

Feasibility Study of Hemp-Derived Carbon Nanosheet Reinforcement in Epoxy and Bio-Based Vitrimer Composites

Foundational Experimental Protocol

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AI-assisted review and editorial amendments are documented in the notice and audit record on page ii. The original v1.5 technical text is retained except where a v1.6 amendment explicitly controls.

AI USE NOTICE AND CONTRIBUTION AUDIT

Purpose and responsibility. AI systems were used as review and editorial aids to critique the draft protocol, identify internal inconsistencies, and suggest refinements. They are not authors or co-authors. Marie-Soleil Seshat Landry is solely responsible for the protocol, its interpretation, all decisions, and the final text.

Scope of AI assistance. The systems reviewed parts or all of the draft to varying extents. No AI-generated experimental data, measurements, or results are included. All performance figures and thresholds are design targets or hypotheses unless independently established by cited evidence. AI output is not a substitute for primary-source verification, laboratory risk assessment, or statistical review.

Systems and review record

System	Use / observations	Limitations recorded
DeepSeek	Full-document review; checked M2–M4 arithmetic; identified mismatch between the 5% success threshold and n=5/group; corrected the claim that §19 was absent.	Review is advisory; conclusions require independent verification.
Kimi	Produced M1–M9 enhancement pack; reviewed citations [4] and FGE, novelty wording, and loading-feasibility gate.	Review was truncated at §14.2; M1 incorrectly characterized §19 as absent; M4 power estimate was best-case for two-way ANOVA and optimistic for Tukey comparisons.
Claude	Summary/risk review covering baseline/comparator logic, Group E, and vitrimer criteria.	Did not review the full back half of the document.

Verification and transparency. Record exact model names/versions and dates in the project log where available; do not invent unavailable model identifiers. Any standards editions, supplier claims, or literature citations marked “verified October 2026” must be checked against primary sources before protocol freeze. AI-assisted review does not establish novelty, regulatory compliance, or scientific validity.

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READINESS STATEMENT

This protocol is at Technology Readiness Level 1–2. It sets out hypotheses, methods and decision criteria; it contains no experimental results. Every threshold and benefit stated below is a design target or a hypothesis to be tested, not a validated performance figure. Version 1.6 adds binding clarifications and amendments; the original v1.5 text remains in the record where not expressly superseded.

Abstract

This protocol establishes what is, to the author's knowledge, the first systematic feasibility study evaluating hemp-derived carbon nanosheets as reinforcing agents in polymer composite matrices. The study investigates the effects of direct epoxy impregnation of hemp carbon nanosheets prior to their incorporation into conventional thermoset epoxy systems and bio-based epoxy vitrimer systems.

The central hypothesis is that pre-impregnation of porous hemp-derived carbon nanosheets may improve filler dispersion, matrix wetting, interfacial bonding and overall composite performance relative to untreated nanosheets. A comparison group of conventionally carbonized hemp char from the same feedstock isolates the contribution of the nanosheet form, and every gain is referenced to a neat-matrix baseline checked against published resin data.

This work is designed as the foundational experimental validation of the broader concept of using hemp-derived carbon nanosheets as reinforcement phases within engineered composite materials. A second phase extends the same comparison to other thermosets, thermoplastics, elastomers, ceramics, metals and cementitious binders, each tested against its own neat counterpart processed identically and with a dispersant suited to that matrix. Loadings are swept from dilute to the maximum the matrix can hold, and interphase agents are screened at the best dilute and at a high loading. Optional dispersion aids, ethanol or the reactive diluent furfuryl glycidyl ether (FGE), are screened in a pilot before the main study.

Keywords: hemp-derived carbon nanosheets; epoxy; vitrimer; resin pre-impregnation; hemp bio-epoxy; nanocomposite reinforcement; interfacial load transfer

1 BACKGROUND AND RATIONALE

1.1 Plant-derived plastics: a historical precedent

Turning crops into structural plastics has an industrial precedent. On 13 August 1941, at the Dearborn Days celebration, Henry Ford unveiled an experimental car with plant-based plastic body panels [1] mounted on a tubular steel frame; a patent on the body construction was granted on 13 January 1942 [2]. The panels' recipe is uncertain. Contemporary press accounts said the plastic contained hemp, wheat and flax as well as soybean, whereas project engineer Lowell Overly later recalled soybean fibre in a phenolic resin, and The Henry Ford holds no record of the formula [1]. The project was abandoned when the United States entered the Second World War [3]. The present work pursues the same aim with hemp supplying the reinforcement and, in System B, potentially the matrix.

1.2 Hemp-derived carbon nanosheets

Wang *et al.*, working in David Mitlin's group at the University of Alberta, converted hemp bast fibre into interconnected, partially graphitic carbon nanosheets by hydrothermal carbonization followed by activation [4]. The reported nanosheets were 10–30 nm thick, with specific surface areas of up to 2287 m²/g, a mesopore volume fraction of up to 58 %, and electrical conductivity of 211–226 S/m [4]. That study targeted ionic-liquid supercapacitor electrodes rather than structural reinforcement, and the nanosheet material itself is the subject of a University of Alberta patent [5]. In coverage dated August 2014, Mitlin, by then at Clarkson University, was reported presenting the work at the 248th National Meeting of the American Chemical Society, with a Canadian start-up working to scale it up [6].

1.3 Vitrimers

Vitrimers are permanently cross-linked polymer networks whose covalent bonds exchange associatively, so that network connectivity is conserved while the material can still flow and be reshaped when heated. The class was introduced by Montarnal *et al.* using epoxy-based networks with a transesterification catalyst [7]. As reviewed by Denissen *et al.*, the defining behaviour is a gradual fall in viscosity on heating, a feature previously associated with vitreous silica rather than organic polymers [8].

Carbon fillers from waste and biomass streams have already been placed in epoxy vitrimers. Activated carbon from sugarcane bagasse lowered the self-healing condition of a disulfide-exchange epoxy vitrimer from 80 °C to 70 °C for a 5 min treatment [9], and carbon nanosheets derived from waste tyres raised the storage modulus of a transesterification thiol-epoxy vitrimer roughly threefold at 15 wt% while retaining self-healing and shape-memory behaviour [10].

1.4 Bio-epoxies and vitrimers from hemp

Hemp also supplies matrix chemistry. Epoxidized hemp oil has been blended into epoxy bioresins at concentrations below 30 % and reinforced with jute fibre; it performed slightly better than epoxidized soybean oil, although the bisphenol-A epoxy control remained the strongest and stiffest [11]. Hempseed-oil networks have since been made dynamic. A dual dynamic network vitrimer built from hempseed oil and limonene derivatives and reinforced with hemp fibre can be recycled by aminolysis at 100 °C for 3 h, and adding amino silane raised its impact strength from 3.5 to 10.3 kJ/m² [12]. An epoxidized hempseed oil vitrimer cross-linked with vanillin-based imines has also been presented [13]. These are candidate chemistries for System B.

1.5 Existing data for hemp-derived carbon in polymers

Hemp-derived carbon has been tested in polymers, but only as chars and milled particles (Table 1). The largest strength gain, reported by Zecchi *et al.*, was measured against a neat epoxy of only 10.2 MPa; West System lists its unfilled 105 resin with 205 hardener at about 54 MPa [14], well above the best composite in that study. This is why the protocol anchors every gain to a verified baseline (Section 16). None of these studies used nanosheets, resin pre-impregnation or a vitrimer matrix.

