

The Ultimate Guide to Graphitic Carbon Nanosheets

Oct 22, 2026 · @Marie

Production, properties, composites and conversion to graphene oxide and reduced graphene oxide, with a 58-experiment laboratory program to close the open questions.

Landry Industries Inc. · LandryIndustries.ca · MarieLandrySpyShop.com · Moncton, New Brunswick, Canada

About this book

Hemp-derived carbon nanosheets are a credible, renewable route to two-dimensional graphitic carbon, but they are not yet a proven substitute for graphite-derived graphene in composites. This book sets out what is established, what is only reported, and what is still hypothesis. It then gives a lab the experiments that would settle the difference.

The central question is simple to state. Per kilogram, per dollar and per kilogram of CO₂-equivalent, can hemp-derived nanosheets, and the graphene oxide (GO) and reduced graphene oxide (rGO) made from them, match graphite-derived materials in real composites? Nobody has answered it with data yet.

How the book is organized

Part	Chapters	What it gives you
I. Foundations	1-2	Vocabulary, structure, and every feedstock worth considering
II. Production	3-6	Top-down, bottom-up and hemp routes; conversion to GO and rGO
III. Properties	7	Side-by-side numbers for the standalone materials
IV. Composites	8-11	Polymer, bio-epoxy and vitrimer, cement, metal, ceramic, coating and energy composites; modelling
V. Characterization	12-13	What to measure, how, and to which standard

Part	Chapters	What it gives you
VI. Closing the gaps	14–16	A gap registry, 58 executable experiments, and how to run them
VII. Outlook	17	Where hemp wins, where it does not, and why

Evidence labels

Every significant claim carries one label. Readers, reviewers and funders should be able to see at a glance how much weight each statement can bear.

Label	Meaning	Typical use
[E] Established	Reproduced by many independent peer-reviewed studies	Hummers oxidation of graphite yields GO
[R] Reported	Published, but from one group or a few studies; needs replication	Hemp bast nanosheets reaching 2287 m ² /g
[H] Hypothesis	Reasoned proposal with no direct experimental data yet	Hemp nanosheets toughen a bio-epoxy vitrimer
[T] Design target	A projected number to test against, not a result	A fracture-toughness gain set as a go/no-go gate

Status of the hemp composite claims

All hemp-nanosheet composite performance in this book, including everything about Hempoxies, is a TRL 1–2 forward-looking research hypothesis. Every performance figure for those systems is a projected design target, not a validated result. Chapters 15 and 16 exist to change that.

Terminology used here

- **GCNS** (graphitic carbon nanosheets): the umbrella term for sheet-like sp²-carbon of any origin, from single-layer graphene to platelets tens of nanometres thick.
- **HCNS** (hemp-derived carbon nanosheets): GCNS made from any part of the hemp plant.

- **Mitlinite**: the author’s working name for HCNS made by the Wang–Mitlin hydrothermal-plus-activation route, first proposed in a [Landry Industries priority declaration on Zenodo \(June 2026\)](#). It is not a standard term, so reports for external review should say HCNS and use ISO vocabulary (Chapter 1).
- Earlier related work by the author: [Groundbreaking Integration of Hemp-Derived Carbon Nanosheets in Composite Materials \(February 2025\)](#).

Safety notice

Several procedures here are hazardous. Hummers-type oxidation mixes concentrated sulfuric acid with potassium permanganate and is strongly exothermic; KOH activation runs at 700–800 °C with a corrosive melt; dry nanosheet powders are an inhalation hazard. No protocol in this book replaces an institutional hazard assessment, and Chapter 16 sets the minimum controls.

Chapter 1 — What graphitic carbon nanosheets are

Graphene means exactly one atomic layer of carbon. Nearly every bulk "graphene" product, and every hemp-derived nanosheet reported so far, is a multilayer stack. Name each material by layer count, lateral size, order and chemistry, not by the word graphene.

1.1 The official vocabulary

The reference vocabulary is [ISO/TS 80004-13](#), whose second edition was published on 2024-09-15 and replaced the 2017 edition. Its core layer-count terms are below. **[E]**

Term	Meaning	Layers
Graphene (1LG)	A single layer of carbon atoms, each bound to three neighbours in a honeycomb (ISO wording via the EMPIR GRACE guide)	1
Bilayer graphene (2LG)	Two well-defined stacked graphene layers	2
Few-layer graphene (FLG)	Three to ten well-defined stacked graphene layers	3–10

Term	Meaning	Layers
Graphene / graphite nanoplatelets (GNP)	Stacks thicker than FLG; commercial grades are typically a few to tens of nanometres thick	>10
Graphene oxide (GO)	Graphene-like sheets carrying oxygen groups on basal plane and edges, usually made by oxidizing and exfoliating graphite	Usually 1
Reduced graphene oxide (rGO)	GO after chemical, thermal or other reduction; partly restored sp^2 network, residual oxygen and defects	Usually 1–few

The Carbon journal's editors made the same point in 2013: descriptions of graphene materials should state thickness or layer number, lateral size and in-plane shape ([Bianco et al., Carbon 65:1–6](#)). ISO/TS 21356-1:2021 then sets the measurements that back such a description: layers or thickness, lateral flake size, disorder, layer alignment and specific surface area ([ISO/TS 21356-1](#)).

This matters commercially. A 2018 study of material bought from 60 producers found that most sold graphite microplatelets, and that most samples contained under 10% material meeting the ISO definition of graphene ([Kauling et al., Advanced Materials; Physics World summary](#)). Only one sample exceeded 40% high-quality graphene. **[R]** Any lab buying reference graphene for this program must therefore characterize it on arrival (Experiment C-01).

1.2 Structure in one page

- **Lattice.** sp^2 carbon in a hexagonal honeycomb; C–C bond length 0.142 nm. **[E]**
- **Stacking.** Graphite stacks layers ABAB (Bernal) at 0.335 nm. Turbostratic carbon stacks them at random rotations with wider spacing, near 0.344 nm. **[E]**
- **Why turbostratic matters.** Misoriented layers couple weakly, so they separate more easily in liquids and polymer melts; the Rice flash-graphene team cites this as a dispersion advantage ([Springer Nature behind-the-paper post](#)). **[R]**
- **Crystallite size.** In-plane coherence length L_a and stack height L_c (from X-ray diffraction and Raman, Chapter 12) describe how "graphitic" a carbon really is.
- **Defects.** Vacancies, edges, sp^3 sites, oxygen and nitrogen groups and pores. Each one lowers conductivity and stiffness but can raise reactivity and interfacial bonding.

