replication.

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1 Abstract

Hempoxies HERA-75 is proposed as a next-generation bio-based vitrimer architecture derived from the Hempoxies research program initiated by Marie-Soleil Seshat Landry.

The principal design change is the separation of four previously overlapping materials functions:

1. renewable flexible epoxy functionality;
2. rigid bio-aromatic structural functionality;
3. dynamic covalent network formation;
4. hierarchical hemp-derived reinforcement.

The proposed matrix combines epoxidized hempseed oil (EHSO) with a rigid multifunctional bio-aromatic epoxy, using a controlled succinic-acid/citric-acid curing system and zinc acetylacetonate $[\text{Zn}(\text{acac})_2]$ as the principal transesterification catalyst.

Furfuryl glycidyl ether (FGE) is retained only as an optional low-level reactive diluent and processing modifier.

The reinforcement architecture consists of:

- aligned hemp-derived carbon fiber as the primary macroscopic load-transfer phase;
- hemp cellulose nanocrystals (CNC) as an interfacial/nanoscale reinforcement;
- hemp-derived biochar as a microstructural modifier;
- oxidized hemp-derived carbon nanosheets as a secondary nanoscale functional phase.

The research program is explicitly staged. The neat matrix must first satisfy thermal, mechanical, chemical, and vitrimer-network criteria before reinforcement is introduced.

The central hypothesis is:

EHSO flexibility + bio-aromatic rigidity
+dynamic ester network + hierarchical hemp reinforcement

may provide a superior balance of renewable content, mechanical performance, thermal stability, repairability, and reprocessability relative to an EHSO-dominant vitrimer matrix.

2 Research Questions

The program shall answer the following questions experimentally.

1. Can an EHSO/bio-aromatic epoxy matrix achieve substantially higher glass-transition temperature than an EHSO-only matrix?
2. Can a controlled carboxyl-to-epoxide equivalent ratio produce a reproducible vitrimer network?
3. What $\text{Zn}(\text{acac})_2$ concentration provides the optimal compromise between mechanical properties and dynamic stress relaxation?
4. Does replacing most FGE structural functionality with a multifunctional bio-aromatic epoxy increase crosslink density and thermal stability?

5. Can hierarchical hemp reinforcement increase modulus, strength, fracture resistance, and dimensional stability without destroying vitrimer reprocessability?
6. What performance is retained after thermal reprocessing?
7. What environmental and mechanical penalties arise from converting hemp biomass into carbon reinforcement?

3 Central Hypothesis

The central hypothesis is that the principal limitation of an EHSO-dominant vitrimer is not the absence of additional filler but the intrinsic flexibility of the triglyceride-derived molecular architecture.

Therefore:

Matrix architecture must be optimized before filler optimization.

HERA-75 consequently uses a two-phase epoxy architecture:

$$E_{\text{total}} = E_{\text{EHSO}} + E_{\text{BAE}} + E_{\text{FGE}}$$

where:

$$E_{\text{EHSO}} = \text{epoxy equivalents from EHSO}, \quad (1)$$

$$E_{\text{BAE}} = \text{epoxy equivalents from bio-aromatic epoxy}, \quad (2)$$

$$E_{\text{FGE}} = \text{epoxy equivalents from FGE}. \quad (3)$$

The primary experimental variable is therefore epoxy-equivalent composition, not simply mass fraction.

4 Critical Stoichiometric Correction

Previous Hempoxies formulations contained a potentially consequential equivalent-counting error involving FGE.

Furfuryl glycidyl ether has an approximate molecular mass and epoxy equivalent weight of:

$$EEW_{\text{FGE}} \approx 154.16 \text{ g eq}^{-1}$$

Therefore:

$$n_{\text{FGE}} = \frac{m_{\text{FGE}}}{154.16}$$

For example, 450 g FGE corresponds to:

$$n_{\text{FGE}} = \frac{450}{154.16} = 2.919 \text{ eq}$$

and not approximately 1.50 eq.

All HERA-75 formulations shall therefore be calculated exclusively using functional equivalents.

No formulation shall be accepted based solely on mass ratios.