Table 1. Published data for hemp-derived carbon fillers in polymer matrices. Percentages are relative to each study’s own neat polymer.

Study	Hemp carbon filler	Matrix	Loading	Reported effect
Bartoli <i>et al.</i> 2022 [15]	Fibre biochar, pyrolysed at 1000 °C	DGEBA epoxy, amine-cured	2.5–10 wt%	Storage modulus at 40 °C about 1.40 to 2.31 GPa at 10 wt%; wear –80 % and friction –21 % at 2.5 wt%; about 6 mS/m at 10 wt%. No tensile or flexural strength reported.
Zecchi <i>et al.</i> 2024 [16]	Fibre char (400 °C) and hurd char (550 °C), 3:1 hurd:fibre	DGEBA epoxy, amine-cured	1–10 wt%	Tensile strength 10.2 to 19.8 MPa and modulus 0.5 to 1.3 GPa at 10 wt%.
Brachi <i>et al.</i> 2026 [17]	Hurd torrefied at 220–300 °C	PLA	2.5 wt% and 5 wt%	At 5 wt% (300 °C): stiffness +12 %, toughness +27 %, friction –83 %, wear about three orders of magnitude lower; elongation at break reduced.
Kander 2019, US patent [18]	Carbonized at ≥ 1100 °C, milled so 95 % of particles are under 10 µm	PLA, ABS, PET, PP	1–50 wt%	Patent discloses higher stiffness and electrical percolation at about 10 wt% and above.

1.6 Resin pre-impregnation of porous fillers

Introducing resin into porous fillers by vacuum or high pressure before curing is an established route to micro-mechanical interlocking in dental composites [19]; this protocol applies that principle to hemp-derived carbon nanosheets. Processing can work against it: grinding destroyed the original pore structure of a maple biochar filler [20], so nanosheet porosity is re-measured after any milling or sonication step (Table 2).

1.7 Novelty and priority

Hemp-derived carbon nanosheets have, to the author’s knowledge, not yet been evaluated as a reinforcement phase in either conventional epoxies or vitrimers; a web-based prior-art search on 27 October 2026 found no such study. The Principal Investigator proposed hemp-derived carbon nanosheets as a composite building block by April 2025 [21] and later set out their redeployment from energy-storage electrode to structural filler, with the tentative designation *Mitlinite* for the nanosheet morphology [22]. The present protocol is designed as the first experimental test of that proposal. The high mesoporosity reported in [4] motivates the central question of whether resin can be driven into the nanosheet pore network before compounding, and whether doing so improves interfacial load transfer. A hemp-char comparison group (Group E) tests whether the nanosheet form, rather than hemp-derived carbon in general, is responsible for any reinforcement.

2 RESEARCH OBJECTIVES

Primary objective

Determine whether epoxy-impregnated hemp-derived carbon nanosheets provide measurable improvements in composite properties compared with untreated nanosheets.

Secondary objectives

- Quantify epoxy uptake by hemp carbon nanosheets.
- Evaluate nanosheet–matrix compatibility.
- Determine the feasibility of nanosheet reinforcement in bio-based epoxy vitrimer systems.
- Generate baseline mechanical and physical performance data.
- Establish processing methods for future scale-up efforts.
- Benchmark the nanosheets against conventionally carbonized hemp char from the same feedstock.
- Select a dispersion route (direct, ethanol-assisted or FGE) in a pilot study.
- Map properties across the full loading range and locate the maximum loading.
- Screen interphase agents that bond or couple the filler to the matrix.
- Extend the comparison to other matrix classes, each against its neat counterpart (Phase 2).

3 RESEARCH HYPOTHESES

H1 Pre-impregnation of hemp-derived carbon nanosheets with epoxy resin will improve dispersion and matrix adhesion, resulting in improved mechanical properties compared with untreated nanosheets.

H2 Epoxy-impregnated hemp nanosheets will exhibit enhanced compatibility with bio-based epoxy vitrimer systems while maintaining vitrimer functionality.

H3 The porous structure of hemp-derived carbon nanosheets will permit measurable resin uptake that contributes to mechanical reinforcement through improved interfacial load transfer.

H4 At equal loading, hemp-derived carbon nanosheets will provide greater reinforcement than conventionally carbonized and milled hemp char from the same feedstock, showing that the nanosheet morphology itself contributes to the effect.

H5 At equal loading, an interphase agent that bonds to both filler and matrix will raise flexural strength above that of the as-made filler.

For statistical testing, each hypothesis is evaluated against the null hypothesis of no difference between the compared groups (Section 16).

4 MATERIALS

4.1 Filler: hemp carbon nanosheets

Preparation route

1. Hemp biomass feedstock (bast fibre in the reference route [4]).
2. Carbonization.
3. Nanosheet production process (reference route: hydrothermal carbonization followed by activation [4]).
4. Dry storage before use, in sealed containers with desiccant.

Record for every batch: feedstock source and lot, process temperatures and times, activation agent and ratio, yield, and storage date.

Recommended characterization before use

Table 2. Filler characterization, applied to both the nanosheets and the comparison char. Techniques are recommendations; equivalent methods may be substituted if recorded.

Property	Suggested technique	Use in this protocol
Particle size distribution	Laser diffraction, or image analysis of SEM micrographs	Batch consistency
Thickness	SEM, TEM or AFM	Comparison with reference route
Surface area	Nitrogen adsorption (BET)	Interpretation of uptake (Sec. 7)
Porosity	Nitrogen adsorption pore-size distribution	Interpretation of uptake (H3)
Porosity after processing	Repeat surface area and pore-size distribution after any milling or sonication	Confirms pores survive dispersion (H3)
Morphology	SEM imaging	Baseline for Sec. 12
Skeletal density, ρ_f	Helium pycnometry	Void-content calculation (Sec. 11)

4.2 Comparison filler: hemp char

Prepare a conventional char from the same feedstock lot used for the nanosheets. Carbonize it under nitrogen at the same peak temperature used in the nanosheet route, omitting the hydrothermal pretreatment and activation steps; then mill it to a particle size comparable to the nanosheets, and dry and store it as above. Characterize it with the methods in Table 2. Group E uses this char to separate the effect of nanosheet morphology from that of hemp-derived carbon in general (H4).

4.3 Matrix systems

System A — conventional thermoset epoxy

Examples: West System epoxy; Pro-Set epoxy; or an equivalent laboratory-grade epoxy.

System B — bio-based epoxy vitrimer

Potential components: a bio-based epoxy resin; a dynamic cross-linking (bond-exchange) catalyst; and a renewable hardener system.

For both systems, record resin and hardener grades, lot numbers, mix ratio, manufacturer's pot life (working time) and the cure and post-cure schedule from the technical data sheet.

4.4 Dispersion aids (optional)

- **Ethanol**, anhydrous reagent grade, as a removable dispersion medium.
- **Furfuryl glycidyl ether (FGE)**, CAS 5380-87-0, $C_8H_{10}O_3$, 154.17 g/mol, density 1.122 g/mL [23], as a reactive diluent that stays in the epoxy network. FGE carries the furan ring of furfuryl alcohol, which is made by hydrogenating furfural sourced from sugarcane bagasse [24].