1.3 Graphitizing and non-graphitizing carbon: the hemp problem in brief

Rosalind Franklin showed in 1951 that carbons split into two classes. Graphitizing carbons become graphite between 1700 and 3000 °C; non-graphitizing carbons show no uniform graphitic structure even at 3000 °C ([Franklin, Proc. R. Soc. A 1951](#)). [E] In the graphitizing class, La reaches about 100 nm at 3000 °C; in the non-graphitizing class it tops out near 10 nm ([review in C — Journal of Carbon Research, 2025](#)).

Cellulose forms non-graphitizing carbon ([Chen et al., BioResources 2018](#)). [E] Hemp bast fibre is cellulose-rich, so hemp carbon will not turn into graphite by heat alone. Catalytic graphitization (iron, nickel) and flash Joule heating are the realistic routes to more order (Chapters 4 and 5). This single fact shapes the whole hemp program.

1.4 Where hemp nanosheets sit

The benchmark hemp material is the Wang–Mitlin carbon nanosheet: partially graphitic, 10–30 nm thick, up to 2287 m²/g specific surface area and 211–226 S/m electrical conductivity ([Wang et al., ACS Nano 2013, 7:5131](#)). [R]

- At about 0.34 nm per layer, 10–30 nm corresponds to roughly 30 to 90 layer-equivalents. By ISO terms these are nanoplatelet-class sheets, not graphene or FLG.
- An ideal graphene monolayer has a theoretical surface area near 2630 m²/g, and a dense 30-layer stack would offer only a few percent of that. The very high area of hemp nanosheets must therefore come mostly from pores inside the sheets, not from thinness. [E] for the arithmetic; [H] for the pore attribution until gas sorption and microscopy confirm it (Experiment C-04).
- For composites, the pores cut both ways: resin can infiltrate and interlock, but a porous, partly disordered sheet is intrinsically less stiff than a dense graphitic one. [H]

The hemp route to GO and rGO runs through a graphitization step

Graphene-family materials by precursor. Accent: hemp route. Dashed: proposed here, tested in Chapter 15.

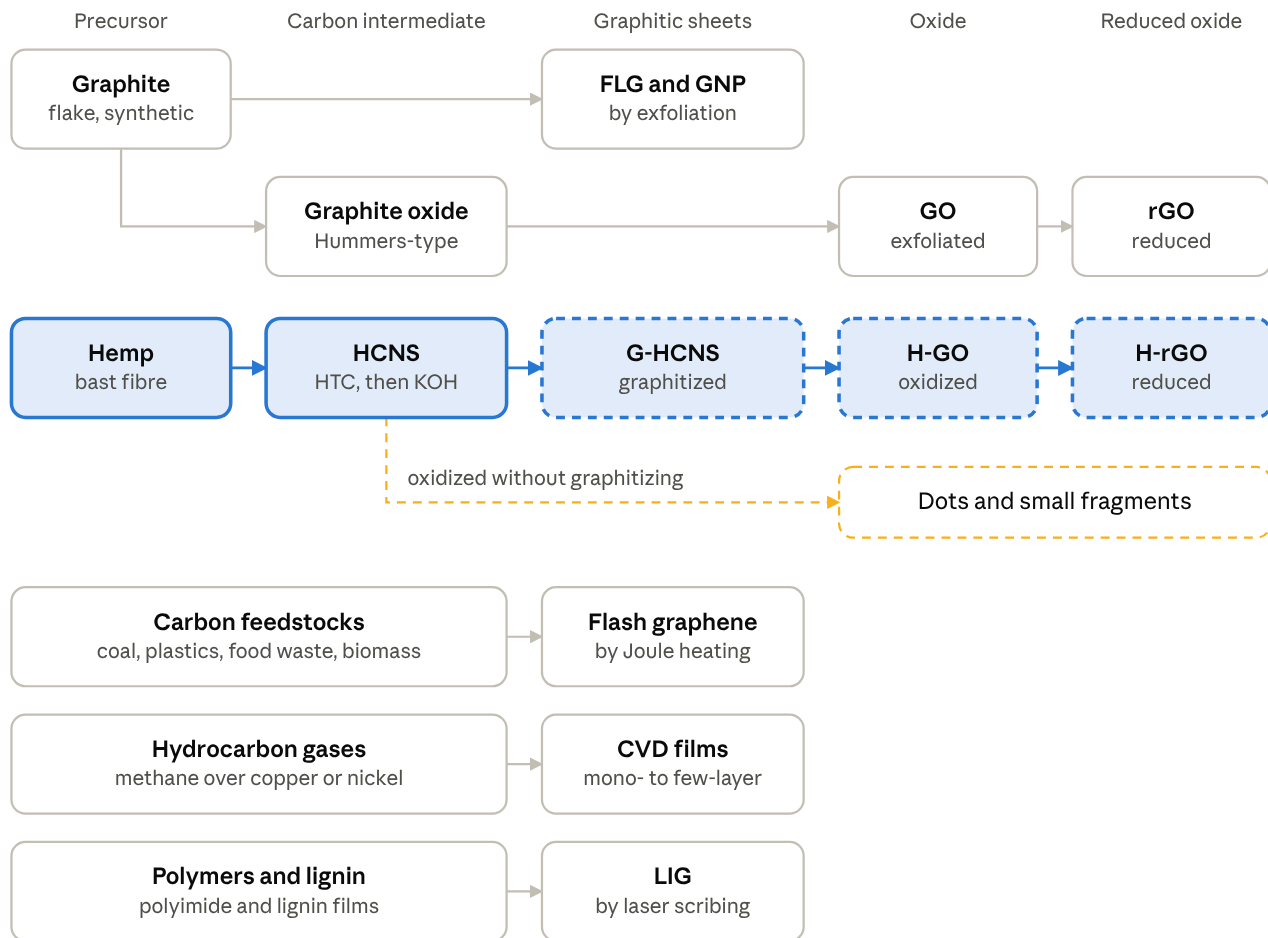


Figure 1.1 · graphene-family materials by precursor and route

Graphite reaches GO and rGO through graphite oxide. Hemp nanosheets are proposed to reach them only after a graphitization step, because oxidizing the disordered carbon directly risks giving dots and fragments rather than sheets.