5 Design Philosophy

HERA-75 assigns different functions to different molecular or structural components.

Table 1: Functional architecture	
Component	Primary function
EHSO	Renewable flexible epoxy phase
Bio-aromatic epoxy	Rigidity, Tg, dimensional stability, network density
FGE	Optional viscosity/reactive-processing modifier
Succinic acid	Primary difunctional carboxylic curing component
Citric acid	Multifunctional network-forming component
Zn(acac) ₂	Transesterification catalyst
Hemp carbon fiber	Macroscopic structural load transfer
Hemp CNC	Nanoscale reinforcement/interface modification
Hemp biochar	Microstructural reinforcement/toughening
Oxidized hemp carbon nanosheets	Nano-interface and functional reinforcement

6 Target Matrix Composition

The preferred starting matrix is defined on an epoxy-equivalent basis.

6.1 Epoxy Equivalent Distribution

The initial screening composition shall be:

$$E_{\text{EHSO}} = 65\% \quad (4)$$

$$E_{\text{BAE}} = 30\% \quad (5)$$

$$E_{\text{FGE}} = 5\% \quad (6)$$

with:

$$E_{\text{total}} = E_{\text{EHSO}} + E_{\text{BAE}} + E_{\text{FGE}}$$

The 65:30:5 composition is a starting hypothesis, not a fixed final formulation. A composition sweep shall subsequently test:

Formulation	E_{EHSO}	E_{BAE}	E_{FGE}
<i>H0</i>	100	0	0
<i>H1</i>	80	20	0
<i>H2</i>	70	30	0
<i>H3</i>	65	30	5
<i>H4</i>	60	35	5
<i>H5</i>	55	40	5

All values represent percentage of total epoxy equivalents.

7 Carboxyl-to-Epoxy Stoichiometry

The primary target shall be:

$$R_{\text{COOH/Epoxy}} = 0.80$$

with an initial screening range:

$$0.70 \leq R_{\text{COOH/Epoxy}} \leq 0.90$$

The total carboxyl equivalent requirement is:

$$N_{\text{COOH}} = R_{\text{COOH/Epoxy}} N_{\text{epoxy}}$$

For a target ratio of 0.80:

$$N_{\text{COOH}} = 0.80 N_{\text{epoxy}}$$

The formulation must be calculated from measured or manufacturer-certified acid values and epoxy equivalent weights.

Nominal molecular weights shall never substitute for measured functionality when a supplier provides an experimentally determined EEW or acid value.

8 Curative Composition

The initial acid-equivalent distribution shall be:

$$SA_{\text{COOH}} = 70\% \tag{7}$$

$$CA_{\text{COOH}} = 30\% \tag{8}$$

where:

$$SA = \text{succinic acid} \tag{9}$$

$$CA = \text{citric acid.} \tag{10}$$

The total acid equivalents are therefore:

$$N_{\text{SA}} + N_{\text{CA}} = N_{\text{COOH}}$$

and:

$$N_{\text{SA}} = 0.70 N_{\text{COOH}}$$

$$N_{\text{CA}} = 0.30 N_{\text{COOH}}$$

A secondary screening matrix shall test:

Curative	SA	CA	Target ratio
C1	100%	0%	0.80
C2	80%	20%	0.80
C3	70%	30%	0.80
C4	60%	40%	0.80
C5	50%	50%	0.80

9 Catalyst Screening

Zn(acac)₂ shall be screened at:

0.25, 0.50, 1.00, 2.50 wt%

relative to total epoxy matrix mass.

The catalyst shall not be assumed to be optimal at any single concentration without kinetic and rheological evidence.

Primary catalyst-selection criteria are:

1. acceptable cure conversion;
2. adequate Tg;
3. high modulus;
4. measurable dynamic exchange;
5. acceptable stress-relaxation rate;
6. minimal catalyst-induced degradation;
7. reproducible reprocessing.

10 Reinforcement Architecture

The hierarchical reinforcement system shall be introduced only after matrix validation.

10.1 Macro-Reinforcement

Aligned hemp-derived carbon fiber shall provide the principal structural load path.