Use of either aid, and its effect on each matched neat control, is set out in Section 8.

5 EXPERIMENTAL DESIGN

Table 3. Experimental groups. All groups are prepared in both matrix systems.

Group	Description	Composition	Purpose
A	Control (neat matrix)	Resin system only	Baseline mechanical performance
B	Untreated nanosheets	Resin + dry hemp carbon nanosheets	Evaluate reinforcement from untreated filler
C	Epoxy-impregnated nanosheets	Resin + pre-soaked nanosheets (Method 1)	Evaluate effects of nanosheet impregnation on composite performance
D	Vacuum-assisted impregnated nanosheets	Resin + vacuum-infused nanosheets (Method 2)	Maximize resin penetration of nanosheet porosity
E	Hemp char (comparison)	Resin + dry hemp char (Section 4)	Isolate the effect of nanosheet morphology (H4)

Filler loadings. Groups B, C, D and E are each prepared at 0.5 wt%, 1.0 wt%, 2.0 wt%, and 5.0 wt%.

LOADING BASIS

Filler loadings are calculated on the *dry* filler mass (M_d for nanosheets, Section 7). Resin carried into the batch by impregnated nanosheets in Groups C and D is counted as part of the matrix fraction, and the added resin and hardener are reduced so that total resin and the hardener ratio stay correct. Where FGE is used, it is likewise counted as part of the matrix fraction. Groups B, C, D and E are therefore compared at equal carbon content.

SOAK DURATION FOR GROUP C

The ambient-soak study (Section 6) is run at all three durations. Group C composites are then prepared at the single duration selected from the uptake results — the shortest duration at which uptake reaches a plateau within the resin’s working time — and the choice is recorded with its justification.

Table 4. Minimum test matrix per matrix system (n = 5 replicates per test).

Item	Count	Per system	Both systems
Formulations (A: 1; B, C, D, E: 4 loadings each)	17	17	34
Moulded specimens per formulation (tensile, flexural, compression)	3×5	255	510
Dispersion pilot (3 routes $\times$ neat and 1.0 wt% Group B; tensile only)	6×5	30	60

Hardness readings may be taken on untested flexural-bar ends or dedicated coupons. Vitrimer-specific tests (Section 13) and Phase 2 matrices (Section 15) are additional.

6 NANOSHEET IMPREGNATION PROCEDURE

6.1 Method 1: ambient soak

1. Dry nanosheets at 80 °C for 12 h.
2. Record initial (dry) mass, M_d .
3. Immerse nanosheets in mixed epoxy.
4. Allow soaking for 30 min, 60 min, and 120 min.
5. Remove excess resin.
6. Record impregnated mass, M_i .

6.2 Method 2: vacuum impregnation

1. Place dry nanosheets (dried as in Method 1, step 1) into the vacuum chamber.
2. Apply vacuum.
3. Introduce epoxy resin.
4. Maintain vacuum for 30 min.
5. Return to atmospheric pressure.
6. Remove excess resin.
7. Record mass increase.

Record the absolute chamber pressure, chamber temperature and the time from mixing to resin introduction.

IMPLEMENTATION NOTE

- **Working time.** Soak and vacuum durations must fall within the pot life of the mixed resin. Where a duration exceeds the working time of the chosen hardener, use a slower or extended-working-time hardener for this study, or record that duration as not feasible for that system.
- **Excess-resin removal.** Use one method for every batch (for example, vacuum filtration through a fine mesh, or centrifugation at a fixed speed and time) and record it. Uptake values are only comparable if excess resin is removed identically.
- **Mass measurement.** Weigh on an analytical balance and record its resolution.

7 EPOXY UPTAKE DETERMINATION

With M_d the initial dry mass and M_i the impregnated mass, epoxy uptake is

$$U = \frac{M_i - M_d}{M_d} \times 100 \%. \quad (1)$$

Uptake is recorded for every specimen batch (Appendix A, Table 12). Because M_i includes resin held on external surfaces as well as resin within the pores, uptake is interpreted together with the surface-area and porosity data in Table 2 and the void-content results in Section 11.

8 COMPOSITE MANUFACTURING

Mixing procedure

1. Prepare the resin system.
2. Add nanosheets (or char, Group E) gradually.
3. Stir mechanically.
4. Degas under vacuum.
5. Cast specimens into moulds.
6. Cure according to the resin manufacturer's specifications.

IMPLEMENTATION NOTE

Apply the same stirring speed, stirring time, degassing pressure and degassing time to every group, and record them. Differences in dispersion between groups should then reflect filler treatment rather than processing. Record mould type, release agent, cure schedule and any post-cure.

Dispersion routes

Table 5. Dispersion routes. Each route has its own matched neat control, made the same way without filler.

Route	Procedure	Matched neat control	Checks
R1 Direct	Stir the dry filler into the resin as in the mixing procedure above.	Resin with the same stirring and degassing.	None beyond the standard records.
R2 Ethanol-assisted	Ultrasonicate the filler in ethanol; stir the dispersion into the warmed resin until the ethanol has evaporated; then add hardener (and catalyst for System B).	Resin taken through the same ethanol, heating and stirring cycle without filler.	Weigh to constant mass before adding hardener to confirm ethanol removal; record sonication power and time.
R3 FGE reactive diluent	Disperse the filler in FGE (with ultrasonication if needed), blend with the epoxy resin, adjust the hardener for the blend's epoxide content, then cure.	Resin plus the same FGE fraction, no filler.	Hold the FGE fraction constant across all groups and record it.

For route R3 with an amine hardener, the blend's epoxide equivalent weight and the hardener dose follow from

$$\frac{1}{\text{EEW}_{\text{blend}}} = \frac{w_{\text{resin}}}{\text{EEW}_{\text{resin}}} + \frac{w_{\text{FGE}}}{\text{EEW}_{\text{FGE}}}, \quad \text{hardener (phr)} = \frac{100 \text{ AHEW}}{\text{EEW}_{\text{blend}}}, \quad (2)$$

where w are mass fractions of the epoxide components, AHEW is the hardener's amine hydrogen equivalent weight, and $\text{EEW}_{\text{FGE}} \approx 154 \text{ g/eq}$ because each FGE molecule (154.17 g/mol) carries one epoxide group [23]. For proprietary systems sold by fixed mix ratio, obtain the hardener's amine equivalent from the manufacturer before using R3.

WHY MATCHED CONTROLS

Solvent-assisted dispersion is common: Vaishnav *et al.* dispersed carbon nanosheets in ethanol by ultrasonication and evaporated it from the resin at 60 °C before adding cross-linker [10]. Retained solvent is harmful, however; acetone left in epoxy nanocomposites caused pores of up to about 80 µm and lowered properties [25]. FGE avoids the removal step because it becomes part of the network, but it changes the matrix on its own. In studies reviewed by Malburet *et al.*, 10–15 wt% FGE raised tensile and flexural strength by about 16 % without changing the glass transition, and 20 wt% FGE in an amine-cured bisphenol-A epoxy raised tensile strength by up to 40 % [26]. Every FGE formulation is therefore compared with an FGE-matched neat control, never with plain resin.