Chapter 2 — Feedstocks

Graphite gives the most ordered nanosheets but carries real supply risk. Hemp gives renewable, naturally templated nanosheets with low graphitic order. Other biomass and wastes sit between the two, and each is a useful comparator.

2.1 Natural and synthetic graphite

- **Natural flake graphite** is the most crystalline common feedstock and the standard input for liquid-phase exfoliation and Hummers-type graphene oxide. [E]
- **Synthetic graphite** comes from graphitizing carbons such as petroleum and needle coke, heated toward 3000 °C (Chapter 1.3). [E]
- **Supply concentration.** One market analysis puts China at roughly 75% of natural graphite production ([Crux Investor](#)). China has required export licences for nine graphite items since 1 December 2023, announced further controls on artificial graphite anode materials and related equipment in October 2025, then suspended those additional controls until November 2026 ([Meticulous Research](#); [SMM on Announcement No. 58](#)).

For a Canadian program, that history is the strategic case for a non-graphite route. It is not, by itself, a technical case: hemp must still win on performance or cost.

2.2 Hemp

Anatomy. A hemp stalk is roughly 65% hurd (woody core) and 35% bast fibre ([Int. J. Fiber Sci. review](#)).

Fraction	Cellulose	Hemicellulose	Lignin	Notes
Bast fibre	57–77%	Lower than hurd	5–9%	Long crystalline-cellulose fibrils; the Mitlin precursor (review)
Hurd (Carmagnola)	44.0% α -cellulose	25.0%	23.0%	4.0% extractives, 1.2% ash (Gandolfi et al., BioResources 2013)
Hurd (decorticated, US)	45.66%	24.57%	21.67%	Waste industrial hemp stalk (Bokhari et al.)

The bast fibre used in the original hemp nanosheet work had 81.98 wt% volatiles and 2.95 wt% ash ([US Patent 9,478,365](#)). [R] Ash matters: potassium, calcium and silicon change activation chemistry and leave residues in the final carbon.

Why hemp might be special. The patent describes the nanosheets as carbonized, exfoliated crystalline-cellulose hemp fibrils ([US Patent 10,170,251](#)). The layered cell wall acts as a natural template for sheets. [R] Whether other bast fibres (flax, kenaf, jute) do the same is untested here and becomes Experiment A-04.

Canadian supply, 2025. Growers reported 15,380 hectares of hemp, of which 12,859 hectares (over 80%) were grown for grain; fibre acreage kept falling, and active licences dropped to 585 ([HempToday, citing Health Canada](#)). Alberta, Saskatchewan and Manitoba hold most of it. The practical feedstock is therefore straw from grain crops, not dedicated fibre hemp. How that straw's bast behaves in the Mitlin route is an open question (Experiment A-01).

2.3 Other biomass

- **Peat moss.** Mitlin's group made carbon nanosheet frameworks from peat moss for sodium-ion anodes ([Ding et al., ACS Nano 2013, 7:11004](#)). The cross-linked tissue suppressed graphite formation at high temperature ([Ding PhD thesis, U. Alberta](#)). [R]
- **Peanut shells.** The same group built a sodium-ion capacitor with both electrodes made from peanut shells ([author profile listing](#)). [R]
- **Agricultural residues.** A 2024 review found coconut shells gave crumpled multilayer graphene, rice husks a mix of graphene layers and amorphous carbon, and wheat straw few-layer graphene. It flagged low yield, inconsistent quality, poor scalability and toxic reagents as the main barriers ([Singh et al., 2024](#)). [R]
- **Lignin.** Being aromatic, lignin converts more readily to graphitic carbon under laser or flash heating. A 2026 Green Chemistry critical review covers lignin-derived laser-induced graphene ([RSC](#)). Hemp hurd carries 21–24% lignin, which suggests a split: bast for templated sheets, hurd for flash or laser routes. [H]

2.4 Fossil and waste carbon

- **Flash Joule heating** turned coal, petroleum coke, biochar, carbon black, food waste, rubber tyres and mixed plastic into turbostratic graphene in under a second ([Luong et al., Nature 2020, 577:647](#)). [R], replicated by later work from the same and other groups.
- **Methane and other hydrocarbons** make high-quality graphene films by chemical vapour deposition on copper. These are films for electronics, not bulk composite fillers. [E]

2.5 Feedstock roles in this program

Feedstock	Carbon order reachable	Typical product	Renewable	Role here
Natural flake graphite	Highest	GNP, GO, rGO	No	Primary benchmark control
Synthetic graphite	High	GNP, GO	No	Secondary control
Hemp bast (grain-crop straw)	Low by heat alone; higher with catalyst or flash	Porous HCNS	Yes	Primary candidate
Hemp hurd	Low; lignin-rich	Porous carbon; flash and laser feed	Yes	Secondary candidate
Flax or kenaf bast	Low	Possibly templated sheets	Yes	Comparator (is hemp unique?)
Peat moss, peanut shell, coconut shell	Low to moderate	Nanosheet frameworks	Yes	Literature comparators
Kraft or organosolv lignin	Moderate under laser or flash	LIG, flash graphene	Yes	Comparator
Coal, coke, carbon black	High under flash	Turbostratic flash graphene	No	Comparator for the flash route

Chapter 3 — Top-down production from graphite

Top-down routes peel ordered graphite apart and give the cleanest sheets; the costs are yield, a broad thickness distribution and solvent burden. Each route is also a candidate for exfoliating hemp carbon, once that carbon has been made ordered enough to peel (Chapter 5).