The initial fiber-volume-fraction sweep shall be:

$V_f = 10, 20, 30, 40\%$

subject to processing feasibility and void-content control.

Fiber orientation shall be explicitly documented.

Random short-fiber composites shall not be used as substitutes for continuous aligned reinforcement when evaluating structural properties.

10.2 Hemp CNC

Initial concentration:

0.50–1.00 wt%

A screening series shall include:

0, 0.25, 0.50, 1.00, 2.00 wt%

with respect to matrix mass.

10.3 Hemp Biochar

Initial concentration:

1.00–2.00 wt%

Screen:

0, 0.5, 1.0, 2.0, 4.0 wt%

10.4 Hemp-Derived Carbon Nanosheets

Initial concentration:

0.25–0.75 wt%

The material shall be characterized before being described as graphene, graphene oxide, or another formally defined carbon allotrope.

Required characterization includes, where available:

- Raman spectroscopy;
- XPS;
- SEM/TEM;
- elemental analysis;
- surface-area measurement;
- ash content;
- particle-size distribution.

11 Experimental Series

The experimental program shall proceed sequentially.

11.1 Phase I: Matrix

1. EHSO-only control.
2. EHSO + bio-aromatic epoxy.
3. EHSO + bio-aromatic epoxy + FGE.
4. Curative-ratio optimization.
5. Catalyst optimization.

11.2 Phase II: Fiber

The optimized matrix shall be combined with aligned hemp-derived carbon fiber.

11.3 Phase III: Hierarchical Nano/Micro Reinforcement

The optimized fiber composite shall receive controlled CNC, biochar, and carbon-nanosheet additions.

11.4 Phase IV: Reprocessing

Reprocessing shall be conducted only after the original network has been characterized.

12 Control Groups

At minimum, the following controls shall be fabricated.

ID	Control
M0	EHSO + curing system, no bio-aromatic epoxy
M1	EHSO + bio-aromatic epoxy, no FGE
M2	EHSO + bio-aromatic epoxy + FGE
M3	Optimized matrix without reinforcement
F1	Optimized matrix + aligned hemp carbon fiber
N1	Matrix + CNC
N2	Matrix + biochar
N3	Matrix + carbon nanosheets
H1	Matrix + complete hierarchical reinforcement
CFRP	Conventional epoxy/carbon-fiber benchmark, where available

The CFRP benchmark shall be treated strictly as a comparative engineering reference and not as a predetermined target.

13 Processing Protocol

Exact processing temperatures and durations shall be established by differential scanning calorimetry (DSC), rheology, and preliminary small-batch curing trials rather than assumed from unrelated formulations.

The generic sequence is:

1. Dry hygroscopic reinforcement under controlled conditions.
2. Determine moisture content.
3. Measure EHSO epoxy value/EEW.
4. Measure bio-aromatic epoxy EEW.
5. Verify FGE identity and purity.
6. Calculate all epoxy equivalents.
7. Calculate required acid equivalents.
8. Prepare acid/catalyst phase.
9. Combine epoxy phases under controlled temperature.
10. Introduce FGE only if required for processability.
11. Disperse nano/micro reinforcement under controlled shear.
12. Degas under controlled vacuum.
13. Impregnate aligned carbon fiber where applicable.

14. Cure using a DSC/rheology-informed temperature schedule.
15. Post-cure only if experimentally justified.
16. Condition specimens under standardized laboratory conditions.

No formulation shall be scaled from a small batch to a large batch without recalculating heat generation, viscosity, mixing efficiency, and cure kinetics.

14 Chemical Characterization

Each matrix shall undergo:

- FTIR;
- epoxy conversion analysis;
- acid-value determination;
- DSC;
- TGA;
- DMA;
- gel-fraction measurement;
- swelling analysis;
- rheology;
- stress-relaxation testing.

The conversion of epoxy groups shall be monitored where analytically practical.

The reaction should not be considered complete merely because a specimen appears visually solid.