FGE AS IMPREGNATION LIQUID

Groups C and D may use FGE instead of mixed epoxy as the soak or vacuum-infusion liquid. With no hardener present, the soak is not limited by pot life, and as a viscosity-reducing diluent [26] FGE may penetrate the nanosheet pores more readily; the absorbed FGE then joins the network when resin and hardener are added. FGE's furan ring also offers a handle for furan–maleimide Diels–Alder chemistry, one of the reversible covalent routes discussed in the vitrimer literature [8], which could be explored for System B.

Dispersion pilot

Before the main study, run a pilot in each matrix system: Group A and Group B at 1.0 wt% for each of routes R1–R3 (six formulations; tensile testing and optical microscopy only). Choose the route that gives the best dispersion while its matched neat control still passes the baseline acceptance check (Section 16), and use it for every group thereafter. If R2 or R3 is chosen, its matched neat control becomes Group A for the main study. The other epoxy options in Table 11 (dispersing in the hardener, or an SDS surfactant) may be added to the pilot as further routes, each with its own matched control.

9 TEST SPECIMENS AND STANDARDS

Minimum replicates: $n = 5$ specimens per condition for each test. Condition specimens before testing in accordance with ASTM D618, and record the conditioning used.

Table 6. Recommended standards (current editions verified October 2026).

Test	Standard	Notes for this protocol
Tensile	ASTM D638-22 [27]	Dumbbell specimens; the method covers thicknesses up to 14 mm, and sheeting under 1.0 mm falls under ASTM D882. The small Type V specimen is suited to batches where filler is limited [28].
Flexural	ASTM D790-25 [29]	Three-point loading of a simply supported rectangular bar; the test ends at rupture or 5.0 % outer-fibre strain, whichever comes first.
Compression	ASTM D695-26 [30]	Rigid plastics, including reinforced grades; technically equivalent to ISO 604.
Hardness	ASTM D2240-15(2021) [31]	Type D durometer (Shore D).
Density	ASTM D792-20 [32]	Displacement method; Test Method A (in water).

10 MECHANICAL TESTING

Tensile properties

- Ultimate tensile strength
- Tensile modulus
- Elongation at break

Flexural properties

- Flexural strength
- Flexural modulus

Compression properties

- Compressive strength
- Compressive modulus

Hardness

- Shore D hardness

Report the failure mode of every specimen. Retain tensile fracture surfaces for SEM (Section 12).

11 PHYSICAL CHARACTERIZATION

11.1 Density

Determine the composite density ρ_c for each formulation by displacement (ASTM D792 [32]).

11.2 Void content

Void content is estimated by density comparison and by microscopy. The theoretical (void-free) density follows from the formulation mass fractions of filler, w_f , and matrix, w_m (with $w_f + w_m = 1$), and the corresponding densities:

$$\rho_t = \frac{1}{\frac{w_f}{\rho_f} + \frac{w_m}{\rho_m}}, \quad V_v = \frac{\rho_t - \rho_c}{\rho_t} \times 100 \%, \quad (3)$$

where ρ_f is the skeletal density of the filler, nanosheets or char (Table 2), and ρ_m is the density of the cured neat matrix (Group A).

METHOD CHOICE

ASTM D2734 determines void content by burning off the resin, and applies only where the effects of ignition on the reinforcement and fillers are known [33]. A carbon filler that may itself oxidize does not meet that condition, so Equation (3) uses formulation mass fractions instead of burn-off resin content. Because ρ_f is a skeletal density, nanosheet pores left unfilled by resin register as voids; a lower V_v in Groups C and D than in Group B at equal loading is therefore an indirect, bulk-level indicator of pore infiltration (H3).

11.3 Surface quality

Record, with photographs: defects; cracks; delamination; and agglomeration.

12 MICROSCOPY ANALYSIS

Optical microscopy — evaluate

- Filler distribution
- Agglomerates
- Void formation

Scanning electron microscopy — evaluate

- Nanosheet morphology
- Matrix adhesion
- Fracture surfaces
- Pull-out mechanisms
- Porosity infiltration

Image fracture surfaces from tensile specimens at matched magnifications across groups. Apply a thin conductive coating if specimens charge.

13 BIO-BASED VITRIMER EVALUATION

Repeat Sections 5 through 12 using bio-based epoxy vitrimer formulations (System B), including the neat vitrimer control (Group A). Then carry out the following additional measurements.

13.1 Stress relaxation

Determine relaxation time constants and dynamic bond-exchange behaviour. Recommended practice: record the normalized relaxation modulus $G(t)/G_0$ at four or more temperatures above the glass transition, take the characteristic relaxation time τ^* where $G(t)/G_0 = 1/e$, and fit

$$\ln \tau^* = \ln \tau_0 + \frac{E_a}{RT} \quad (4)$$

to obtain the apparent activation energy E_a of bond exchange. Comparing τ^* and E_a between the neat vitrimer and the filled formulations tests whether vitrimer functionality is maintained (H2).

13.2 Reprocessability

Evaluate reformation after grinding and recovery of properties. Grind cured specimens, re-mould them under heat and pressure (record temperature, pressure and time), and retest. Report property recovery as

$$R_P = \frac{P_{\text{reprocessed}}}{P_{\text{original}}} \times 100 \%. \quad (5)$$

13.3 Self-healing potential

Measure mechanical property recovery after damage. Introduce controlled damage (for example, a cut or a pre-crack), heal under recorded temperature, time and contact pressure, and retest. Report healing efficiency as

$$\eta = \frac{P_{\text{healed}}}{P_{\text{virgin}}} \times 100 \%. \quad (6)$$

14 LOADING SWEEP AND INTERPHASE AGENTS

The core design (Section 5) covers 0.5–5 wt%. This section extends the loading from dilute to the maximum the matrix can hold, and adds agents that modify the filler–matrix interphase. It runs in three stages so that each stage decides what the next one tests.

14.1 Loading sweep

Table 7. Loading sweep. Groups B (nanosheets) and E (hemp char) are run at every loading with the dispersion route chosen in the pilot.

Regime	Loadings (wt%)	Processing	Neat counterpart
Dilute	0.1, 0.25, 0.5, 1, 2	Cast (Section 8)	Cast neat matrix (Group A)
Moderate	5, 10, 15, 20	Cast while the degassed mix still fills the mould without voids above the limit below; otherwise pressed	Cast neat matrix; pressed neat matrix where pressing is used
High	30, 40, 50, 60	Compression moulding of resin-coated or resin-soaked filler (the Group C and D routes) in a closed mould	Neat resin cured in the same closed mould under the same pressure and temperature cycle
Limit search	Steps of 5 above 60, then 2.5 near the limit	As for the high regime	As for the high regime

Record the viscosity of each uncured mix (rotational rheometer, fixed shear rate and temperature) and the moulding pressure, temperature and time. Run one moderate loading by both casting and pressing, so that the effect of the change in process is measured rather than assumed. Each loading is tested in flexure (ASTM D790, $n = 5$) and for density and void content (Section 11); tensile tests (ASTM D638) are added at five loadings chosen to span the range. Measure electrical conductivity on specimens that conduct, using ASTM D4496 for moderately conductive materials [34], and report loadings in both wt% and vol%.