3.1 Liquid-phase exfoliation by sonication

Graphite sonicated in N-methyl-pyrrolidone gave graphene dispersions up to about 0.01 mg/mL, because solvents whose surface energy matches graphene's pay the energy cost of separating layers ([Hernandez et al., Nature Nanotechnology 2008, 3:563](#)). The monolayer yield was about 1 wt%, with 7–12 wt% possible after further processing. [E]

Sonication also shortens flakes and adds edge defects, and NMP is a reproductive toxicant that is hard to remove from composites. For this program, sonication is a characterization tool (dispersing samples for microscopy), not a production route.

3.2 Shear exfoliation

High-shear mixing of graphite in stabilizing liquids exfoliates it once the local shear rate passes about 10^4 s^{-1} , and the process scaled from hundreds of millilitres to hundreds of litres ([Paton et al., Nature Materials 2014, 13:624](#)). Flakes were unoxidized, 300–800 nm across and under ten layers; the team ran a 100-litre batch ([ChemistryViews](#)). [E]

Shear works in water with surfactant, which suits bio-epoxy and cement work. It is the default top-down route for this program's graphite controls.

3.3 Electrochemical exfoliation

Using graphite as an electrode in aqueous ammonium, sodium or potassium sulfate gave over 85% sheets of three layers or fewer, lateral sizes up to 44 μm and a C/O ratio of 17.2 ([Parvez et al., JACS 2014, 136:6083](#)). [R], with many follow-up studies.

The route needs a monolithic, conductive electrode. Hemp carbon is a powder, so it would first have to be pressed or bound into an electrode. Whether graphitized hemp carbon exfoliates this way is untested (Experiment B-05). [H]

3.4 Mechanochemical exfoliation (ball milling)

Ball-milling graphite with dry ice for 48 hours gave edge-carboxylated graphite that dispersed in polar solvents as single- and few-layer sheets (five or fewer layers). Films decarboxylated by heat reached 1214 S/cm ([Jeon et al., PNAS 2012, 109:5588](#)). [R]

The key feature is chemistry on the edges, not the basal plane. Edge carboxyls can react with epoxy and amine groups while the sheet keeps its conductivity, which makes this route attractive for epoxy interfaces (Experiment E-03).

3.5 Thermal expansion of intercalated graphite

Acid-intercalated graphite expands into a worm-like, low-density form when heated rapidly, and that expanded graphite can be milled or sheared into nanoplatelets. **[E]** The result is usually thicker and cheaper than the routes above, and it is the realistic comparator for bulk composite fillers.

3.6 Oxidative exfoliation

Oxidizing graphite to graphite oxide and exfoliating it in water gives graphene oxide. That route is the bridge to GO and rGO and has its own chapter (Chapter 6).

3.7 Top-down routes compared

Route	Typical layers	Basal-plane oxidation	Demonstrated scale	Main burden	Fit for this program
Sonication in NMP	1 to few; broad spread	None	Laboratory	Toxic solvent, low yield	Sample preparation only
Shear in water and surfactant	Under 10	None	Hundreds of litres	Surfactant removal	Graphite control route
Electrochemical, sulfate salts	Mostly 3 or fewer	Low (C/O 17.2)	Laboratory to pilot	Needs electrode form	Test on graphitized hemp carbon
Ball milling with dry ice	5 or fewer	Edge-only	Laboratory	Long milling, metal pickup	Interface-engineered filler
Thermal expansion	Tens of layers	Low	Industrial	Acids for intercalation	Bulk filler comparator
Oxidation (Hummers family)	Usually 1	Heavy	Industrial	Acid and manganese waste	GO and rGO route (Chapter 6)

Chapter 4 — Bottom-up and pyrolytic routes

Bottom-up routes build sp^2 order from almost any carbon by heat, light or a catalyst. For hemp they are the main lever against the non-graphitizing problem in Chapter 1.3, and flash Joule heating is the most promising of them.

4.1 Chemical vapour deposition

Methane over copper foil grew centimetre-scale films that were mostly single-layer, with under 5% of the area few-layer; low carbon solubility in copper makes growth self-limiting ([Li et al., Science 2009, 324:1312](#)). [E] CVD makes films for electronics and barrier coatings, not bulk fillers, so it appears here only as a quality reference.

4.2 Flash Joule heating

A high-current pulse through a conductive carbon bed takes it to about 3000 K in milliseconds. The original work turned coal, coke, biochar, carbon black, food waste, tyres and mixed plastic into gram quantities of turbostratic graphene in under a second, with no furnace, solvent or reactive gas, at about 7.2 kJ of electricity per gram ([Luong et al., Nature 2020](#)). The Raman D band was weak or absent. [R], now widely reproduced.

Biomass work has followed:

- **Continuous biomass flash graphene.** An automated system paired pyrolysis with flashing; graphene from medium-temperature biochar, without carbon black, came in at 1.9 g CO₂-eq per gram, about 86% less than flashing raw biomass ([Zhu et al., Nature Communications 2024](#)). [R]
- **Peanut shells.** A short Joule pre-carbonization followed by a flash pulse made graphene at 15.6 MJ of electricity per kilogram, about USD 1.30/kg; the authors identified precursor pre-treatment as the decisive variable ([Graphene-Info report](#)). [R]
- **Lignin.** Insulating lignin had to be mixed with ferric chloride to conduct enough to flash ([Chen et al., Res. Chem. Intermed. 2023](#)). [R]
- **Composites.** Coal-derived flash graphene at 20–50 wt% in a DGEBA epoxy raised Young's modulus by 92% at a 1:2 ratio, and hardness by 140% and toughness by 496% at 1:3 ([Advincula et al., Macromol. Mater. Eng. 2023](#)). [R]

Why this matters for hemp. KOH-activated hemp nanosheets already conduct at 211–226 S/m, so they should flash without additives. Flashing could add graphitic order while the sheet morphology survives, or it could collapse it. Nobody has tested this (Experiment B-03). [H]

4.3 Laser-induced graphene

A CO₂ infrared laser, in air, photothermally converts polyimide film into porous graphene that can be written in patterns ([Lin et al., Nature Communications 2014, 5:5714](#)). [E] Lignin-based precursors now work too ([2026 Green Chemistry review](#)).