15 Thermal Characterization

Required parameters include:

$$T_g = \text{glass-transition temperature} \quad (11)$$

$$T_{5\%} = \text{temperature at 5\% mass loss} \quad (12)$$

$$T_{10\%} = \text{temperature at 10\% mass loss} \quad (13)$$

$$T_{\text{char}} = \text{residual char fraction.} \quad (14)$$

The initial matrix-development target is:

$$T_g \geq 90^\circ\text{C}$$

This is a development criterion, not a guaranteed outcome.

A secondary objective is:

$$T_{5\%} \geq 250^\circ\text{C}$$

where experimentally achievable.

16 Mechanical Characterization

At minimum:

- tensile strength;
- tensile modulus;
- elongation at break;
- flexural strength;
- flexural modulus;
- interlaminar shear strength;
- fracture toughness;
- impact resistance;
- fatigue;
- compression;
- hardness.

All reported mechanical values shall include:

- specimen geometry;
- test standard;
- strain rate;
- conditioning;
- number of specimens;
- mean;
- standard deviation;
- coefficient of variation;
- individual specimen values where possible.

No single best specimen shall be reported as representative performance.

17 Vitrimer Characterization

Dynamic network behavior shall be demonstrated experimentally.

Stress relaxation shall be measured at multiple temperatures.

The normalized relaxation function is:

$$\phi(t) = \frac{\sigma(t)}{\sigma_0}$$

where:

$$\sigma_0 = \text{initial stress} \quad (15)$$

$$\sigma(t) = \text{stress remaining after time } t. \quad (16)$$

The characteristic relaxation time τ shall be defined as:

$$\phi(\tau) = e^{-1}$$

where experimentally appropriate.

Temperature-dependent relaxation shall be analyzed using an Arrhenius relationship:

$$\ln\left(\frac{1}{\tau}\right) = \ln A - \frac{E_a}{RT}$$

where:

$$E_a = \text{apparent activation energy} \quad (17)$$

$$R = \text{gas constant} \quad (18)$$

$$T = \text{absolute temperature.} \quad (19)$$

If Arrhenius behavior is not adequate across the measured temperature range, the alternative kinetic model shall be explicitly reported rather than forced onto the data.

18 Reprocessing Experiment

Reprocessing shall use identical processing conditions for each cycle.

The following sequence shall be performed:

$$P_0 \rightarrow P_1 \rightarrow P_2 \rightarrow P_3$$

where:

$$P_0 = \text{original specimen} \quad (20)$$

$$P_1 = \text{first reprocessed specimen} \quad (21)$$

$$P_2 = \text{second reprocessed specimen} \quad (22)$$

$$P_3 = \text{third reprocessed specimen.} \quad (23)$$

Strength retention shall be calculated as:

$$R_n = 100 \left(\frac{S_n}{S_0} \right)$$

where:

$$S_0 = \text{original strength} \quad (24)$$

$$S_n = \text{strength after } n \text{ reprocessing cycles.} \quad (25)$$

An initial development criterion is:

$$R_3 \geq 80\%$$

but this shall not be interpreted as proof of indefinite recyclability.

19 Moisture and Environmental Conditioning

Because hemp-derived materials can be hygroscopic, all composites shall be tested under at least three conditions:

1. dry conditioned;
2. standard laboratory conditioned;
3. elevated-humidity conditioned.

Mass uptake shall be calculated:

$$M_t = 100 \frac{m_t - m_0}{m_0}$$

where:

$$m_0 = \text{initial dry mass} \tag{26}$$

$$m_t = \text{mass at time } t. \tag{27}$$

Mechanical properties shall be measured after conditioning.

20 Failure Criteria

HERA-75 shall be considered unsuccessful as a structural material candidate if any of the following occur:

1. matrix cannot be reproduced with acceptable batch-to-batch variation;
2. epoxy conversion remains inadequate;
3. severe phase separation occurs;
4. Tg decreases rather than increases relative to the EHSO control;
5. reinforcement produces statistically insignificant improvement;
6. moisture uptake causes unacceptable mechanical degradation;
7. reprocessing destroys the network;
8. catalyst concentration required for dynamic behavior produces unacceptable mechanical or thermal degradation.

A failed hypothesis is a valid experimental result.