14.2 Maximum loading

The maximum loading is the highest loading at which specimens, under the chosen process, (i) consolidate into bars that can be machined and tested intact, (ii) have a void content no more than 2 percentage points above their neat counterpart, and (iii) show filler wetted by matrix on fracture surfaces, with no dry clusters in SEM. The 2-point void limit is a starting value and may be set per matrix class before the sweep begins.

Before moulding, estimate the limit from the filler's resin demand. Measure the resin absorption OA_r , in grams of resin per 100 g of filler, by the spatula rub-out method of ASTM D281 [35] using the actual resin system in place of linseed oil (record this deviation), or by an absorptometer as in ASTM D2414 [36]. Then

$$w_{\max} \approx \frac{100}{100 + OA_r} \times 100 \%, \quad \phi_{\max} \approx \frac{1}{1 + \frac{\rho_f OA_r}{100 \rho_r}}, \quad (7)$$

where $w_{\max}$ and $\phi_{\max}$ are the estimated maximum mass and volume fractions of filler, and ρ_f and ρ_r are the filler skeletal density and the resin density. The volume form is the critical pigment volume concentration used in coatings: above it there is not enough binder to fill the space between packed particles, and air takes its place [37]. Because OA_r counts resin between particles as well as in pores, it should be at least the uptake U of Equation (1),

so $100/(100 + U)$ is an upper bound on $w_{\max}$. Compaction under pressure can raise the practical limit above this estimate, which is why the sweep locates it experimentally.

14.3 Interphase agents

Table 8. Interphase agents. Each agent is applied to both the nanosheets and the Group E char.

Agent	Action	Matrices	Evidence	Matched control
None (as-made filler)	Reference	All	—	Neat matrix
Resin pre-impregnation (Groups C, D; FGE soak)	Resin in the pores; mechanical interlocking	Epoxies, vitrimers	Resin infiltration of porous fillers is an established interlocking route in dental composites [19].	Neat matrix
Epoxy-terminated silane (for example, a glycidoxypopyl silane)	Covalent bridge to an epoxy network	Epoxies, epoxy vitrimers	Silane-functionalized graphene oxide with epoxy end groups raised tensile and flexural properties, fracture toughness and T_g above both neat epoxy and untreated graphene oxide [38].	Neat matrix (grafted and washed)
Amino silane (APTES); methacryl silane (MPS) for polyesters	Covalent bridge via amine or vinyl groups	Epoxies, vitrimers, polyesters	Amino silane added directly to a hempseed-oil vitrimer raised impact strength from 3.5 to 10.3 kJ/m ² [12]; silane-treated hybrids raised polyester modulus about 21.5 % [39].	Neat matrix; with the free silane dose if added directly
Polydopamine coating	Adhesive catechol-amine layer	Epoxies, polyamides	Polydopamine-modified whiskers at 7.5 wt% raised epoxy tensile and bending strength by about 72 % and 66 % [40]; polydopamine also bridged graphene oxide onto basalt fibres in polyamide 6 [41].	Neat matrix
Surfactant (SDS)	Physical adsorption	Epoxies, latex	See Table 11 [42, 43].	Neat matrix with the same SDS dose
Reactive diluent (FGE)	Joins the network	Epoxies	See Section 8 [26].	Neat matrix with the same FGE fraction
Maleic-anhydride-grafted polyolefin	Compatibilizer	PP, HDPE	See Table 11 [44].	Neat polymer with the same dose
Mild surface oxidation	Adds oxygen groups for the agents above	All	Acid treatment can damage graphene sheets [45]; keep it mild and measure its effect.	Neat matrix; oxidized char in Group E

Confirm each surface treatment before compounding: FTIR or XPS for the new functional groups, and thermogravimetric mass loss for the amount grafted. Read the interphase in the composite from dynamic mechanical analysis (loss-factor peak height and position), from SEM of fracture surfaces (clean pull-out versus matrix-coated sheets) and from void content.

STAGES

- **Stage 1, loading sweep.** As-made nanosheets and char across Table 7; locate the best dilute loading and the maximum loading.
- **Stage 2, interphase screen.** Every agent in Table 8 that suits the matrix, at the best dilute loading and at one high loading, for nanosheets and char (flexure, $n = 5$; H5).
- **Stage 3, confirmation.** The full Group A–E design of Section 5, with all tests, at the best loadings and agent from Stages 1 and 2. The vitrimer evaluation and Phase 2 inherit these settings.

Table 9. Approximate specimen counts for Stages 1 and 2, per matrix system.

Item	Formulations	Specimens
Stage 1: up to 17 loadings $\times$ 2 fillers, plus cast and pressed neat (flexure, $n = 5$)	up to 36	up to 180
Stage 1: tensile at 5 loadings $\times$ 2 fillers ($n = 5$)	10	50
Stage 2 (epoxy): 7 agents $\times$ 2 loadings $\times$ 2 fillers, plus 2 free-agent neat controls (flexure, $n = 5$)	30	150

15 PHASE 2: EXTENSION TO OTHER MATRIX CLASSES

Phase 2 tests whether hemp-derived carbon nanosheets reinforce matrices beyond epoxies and vitrimers. Each class is a separate sub-study with one governing rule: every filled formulation is compared with its *neat counterpart*, made from the same matrix lot and taken through the identical thermal, mechanical and chemical history, including any dispersion aid. A control that skips a processing step (an extra extrusion pass, a milling stage, a sintering cycle) is not a valid comparator.

Table 10. Phase 2 matrix classes. Standards are listed by designation; use the edition current at the time of testing.

Class and examples	Incorporation	Neat counterpart	Key tests	Notes
Other thermosets: unsaturated polyester, vinyl ester, phenolic, other bio-epoxies	As Phase 1, including resin pre-impregnation	Same resin and cure, no filler	ASTM D638, D790	Groups A–E apply. Phenolics link to the plant-plastic precedent (Section 1).
Vitrimers of other chemistries: transesterification, imine, disulfide	As Section 13	Same network, no filler	D638, D790; stress relaxation	Compare τ^* and E_a with the neat network (H2).
Thermoplastics: PLA, PP, HDPE, PET, ABS	Melt compounding (twin-screw), then injection moulding or extrusion	Same polymer through the same number of compounding passes and moulding	D638, D790	Groups A, B, E. Hemp-char precedents exist in PLA and other thermoplastics [17, 18].
Elastomers: natural rubber, SBR, silicone, TPE	Two-roll mill or internal mixer, then vulcanize or cure	Same compound without filler	ASTM D412 [46]	Groups A, B, E, plus carbon black at equal loading as the industry reference filler.
Ceramics: alumina, zirconia, silicon carbide	Wet-mix powders (for example, ethanol slurry), dry, sinter under vacuum or inert gas (for example, spark plasma sintering)	Same powder through the same milling and sintering cycle	ASTM C1161 [47], C1421 [48]	Carbon oxidizes if fired in air. Graphene platelets in spark-plasma-sintered alumina raised flexural strength by about 31 % and fracture toughness by about 27 % [49].
Metals: aluminium and its alloys	Powder metallurgy: ball-mill, compact, sinter or extrude	Same powder through the same milling and consolidation	ASTM E8/E8M [50]	Aluminium carbide (Al_4C_3) can form at carbon–aluminium interfaces; nano-sized carbide formed after annealing at 610 °C for 3 h, and coarse carbide is reported to be brittle [51].
Cementitious: Portland cement mortar, lime binders	Disperse in mixing water (ultrasonication, superplasticizer), then mix and cure	Same mix design and water-to-binder ratio, no filler	ASTM C109/C109M [52]	Record water demand. Biochar at 10 wt% cement replacement gave similar or higher mortar strength, mainly through internal curing [53].
Others: coatings, adhesives, geopolymers, asphalt	Per product class	Same formulation without filler	Governing standard for the class	Added case by case under the same rule.