Laser writing is a surface process, so it does not make bulk filler. Its role here is different: writing conductive tracks for strain sensing or heating directly into hemp-lignin-rich composite surfaces. [H]

4.4 Catalytic graphitization

Raw sawdust soaked in iron nitrate forms Fe₃C nanoparticles on heating; these etch through the char and leave graphitic tubules at only 800 °C. Five grams of sawdust gave 1.5 g of product ([Thompson et al., Green Chemistry 2015, 17:551](#)). [R] Precursor choice controls catalyst particle size and therefore porosity; cellulose gives mesoporous carbon ([Materials Advances 2020](#)). Nitrogen in the precursor inhibits the process ([Hunter, PhD thesis, Birmingham 2023](#)).

The risk for hemp is shape. Iron catalysis tends to make tubules and shells, which could destroy the sheet template that makes hemp interesting. Residual iron must also be leached out before oxidation or use in composites. Whether a light catalyst loading raises order while keeping sheets is the question behind Experiments B-01 and B-02. [H]

4.5 High-temperature annealing

Heating to 2000–3000 °C in argon removes heteroatoms and raises conductivity, but in non-graphitizing carbon the in-plane crystallite size stops near 10 nm (Chapter 1.3). It is expensive and energy-hungry, and serves here only as an upper-bound control (Experiment B-04).

4.6 Other biomass-specific routes

A review of biomass pyrolysis lists salt-based activation, chemical blowing, template confinement, hydrothermal pre-treatment and post-exfoliation as routes to graphene-like carbon ([Kong et al., ORNL record](#)). The Mitlin route is the hydrothermal-plus-activation member of this family.

4.7 Bottom-up routes compared

Route	Order achieved	Bulk powder?	Energy and chemicals	Status for hemp
CVD on copper	Highest (films)	No	Methane, hydrogen, ~1000 °C	Not applicable

Route	Order achieved	Bulk powder?	Energy and chemicals	Status for hemp
Flash Joule heating	High, turbostratic	Yes	Electricity only, ms pulses	Untested on HCNS; top priority
Laser-induced graphene	Moderate, porous	No (surface)	Electricity only	Sensors in composite surfaces
Fe-catalysed graphitization	High, tubular	Yes	Iron salt, 800 °C, acid leach	Untested on hemp bast
Anneal at 2000–3000 °C	Limited for hemp	Yes	Very high energy	Upper-bound control

Chapter 5 — Hemp-derived carbon nanosheets

The Wang–Mitlin route turns hemp bast into porous, partly graphitic sheets in two steps: hydrothermal carbonization, then KOH activation. It was tuned for supercapacitors, not for composites or oxidation, so nearly every parameter must be re-tuned for the uses in this book.

5.1 The original process

1. **Hydrothermal carbonization.** Hemp bast fibre and dilute sulfuric acid are sealed in a steel autoclave and held at 180 °C for 24 hours ([US Patent 12,300,438 example](#)). This removes the inner and outer cell-wall layers and loosens the links between microfibrils in the middle layer ([Zhi Li, Mitlin group, via Nanowerk](#)).
2. **KOH activation.** The resulting biochar is mixed 1:1 by mass with KOH and heated to 700–800 °C under argon ([ScienceDirect topic summary](#)). The KOH melt penetrates between microfibrils, separates them into sheets, and carbonizes and activates them, creating micro- and mesopores.
3. **Washing and drying.** The sources reviewed do not specify the wash sequence; Experiment A-05 fixes one.

The hydrothermal step is not optional. Hemp carbonized directly and then KOH-activated gave a smooth, dense structure instead of sheets ([US Patent 9,478,365](#)). **[R]** Acid concentration, fibre-to-liquid ratio, heating ramp, hold time at activation temperature and

yield are not given in the sources reviewed here. They are open parameters (Experiments A-02 and A-03).

5.2 What the three activation temperatures produced

Sample	Sheet thickness	BET area (m ² /g)	Conductivity (S/m)	C / N / O (atomic %, XPS)
Hydrothermal biochar	—	—	—	73.54 / 1.77 / 24.69
CNS-700	50–100 nm	1690	217	93.69 / 0.90 / 5.41
CNS-750	40–70 nm	2287	211	93.39 / 1.01 / 5.6
CNS-800	10–30 nm	1505	226	94.33 / 1.48 / 4.19
Commercial GNP (patent's reference)	—	—	—	93.97 / — / 6.03

Sources: thickness from [US 10,170,251 \(Google Patents\)](#); area and composition from [US 9,478,365](#); conductivity from [US 12,300,438](#). [R]

No single sample is both thinnest and highest in area. The often-quoted pairing of "10–30 nm" with "2287 m²/g" combines CNS-800's thickness with CNS-750's area. For composites, CNS-800 is the natural starting point: thinnest sheets, slightly higher conductivity, least oxygen. High-resolution TEM showed CNS-800 to be porous and only partially ordered ([US 9,478,365](#), [Google Patents](#)).

The electrochemical record shows the material is stable. CNS-750 electrodes reached 19, 34 and 40 Wh/kg at 20, 60 and 100 °C, and CNS-800 kept 96% of its capacitance after 10,000 cycles ([US 12,300,438](#)); the assembled device gave 12 Wh/kg ([ACS Nano abstract](#)).

5.3 Related hemp work since 2013

- **More KOH, more area.** Hemp bast processed in hot water, then activated with KOH or NaOH, reached 2425 m²/g; area rose with KOH up to a 1:3 biochar-to-KOH ratio, and so did surface oxygen ([C — Journal of Carbon Research, 2018](#)). [R]

- **Acid pre-treatment.** A non-peer-review-verified report used concentrated sulfuric and phosphoric acids on hemp bast before KOH and described one- to few-layer delaminated sheets ([ResearchGate preprint](#)). Treat as unconfirmed.
- **Nearest composite analogue.** Carbon nanosheets made from tyre waste by two-stage pyrolysis have been built into a vitrimeric thiol-epoxy with self-healing and shape memory ([Polymers, September 2026](#)). [R] It shows the concept works with a waste-derived nanosheet; it says nothing yet about hemp.
- **Nearest oxidation analogue.** Biochars from pine leaves and other residues have been oxidized to GO by modified Hummers methods ([Springer review of GO from waste sources, 2024](#)), and coal char has been oxidized by a nitric-acid route ([US Patent 11,731,875](#)). [R]