21 Success Criteria

The following are development targets rather than guaranteed properties.

Table 3: Initial HERA-75 development targets

Property	Target
Matrix T_g	$\geq 90^\circ\text{C}$
TGA $T_{5\%}$	$\geq 250^\circ\text{C}$
Matrix tensile strength	$\geq 50\text{ MPa}$
Matrix tensile modulus	$\geq 2.5\text{ GPa}$
Reprocessing strength retention	$\geq 80\%$ after 3 cycles
Reproducibility	CV $\leq 15\%$ preferred
Stress relaxation	experimentally measurable
Fiber composite strength	determined experimentally
Fracture toughness	statistically improved vs. matrix control
Moisture degradation	quantified and minimized

These criteria shall be revised after the first statistically valid experimental campaign.

22 Statistical Design

The experimental program shall avoid optimization from single specimens.

For primary mechanical testing:

$$n \geq 5$$

is recommended as a minimum exploratory sample size, with larger n required for definitive comparisons.

Results shall be reported as:

$$\bar{x} \pm s$$

where:

$$\bar{x} = \text{sample mean} \tag{28}$$

$$s = \text{sample standard deviation.} \tag{29}$$

Where multiple formulations are compared, analysis of variance (ANOVA) or another appropriate statistical model shall be used.

Effect sizes and confidence intervals should accompany p -values.

23 Material Identity and Traceability

Every experimental batch shall receive a unique identifier.

Recommended structure:

$$\text{HERA75-YYYYMMDD-MXX-CXX-RXX}$$

where:

- YYYYMMDD = fabrication date;

- MXX = matrix formulation;
- CXX = catalyst/curative condition;
- RXX = reinforcement condition.

Each batch record shall contain:

- supplier;
- product identification;
- lot number;
- purity;
- EEW or acid value;
- moisture content;
- exact masses;
- calculated equivalents;
- processing temperatures;
- processing times;
- mixing conditions;
- cure schedule;
- specimen preparation method.

24 Data Integrity

Raw measurements shall be retained.

The following shall never be replaced by rounded summary values:

- raw force-displacement curves;
- raw DSC traces;
- raw TGA traces;
- raw DMA curves;
- raw FTIR spectra;
- raw rheology data;
- specimen dimensions;
- individual failure loads.

The final dataset shall distinguish between:

Measured $\neq$ Calculated $\neq$ Modeled $\neq$ Projected
--

This distinction is mandatory.

25 Environmental Assessment

Environmental claims shall not be inferred solely from the word “bio-based.”

A future life-cycle assessment shall quantify:

- agricultural inputs;
- oil extraction/processing;
- epoxidation;
- bio-aromatic precursor synthesis;
- acid production;
- catalyst production;
- carbonization energy;
- fiber conversion;
- transportation;
- processing energy;
- reprocessing;
- end-of-life.

The carbon balance shall be expressed as:

$$GWP_{\text{net}} = GWP_{\text{feedstock}} + GWP_{\text{processing}} + GWP_{\text{transport}} + GWP_{\text{end-of-life}} - C_{\text{biogenic}}$$

with system boundaries explicitly stated.

No “carbon negative” designation shall be used without a completed life-cycle assessment.

26 Scientific Decision Tree

The program shall follow this decision sequence:

Validate chemistry → Validate network → Validate mechanics
 → Add reinforcement → Validate interface
 → Validate reprocessing → Scale

If the neat matrix fails, reinforcement optimization shall stop.

If the reinforced material fails, additional filler combinations shall not be added merely to rescue the formulation.

27 Predicted Failure Modes

The principal anticipated failure modes are:

1. insufficient crosslink density;
2. excessive flexible EHSO phase;
3. excessive FGE dilution;
4. incomplete epoxy conversion;
5. catalyst-induced degradation;
6. phase separation;
7. poor carbon-fiber/matrix adhesion;
8. moisture-induced interfacial failure;
9. nano-filler agglomeration;
10. excessive viscosity;
11. void formation;
12. thermal degradation during processing;
13. insufficient dynamic exchange;
14. excessive dynamic exchange causing poor dimensional stability.