15.1 Dispersants by matrix class

Table 11 gives the recommended dispersant or dispersion aid for every matrix in this protocol, with the evidence behind it. The recommendations come from work on graphene, graphene oxide and related carbons; hemp-derived nanosheets may respond differently, so each choice is confirmed in a pilot before the main runs.

Table 11. Recommended dispersants by matrix class. Each dispersant is used at a fixed, recorded dose and included at the same dose in that matrix's neat counterpart.

Matrix	Recommended dispersant	Evidence	Cautions and matched control
Epoxy and bio-epoxy (System A); epoxy-based vitrimers (System B)	FGE reactive diluent (R3); ethanol with ultrasonication, then evaporated (R2); dispersion in the low-viscosity hardener; SDS surfactant	FGE at 10–20 wt% raised strength in published DGEBA systems [26]. Ethanol sonication alone can exfoliate graphene without surfactants [54] and was used for carbon nanosheets in a vitrimer [10]. Dispersing in the hardener avoided solvent and raised modulus about 24 % and fracture toughness about 29 % at 0.3 wt% graphene [25]. With SDS, 0.3 wt% graphene raised tensile strength from 57.2 to 70.4 MPa (64.5 MPa without SDS) and T_g from 66 to 77 °C [42].	FGE and SDS stay in the matrix, so Group A carries the same dose. Remove ethanol to constant mass. In System B, confirm that the aid does not shift τ^* or E_a of the neat vitrimer (Section 13).
Unsaturated polyester, vinyl ester	Silane coupling treatment of the filler (APTES or MPS) with ultrasonication	Silane-treated graphene oxide/nanoclay hybrids raised the tensile modulus of unsaturated polyester by about 21.5 % [39].	Silanes bond to surface oxygen groups, so confirm the nanosheet surface chemistry first. Treat the Group E char the same way.
Phenolic	No class-specific dispersant identified; screen R1 and R2 in the pilot	—	Record solvent content of the resin as supplied.
Polypropylene, HDPE	Maleic-anhydride-grafted polyolefin compatibilizer (PP-g-MA; PE-g-MA as the polyethylene analogue), added in melt compounding	PP-g-MA with carboxyl-functionalized graphene raised PP tensile strength by 62 % and modulus by 20 % [44]; a pyrene-functionalized PP-g-MA with solvent-assisted melt blending raised modulus by 43 % at 2 wt% graphene nanoplatelets [55].	The compatibilizer changes the polymer; the neat counterpart contains the same dose. PE-g-MA was not verified here and is screened first.
PLA, PET, ABS	No class-specific dispersant identified; melt compounding, screened in the pilot	—	Neat counterpart passes through the same compounding history.
Natural rubber (latex route)	Aqueous surfactant in latex mixing: SDS, SDBS or an aromatic-headgroup surfactant	An anilinium aromatic tri-chain surfactant dispersed graphene nanoplatelets in natural rubber latex and raised conductivity by eleven orders of magnitude [43].	Surfactant remains in the rubber and goes into the neat compound. Dry rubbers (SBR, silicone) are mill-mixed; no dispersant identified.
Ceramics (alumina)	Colloidal adsorption–precipitation assembly on an oxygenated carbon surface; or co-milling of the powders	Graphene oxide assembled with Al^{3+} on alumina, then spark-plasma sintered: flexural strength 428 MPa (+43 %) and fracture toughness 4.40 MPa m ^{1/2} (+34 %), about 15 % above ball-milled equivalents [56]. Co-milling alumina with graphite for 30 h produced graphene sheets in situ [57].	The adsorption route relies on an oxidized, graphene-oxide-like surface; hemp nanosheets may need mild oxidation, applied equally to Group E. Monolithic alumina gets the same milling and sintering cycle.
Metals (aluminium)	Dry milling with stearic acid as process control agent; or wet dispersion with ethyl cellulose (non-ionic, steric)	With 2 wt% stearic acid, milling under argon and degassing at 400 °C for 2 h, 1 vol% graphene nanosheets raised hot-pressed alloy hardness from 81.2 to 174.7 HV [58]. Ethyl cellulose dispersed graphene nanoplatelets in aluminium better than SDBS (hardness +31 % at 0.5 wt%) [59].	Remove organics completely before consolidation and account for any residue. The neat powder receives the same agent, milling and degassing.

Matrix	Recommended dispersant	Evidence	Cautions and matched control
Cement mortar	Polycarboxylate-polymer superplasticizer with probe ultrasonication in part of the mixing water	With 0.1 % graphene nanoplatelets and 0.6 % superplasticizer by cement mass, the best polycarboxylate polymer cut paste resistivity by about 52 %; polycarboxylates outperformed naphthalene sulfonate, an ester-type product agglomerated, and acid functionalization damaged the sheets and suits the alkaline paste poorly [45].	Plain mortar carries the same superplasticizer dose and water-to-cement ratio; record flow.
Others (coatings, adhesives, geopolymers, asphalt)	Follow the matrix chemistry (an epoxy coating follows the epoxy row) and screen	—	Same rule: identical dose in the neat counterpart.

CHOOSING AMONG DISPERSANTS

Prefer aids that leave nothing foreign behind: a reactive diluent or the hardener that becomes part of the network, a compatibilizer from the same polymer family, or a solvent removed to constant mass. Use aids that remain in the material (surfactants, superplasticizers, coupling agents) when the matrix calls for them, at a fixed dose that the neat counterpart also receives.

PHASE 2 CONVENTIONS

- **Groups.** Resin pre-impregnation (Groups C and D) applies only where the matrix is a liquid resin during processing. Melt, powder and cement routes run Groups A, B and E.
- **Dispersion aids.** Use the dispersant recommended for each class in Table 11. Ethanol suits powder wet-mixing as well as resins. FGE is an epoxide and is used only in epoxy-based matrices.
- **Loadings.** Apply the loading sweep and maximum-loading method of Section 14 within each class, and report both wt% and vol%, because matrix densities differ widely.
- **Order.** A suggested sequence runs from lowest to highest processing risk: other thermosets and vitrimers (shared Phase 1 methods), thermoplastics, elastomers, cementitious binders, ceramics, then metals.
- **Success.** The criteria of Section 17 apply within each class, judged against that class's neat counterpart.

16 DATA ANALYSIS

Baseline acceptance

Percentage gains are only meaningful against a properly cured control. Before any filled group is compared with it, Group A must reach at least 90 % of the resin manufacturer's published neat-resin tensile strength and modulus, cured under the conditions on which those figures are based.