In the searches made for this book, no published report was found of Wang–Mitlin hemp nanosheets used as a structural composite filler, or oxidized to GO. Those two steps are the open frontier this program targets, and the Landry proposals cited in the front matter frame them as hypotheses. [H]

5.4 What composites need differs from what supercapacitors needed

Property	Supercapacitor optimum	Composite optimum	Implication for the process
Surface area	As high as possible	Moderate; very high area raises resin demand and viscosity	Lower KOH ratio or temperature (A-03)
Micropores	Many	Few; they trap air and absorb resin	Milder activation or a closing anneal (B-04)
Sheet geometry	Thin	Thin and laterally large: high aspect ratio	Avoid harsh milling; measure lateral size (C-03)
Oxygen groups	Secondary	A controlled amount bonds to epoxy and amines	Tune by annealing or grafting (E-02, E-03)
Graphitic order	Helps conductivity	Helps stiffness, conductivity and later oxidation	Graphitization step (B-01 to B-04)

Property	Supercapacitor optimum	Composite optimum	Implication for the process
Potassium and ash	Low	Very low; residues can upset cure and corrode	Strict washing, ICP check (A-05)
3D interconnection	Useful	Hinders dispersion, though it may help percolation	Controlled de-agglomeration (E-01)

The composite column is **[H]**: reasoned from composite science, not yet measured on HCNS.

5.5 Making hemp nanosheets more graphitic

Four levers exist, and each will trade surface area for order:

1. **Flash Joule heating** of finished HCNS (Experiment B-03). Highest potential; untested.
2. **Iron-catalysed graphitization** before or after activation (B-01, B-02). Proven on sawdust; risks turning sheets into tubules.
3. **Annealing at 2000–2800 °C** (B-04). Upper-bound control; limited by non-graphitizing chemistry.
4. **A graphitizable coating**, such as lignin- or pitch-derived carbon on the sheets before annealing (B-06). **[H]**

The decision rule is set in Chapter 15: a graphitized HCNS proceeds to oxidation only if X-ray diffraction and Raman show real stacking order (gate after B-07).

5.6 Scale-up and safety notes

- The hydrothermal step is a pressure-vessel operation and needs a rated autoclave with a burst disc.
- KOH activation at 700–800 °C is corrosive to many crucible materials and releases hydrogen; metallic potassium can form. Run it under inert gas and quench with care (Chapter 16).
- Mass yield of HCNS per kilogram of fibre has not been reported in the sources reviewed. It is the first number a techno-economic model needs (Experiment I-03).

Every hemp material passes a gate before it enters a composite

The Chapter 5 route with its Chapter 15 gates. Accent: products for composites. Tinted: go/no-go gates.

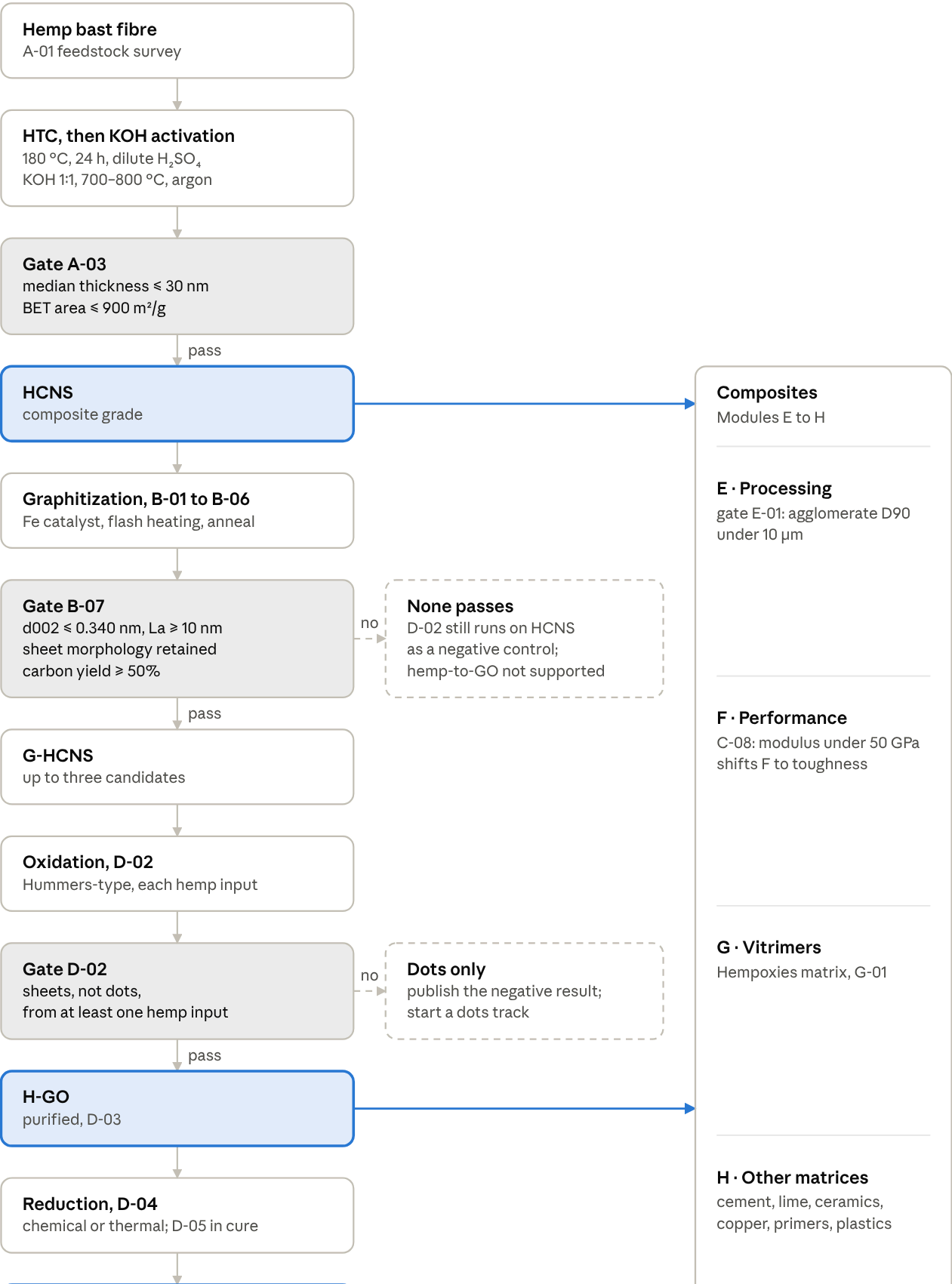




Figure 5.1 · the hemp route, its gates and where each product enters a composite

HCNS enter the composite modules once they pass A-03, and H-GO once D-02 shows sheets rather than dots. Oxidation runs on graphitized candidates that pass B-07, or on HCNS as a negative control if none does.