Each failure mode shall have a corresponding diagnostic measurement.

28 Primary Experimental Matrix

ID	EHSO	BAE	FGE	Purpose
H0	100%	0%	0%	EHSO control
H1	80%	20%	0%	Low aromatic fraction
H2	70%	30%	0%	Intermediate aromatic fraction
H3	65%	30%	5%	Primary HERA-75 candidate
H4	60%	35%	5%	Higher rigidity
H5	55%	40%	5%	Maximum initial aromatic fraction

All percentages refer to epoxy equivalents.

29 Primary HERA-75 Formulation

The initial candidate is therefore defined as:

$$\begin{aligned}
 E_{\text{EHSO}} &= 65\% \\
 E_{\text{BAE}} &= 30\% \\
 E_{\text{FGE}} &= 5\% \\
 R_{\text{COOH/Epoxy}} &= 0.80 \\
 SA_{\text{COOH}} &= 70\% \\
 CA_{\text{COOH}} &= 30\% \\
 \text{Zn}(\text{acac})_2 &= 0.50\text{--}1.00 \text{ wt}\%
 \end{aligned}$$

The reinforcement-free HERA-75 matrix shall be considered the primary chemical candidate. The hierarchical composite candidate is:

$$\text{HERA-75-M} + \text{aligned HDCF} + \text{CNC} + \text{biochar} + \text{oxidized HDCNS}$$

with all reinforcement concentrations experimentally optimized rather than assumed.

30 Required Analytical Evidence Before Claims

The following evidence shall be required before making each corresponding claim.

Table 5: Claim-evidence requirements

Claim	Minimum evidence
Bio-based	Feedstock provenance and compositional accounting
Vitrimer	Dynamic stress relaxation and network chemistry evidence
Reprocessable	Multiple successful reprocessing cycles with retained properties
High-Tg	DSC/DMA measurement
High-strength	Standardized tensile testing with replicates
Structural composite	Fiber-volume fraction and mechanical characterization
Graphene-containing	Appropriate structural characterization
Carbon-negative	Life-cycle assessment
Recyclable	Defined recycling pathway and retained properties
Scalable	Process-window and scale-up demonstration

31 Expected Scientific Contribution

If successful, HERA-75 would establish a testable materials platform rather than a single formulation.

The scientific contribution would be the investigation of whether a hemp-derived epoxidized-oil vitrimer can be upgraded through deliberate molecular-scale rigidity and hierarchical reinforcement while preserving dynamic-network behavior.

The key comparison is therefore not:

Hempoxies vs. all conventional materials

but:

EHSO vitrimer $\rightarrow$ EHSO/bio-aromatic vitrimer $\rightarrow$ hierarchical hemp composite

This progression permits attribution of performance changes to specific design decisions.

32 Research Integrity Statement

Hempoxies HERA-75 is a proposed experimental architecture authored by Marie-Soleil Seshat Landry.

The formulation shall not be represented as experimentally validated until independent or appropriately documented experimental measurements establish the stated properties.

Reported historical Hempoxies measurements shall be clearly distinguished from literature-derived values, calculated predictions, simulations, engineering targets, and newly measured HERA-75 results.

No numerical performance claim shall be promoted from “target” to “validated” without traceable raw data.

33 Conclusion

Hempoxies HERA-75 represents a deliberate transition from formulation accumulation toward experimentally falsifiable materials engineering.

Its architecture is:

EHSO + Rigid Bio-Aromatic Epoxy
+SA/CA Dynamic Network + Zn(acac)₂
+Hierarchical Hemp Carbon

The central experimental proposition is that the combination of molecular rigidity, controlled dynamic-network stoichiometry, and hierarchical reinforcement can overcome limitations associated with an EHSO-dominant vitrimer without abandoning the renewable hemp-based platform.

The first objective is therefore not to obtain the largest possible strength number.

The first objective is to establish a reproducible causal relationship between:

composition $\rightarrow$ network structure $\rightarrow$ processing $\rightarrow$ microstructure $\rightarrow$ properties

Only after that relationship is experimentally demonstrated should HERA-75 be optimized for structural applications.

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