REFERENCE VALUES

For example, West System lists its 105 resin with 205 hardener at 7846 psi (about 54.1 MPa) tensile strength, 4.08×10^5 psi (about 2.81 GPa) tensile modulus and 3.4 % elongation after a two-week room-temperature cure, and states that these figures are not specifications [14]. If Group A falls short, correct the mixing or cure and remake it before testing filled groups. For System B, where no published reference exists, report Group A in absolute units with its coefficient of variation and compare all filled groups against that batch. All results are reported in absolute units as well as percentages.

Comparisons

Neat matrix (A); untreated nanosheets (B); impregnated nanosheets (C); vacuum-impregnated nanosheets (D); hemp char (E) — in each matrix system. In the dispersion pilot, each route is judged against its own matched neat control. In Phase 2, each matrix class is analysed separately against its own neat counterpart.

Quantities

- **Percent improvement** relative to a reference group: $\Delta P = \frac{P - P_{\text{ref}}}{P_{\text{ref}}} \times 100 \%$.
- **Statistical significance.** Report mean $\pm$ standard deviation. Recommended: two-way ANOVA (filler type or treatment $\times$ loading) for Groups B–E, one-way ANOVA with Tukey's post-hoc test for comparisons including Group A, and $\alpha = 0.05$. Where normality or equal-variance assumptions fail, use Welch's ANOVA or the Kruskal–Wallis test and record the substitution.
- **Correlation between epoxy uptake and performance.** Pearson or Spearman correlation of U (Equation 1) against each mechanical property and against V_v .

Report every specimen tested, including outliers and invalid tests, with the reason for any exclusion.

17 SUCCESS CRITERIA

The project will be considered technically feasible if any of the following are observed:

- $\geq 5 \%$ increase in tensile strength;
- $\geq 5 \%$ increase in flexural strength;
- reduced void content;
- improved nanosheet distribution;
- improved matrix adhesion;
- maintained vitrimer reprocessability.

COMPARATORS

The impregnation effect (H1, H3) is judged for Groups C and D against Group B at the same loading; the morphology effect (H4) is judged for Group B against Group E at the same loading; and the reinforcement effect is judged for Groups B–E against Group A, once Group A has passed the baseline acceptance check (Section 16). A quantitative criterion counts as met only when the difference is also statistically significant at $\alpha = 0.05$. Distribution and adhesion are judged from microscopy at matched magnifications. Reprocessability is judged against the neat vitrimer (Section 13). Where a dispersion aid is used, Group A is the matched neat control for that route (Table 5).

18 ANTICIPATED OUTCOMES

The following potential benefits are hypotheses to be tested, not projected results:

- Improved interfacial bonding
- Enhanced matrix wetting
- Reduced agglomeration
- Increased mechanical performance
- Improved sustainability profile
- Establishment of a new hemp-derived nanocarbon composite platform

VERSION 1.6 BINDING AMENDMENT — §17 SUCCESS CRITERIA

Scope. This page is a binding clarification to §17 of the retained v1.5 technical text. Where §17's original wording conflicts with this amendment, this amendment controls.

Statistical feasibility correction. The original §17 lists a $\geq 5\%$ increase in tensile or flexural strength as a potential technical-feasibility signal, then requires statistical significance at $\alpha = 0.05$. With only $n = 5$ specimens per group, a 5% difference should not be treated as a confirmatory success threshold without a documented, endpoint-specific power analysis using defensible variance estimates. A universal minimum detectable effect cannot be established from the protocol alone.

Revised interpretation of the 5% figure. Treat the 5% improvement as a minimum practically important difference to inform a subsequent, adequately powered study—not as a stand-alone pass/fail criterion for the initial $n = 5$ /group feasibility screen.

Initial feasibility-stage decision rule. For each pre-specified primary endpoint, report all individual observations, group means, standard deviations, effect estimates, confidence intervals, and the exact sample size. Describe observed differences as exploratory. Do not claim statistical confirmation unless the planned design and analysis are justified by an endpoint-specific power analysis and the test assumptions are evaluated. A non-significant result at this screening sample size must not be interpreted as evidence of no effect.

Before confirmatory testing. Use pilot variance estimates and the intended comparator, endpoint, alpha, target power, and multiplicity plan to calculate the required sample size. Pre-specify one primary mechanical endpoint (or a justified hierarchy) before collecting confirmatory data. Keep microscopy, void content, adhesion, and vitrimer reprocessability as separate secondary or mechanistic criteria unless a formal composite endpoint is justified.

Other success signals. Reduced void content, improved nanosheet distribution, improved matrix adhesion, and maintained vitrimer reprocessability remain exploratory technical signals. Define the measurement method and acceptance rule for each before testing; do not retrospectively promote an outcome to primary status.

Interpretation. This amendment does not assert that the protocol is statistically powered or that any performance gain has occurred. No experimental results are reported.

19 SAFETY AND HANDLING

- Follow the safety data sheet for every resin, hardener and catalyst; wear nitrile gloves, eye protection and a laboratory coat when mixing and casting.
- Handle dry nanosheet powders in a fume hood or ventilated enclosure, with respiratory protection appropriate to fine carbon particulates, and avoid generating airborne dust when weighing and transferring.
- Inspect vacuum chambers and desiccators for damage before use and operate them behind a shield.
- Ethanol is highly flammable: keep ignition sources away, sonicate and evaporate under extraction, and never heat it on an open flame.
- FGE is harmful if inhaled and may form explosive peroxides [23]: handle it under extraction, date containers on opening, store them as the safety data sheet directs, and test for peroxides before use.
- Handle fine ceramic and metal powders according to their safety data sheets, under inert atmosphere where specified, and follow institutional procedures for sintering furnaces and compressed gases.
- Dispose of uncured resin, solvents and nanomaterial waste according to institutional and local requirements.

20 PROPOSED PUBLICATION

A web-based prior-art search (27 October 2026) found no earlier experimental study of hemp-derived carbon nanosheets as a reinforcement phase. Confirm the priority claim with a professional database and patent search before submission.

FIRST EXPERIMENTAL EVALUATION OF EPOXY-IMPREGNATED HEMP-DERIVED CARBON NANOSHEETS AS REINFORCEMENT PHASES IN THERMOSET EPOXY AND BIO-BASED VITRIMER COMPOSITE SYSTEMS

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VERSION 1.6 SAFETY SUPPLEMENT — §19

Scope. Retain §19, “Safety and Handling,” in full. The following items supplement rather than replace the existing safety instructions. Complete a site-specific risk assessment and consult current supplier safety data sheets before work begins.

1. Combustible-dust / dust-explosion hazard. Treat dry fine carbon and other combustible powders as an inhalation and potential combustible-dust hazard. Minimize dust generation and accumulation; use an appropriate ventilated enclosure and compatible dust-control/collection method. Keep ignition sources away from powder handling, avoid dispersing dust into air, and do not use ordinary vacuum equipment unless it is specifically suitable for the hazard. Have the laboratory safety officer assess whether the quantities and process create a deflagration risk and specify controls.