Chapter 6 — Transformation into graphene oxide and reduced graphene oxide

Oxidation makes carbon sheets water-dispersible and chemically reactive; reduction then restores part of the sp^2 network and its conductivity. For hemp the decisive question is whether its carbon is ordered enough to oxidize into sheets rather than into nanometre-sized dots.

6.1 Oxidation chemistry, from Brodie to the improved Hummers method

Brodie made graphite oxide with potassium chlorate in 1859, and Staudenmaier improved the route in 1898 ([Materials Research Express 2019 review](#)). Hummers and Offeman's 1958 method used sodium nitrate and potassium permanganate in concentrated sulfuric acid and cut the reaction to a few hours ([Scientific Reports 2016](#)). [E]

The improved method of Marcano and colleagues dropped sodium nitrate, raised the permanganate, and used a 9:1 mixture of sulfuric and phosphoric acids; it gives higher yield, evolves no toxic nitrogen oxides, and loses no conductivity after reduction ([Marcano et al., ACS Nano 2010, 4:4806, as summarized in US Patent 10,989,681](#)). [E] A documented laboratory run of that method:

1. Stir 1 g graphite flakes in 130 mL of 9:1 H_2SO_4/H_3PO_4 and add 6.0 g $KMnO_4$; the mixture self-heats to about 40 °C.
2. Hold at 50 °C with stirring for 12 hours, then cool to 20 °C.
3. Pour onto about 120 mL of ice water containing 1 mL of 30% H_2O_2 .
4. Sieve (300 μm), filter through glass wool, and centrifuge at 4000 rpm for 4 hours.
5. Wash twice with a 1:1:1 mix of water, 30% HCl and ethanol; sieve and centrifuge again.
6. Coagulate with diethyl ether, filter on a 0.45 μm PTFE membrane, and vacuum-dry for 12 hours. Yield: 1.8 g GO.

Source: [US Patent 10,989,681, Example 2](#). Experiment D-01 reproduces this exactly on graphite before anyone tries hemp carbon.

6.2 What graphene oxide is

The most cited structure is the Lerf–Klinowski model of 1998, built on solid-state NMR: hydroxyl and epoxide groups on the basal plane, carboxyl groups at the edges ([arXiv:1008.0937](#)). The model is useful but contested; spectroscopy has found local oxygen contents far above what it predicts ([arXiv:1612.03594](#)), and acid-rich "oxidative debris" clings to the sheets ([PMC6724119](#)). Oxidation also widens the interlayer spacing. **[E]** for the broad picture; **[R]** for any specific model.

6.3 Why order matters: sheets or dots

When coal was oxidized, its small crystalline domains were displaced more easily than pure sp^2 carbon, giving graphene quantum dots in up to 20% isolated yield ([Ye et al., Nature Communications 2013, 4:2943](#)). Dot size tracked the source: 2–4 nm from bituminous coal, 4–8 nm from coke, 18–40 nm stacked structures from anthracite ([Rice University via Nanowerk](#)). **[R]**

Non-graphitized hemp nanosheets are made of small, disordered domains too. Oxidizing them as they are will most likely give dots and small oxidized fragments, not large GO sheets. **[H]** Dots have uses of their own, but reinforcement needs sheets with high aspect ratio. So the hemp pathway puts a graphitization step and a measured gate before oxidation (Figure 5.1; Experiments B-07 and D-02).

Precedents show the chemistry itself transfers. Pine-leaf biochar made at 750 °C has been oxidized to GO by a modified Hummers method ([Springer review, 2024](#)). Coal char oxidized by a nitric-acid route and reduced at 2500 °C reached about 4000–5000 S/m, against about 2000 S/m for a modified-Hummers comparison ([US Patent 11,731,875](#)). **[R]**

6.4 Safety of permanganate oxidation

Potassium permanganate in sulfuric acid forms manganese heptoxide, which decomposes explosively above 55 °C or on contact with oxidizable organic matter ([Mn2O7 data](#)). **[E]** Reactor explosions from Hummers-type chemistry are recorded in the patent literature ([US Patent 11,021,370](#)).

Hemp carbon raises the stakes. It is more porous and may hold more residual volatiles than graphite, so heat release could be faster and less predictable. **[H]** Experiment D-02 therefore starts at 0.25 g, measures heat flow in a reaction calorimeter or with logged

thermocouples, adds permanganate in portions under an ice bath, and runs behind a blast shield.

6.5 Other oxidation routes

- **Partial ferrate substitution.** A NaNO₃-free method partly replaced KMnO₄ with K₂FeO₄ and used less sulfuric acid ([Scientific Reports 2016](#)). [R]
- **Nitric-acid oxidation** of coal and char, noted above. [R]
- **Electrochemical routes** give mildly oxidized graphene (C/O 17.2 in Chapter 3.3) rather than true GO, and need an electrode-form feed.