2. FGE skin sensitization and exposure control. Handle furfuryl glycidyl ether (FGE) in a functioning fume hood or equivalent containment. Prevent skin and eye contact; select gloves and other PPE from the current SDS and institutional chemical-resistance guidance rather than assuming one glove material is universally suitable. Keep containers closed, label secondary vessels, and follow the SDS for spill response, storage, and waste. Stop work and seek occupational-health advice if sensitization or exposure symptoms are suspected.

3. Press and mechanical-equipment guarding. Before operating any hot press, hydraulic press, or compaction equipment, verify that guards, interlocks, emergency stops, and pinch-point protections are in place and functional. Keep hands and tools out of closing zones; use approved loading/unloading tools and lockout/tagout procedures for adjustment, clearing, or maintenance. Do not bypass safety interlocks.

4. Per-batch nanomaterial SOP and traceability. Before each batch, document material identity and source, batch/lot number, mass, morphology, storage condition, handling enclosure, PPE, dispersion/sonication conditions, spills or deviations, waste route, and responsible operator. Use a written batch-specific SOP and risk review for each nanomaterial and matrix combination; record changes rather than silently reusing a prior batch record. Label samples and waste so the material and hazard remain traceable.

Hold point. No experimental execution should begin until the responsible laboratory has reviewed and approved the chemical, nanomaterial, dust, equipment, ventilation, emergency-response, and waste controls for its actual facilities and scale.

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A RECORD SHEETS

Complete one row per batch. Retain raw balance printouts or exported files with the batch ID.

Table 12. Epoxy uptake log (Sections 6–7).

Batch ID	System	Method / time	M_d (g)	M_i (g)	U (%)	Notes (pressure, removal method)

Table 13. Formulation and cure log (Section 8).

Batch ID	Group	Route	Loading (wt%)	Resin + hardener (g)	Mixing (rpm / min)	Degassing (kPa / min)	Cure and post-cure

PREPARED FOR	Landry Industries Inc. / Seshat's Composites
RESEARCH FOCUS	Hemp-Derived Carbon Nanosheet Composite Reinforcement Platform
REVISION	1.5 — final, for experimental execution and publication development. Version 1.1 added the baseline acceptance check, the hemp-char comparison group (Group E, H4) and prior-art references; version 1.2 expanded the Background with the historical, Mitlin, vitrimer and hemp bio-epoxy literature and a table of published hemp-carbon composite data; version 1.3 added the dispersion routes (ethanol and furfuryl glycidyl ether), the dispersion pilot and the Phase 2 extension to other matrix classes; version 1.4 added a recommended dispersant for every matrix class (Table 11); version 1.5 adds the full loading sweep, the maximum-loading method and the interphase-agent screen (Section 14).
CREDITS	LandryIndustries.ca • MarieLandrySpyShop.com

VERSION 1.6 REVISION RECORD AND PRE-FREEZE CHECKLIST

Revision history update. v1.6 adds the AI use notice and contribution record; corrects the §17 success-threshold/power mismatch; integrates selected enhancement-pack items (loading-feasibility gate, primary endpoint, stage gates, instrumentation, positive control, and vitrimer pass criteria where specified in the retained protocol); confirms §19 Safety and Handling remains in force and is supplemented rather than replaced.

Important document-control note. The retained technical pages originated in v1.5 and retain their original page headers and text. This v1.6 file incorporates front matter and binding amendments. The amendments control in the event of conflict; they do not silently rewrite every occurrence of v1.5 wording. Review the full protocol for any further cross-references that should be harmonized before treating this as a clean, frozen laboratory SOP.

Pre-freeze checklist

- Confirm that §14's loading-feasibility gate, primary endpoint, stage gates, instrumentation, positive control, and vitrimer pass criteria are explicit and operational in the main body; the AI audit record alone does not verify their completeness.
- Retain §19 and implement the four safety supplements on the preceding page: combustible-dust hazard, FGE skin-sensitization/exposure control, press guarding, and per-batch nanomaterial SOP.
- Use the 5% mechanical improvement as a minimum practically important difference for a follow-on adequately powered study unless a defensible endpoint-specific power analysis justifies another rule.
- Verify each ASTM standard edition, supplier specification/SDS, and literature citation against the primary source before protocol freeze; no claim of independent verification is made by this revision.
- Confirm all process settings and acceptance rules are appropriate to the actual equipment, facilities, and laboratory safety review.
- PDF metadata: Title — Feasibility Study of Hemp-Derived Carbon Nanosheet Reinforcement in Epoxy and Bio-Based Vitrimer Composites; Author — Marie-Soleil Seshat Landry; Subject — Foundational Experimental Protocol v1.6; Keywords — hemp-derived carbon nanosheets; epoxy; vitrimer; nanocomposite reinforcement; AI-assisted review.

Principal Investigator: Marie-Soleil Seshat Landry

Signature: _____ Date: _____

NOTE TO PARTICIPATING LABORATORIES

Research objective: evaluate the full potential of hemp-derived nanosheets

The overarching goal of this programme is to test the relevant variables systematically and build an evidence-based understanding of the future potential of hemp-derived nanosheets across composite-material applications. The work is exploratory and comparative: it is intended to identify which material forms, processing routes, formulations and performance trade-offs warrant further development, rather than assume in advance that any one route will succeed.

Three complementary research pathways

1. Nanosheets as a standalone material. Characterize the hemp-derived nanosheet material itself, including its composition, morphology, structure, surface chemistry, purity, defects and relevant physical or chemical properties, to the extent supported by available instrumentation and sample quantity.

2. Nanosheets in functionalized form. Evaluate appropriate functionalization or surface-modification strategies and determine how those changes affect dispersion, compatibility, interfacial behaviour, processability, stability and composite performance. Record treatment conditions and include suitable untreated controls wherever feasible.

3. Hemp nanosheets as a precursor to graphene oxide (GO) and reduced graphene oxide (rGO). Assess whether hemp-derived nanosheets are a viable feedstock or precursor for GO/rGO-type materials. Treat this as a distinct research pathway requiring its own characterization, controls, yield and reproducibility measurements, and safety review. Do not assume that hemp-derived nanosheets are equivalent to GO or rGO without analytical evidence.

Experimental approach and reporting expectations

Where technically feasible, use a staged design that screens the relevant variables, documents interactions and uncertainty, and advances only promising conditions to confirmatory testing. Laboratories should report negative and inconclusive findings as well as positive results; distinguish measured results from hypotheses and interpretation; identify deviations from the protocol; and document sample provenance, preparation, batch identity, methods, controls, replicates, raw-data location and analytical limitations. Proposed variables and pathways may be refined jointly with the principal investigator before execution, subject to facility capability, ethics and safety approval.

Scope principle: The programme is not limited to proving reinforcement in epoxy. Its purpose is to map the scientifically defensible opportunity space for hemp-derived nanosheets—as a material in their own right, as functionalized materials, and as potential precursors to GO/rGO—and to let reproducible evidence guide subsequent development.

Principal Investigator: Marie-Soleil Seshat Landry

Protocol: Feasibility Study of Hemp-Derived Carbon Nanosheet Reinforcement in Epoxy and Bio-Based Vitrimer Composites, v1.6

Status: Research objective and scope note; not a claim of demonstrated feasibility or performance.