6.6 Reduction to rGO

Method	Conditions reported	Result reported	Notes
Hydriodic acid	55% HI, 100 °C, 1 h	C/O above 12; 29,800 S/m (PMC7948250)	Strongest common reductant; corrosive; iodine residue
Ascorbic acid, then heat	Chemical then thermal, in sequence	C/O 15.5; 1.97×10^4 S/m (Gazi University record)	Green; order of steps matters
Ascorbic acid alone	12 h	Above 1800 S/m (Nanoscale 2023)	Safe, slow
Hydrazine hydrate	Aqueous	C/O from 2.7 to 10.3 (PMC7948250)	Toxic; leaves nitrogen
Thiourea dioxide	Aqueous	C/O up to 16.0 (J. Mater. Chem. 2012)	Industrial reductant with established waste handling
Thermal	1100–2500 °C	Up to about 5000 S/m for coal-char rGO at 2500 °C	Also exfoliates; energy-intensive

Conductivity depends heavily on sample form (film, paper or powder compact), so compare methods only within one laboratory and one measurement geometry. [E] For composites, two practical options stand out: ascorbic acid for a green, separate reduction, and partial thermal reduction during cure or hot-pressing. The second is untested for hemp-derived GO (Experiment D-05). [H]

6.7 GO and rGO in composite processing

- **GO** disperses in water and polar solvents, and its epoxide and hydroxyl groups react with amines ([Nanoscale 2016 on GO reactivity](#)). That makes it a natural partner for amine-cured epoxies. **[R]**
- **rGO** conducts but restacks and disperses poorly unless functionalized or reduced in place.
- **Residues** of manganese, sulfur, potassium and iodine can change cure kinetics and long-term ageing. Experiment D-03 sets limits by ICP-OES before any GO enters a composite. **[H]**

Chapter 7 — Properties of the standalone materials

Single-sheet measurements set a ceiling that bulk powders never reach. Hemp nanosheets sit at the porous, moderately conductive end of the family, not the stiff, crystalline end, and their intrinsic stiffness has never been measured.

7.1 Single-sheet ceilings

Material	Young's modulus	Strength	Thermal conductivity	Source
Pristine monolayer graphene	1.0 TPa	130 GPa, defect-free	4840–5300 W/m·K, suspended, near room temperature	Lee et al., Science 2008 ; Balandin et al., Nano Letters 2008
Monolayer GO	207.6 ± 23.4 GPa, taking 0.7 nm thickness	—	—	Suk et al., ACS Nano 2010
Chemically reduced GO monolayer	0.25 TPa	—	—	Gómez-Navarro et al., Nano Letters 2008

All **[E]** as measurements, with two cautions. Real sheets carry defects, and one review argues 130 GPa overstates practical strength ([arXiv:1903.07695](#)). In reduced GO, conductivity scales inversely with modulus, because oxygen bridges stiffen bonds while

blocking charge transport ([Gómez-Navarro et al.](#)). Bulk assemblies fall far short: graphene laminates have been measured at 40–90 W/m·K ([Balandin group summary](#)).

7.2 Bulk materials as a lab will actually receive or make them

Material	Layers or thickness	Surface area	Conductivity (form)	Oxygen	Source
Commercial "graphene" powders, 2018 survey	Mostly graphite microplatelets	—	—	—	Kauling et al.
Shear-exfoliated few-layer graphene	Under 10 layers; 300–800 nm across	—	—	Unoxidized	Paton et al.
Electrochemically exfoliated graphene	Over 85% at 3 layers or fewer; up to 44 μm	—	11 Ω/sq (painted film)	C/O 17.2	Parvez et al.
Ball-milled, edge-carboxylated	5 layers or fewer	—	121,400 S/m (1214 S/cm, decarboxylated film)	Edge-only	Jeon et al.
Flash graphene	Turbostratic stacks	—	—	Low	Luong et al.
HCNS, CNS-800	10–30 nm	1505 m^2/g	226 S/m	4.19 at%	Chapter 5 table
HCNS, CNS-750	40–70 nm	2287 m^2/g	211 S/m	5.6 at%	Chapter 5 table
GO, modified Hummers	Usually monolayer	—	Insulating	About 30% (one report)	ResearchGate report
rGO, HI-reduced	1 to few	—	29,800 S/m	C/O above 12	PMC7948250

Read the conductivity column with care. Hemp nanosheets at 211–226 S/m sit about 130 times below HI-reduced rGO and about 500 times below decarboxylated ball-milled films, but sample forms differ and the hemp measurement method is not stated in the sources reviewed. Experiment C-05 re-measures every material as a powder compact at a fixed pressure so the numbers can be compared. **[R]** for each row; the cross-row comparison is only indicative.

7.3 What these numbers mean for composites

- **Stiffness.** Reinforcement scales with in-plane modulus, aspect ratio and interface quality (Chapter 11). No in-plane modulus has ever been measured for HCNS. A porous, partly graphitic sheet is very likely far below the 0.25 TPa of reduced GO. [H] Experiment C-08 measures it.
- **Heat.** Porous, disordered carbon conducts heat poorly. Ungraphitized HCNS should not be sold as thermal fillers. [H]
- **Processing.** Areas of 1500–2300 m²/g will soak up resin and raise viscosity, capping the practical loading. [H] Experiment E-04 measures the rheology.
- **Density.** Converting weight fraction to volume fraction, which every model needs, requires skeletal density. Graphite is 2.26 g/cm³; HCNS has not been measured (Experiment C-06).
- **Chemistry.** About 4–6 at% oxygen is already present on HCNS, comparable to the commercial nanoplatelet the patent used as reference. That may be enough to bond with amine hardeners without extra functionalization. [H]

Chapter 8 — Polymer matrix composites

In epoxies, well-dispersed graphene platelets give their largest gains near 0.1 wt%, mostly in fracture toughness. Above that, agglomeration usually erodes the benefit unless the interface is chemically engineered.

8.1 Benchmarks in epoxy

Filler	Loading	Reported change vs neat epoxy	Source
Graphene platelets	0.1 wt%	Modulus +31%, tensile strength +40%, mode I toughness +53%; beat single- and multi-walled nanotubes	Rafiee et al., ACS Nano 2009

Filler	Loading	Reported change vs neat epoxy	Source
Multilayer graphene	0.1 wt%	Young's modulus +26%; K _{IC} from 0.85 to 1.2 MPa·m ^{1/2} (+41%)	Atif, Shyha, Inam
GNP, 25 µm by 6–10 nm, three-roll milled	1.0 wt%	Conductivity up nine orders, thermal conductivity doubled, toughness +1/3; tensile and flexural nearly unchanged	Imran and Shivakumar
Amine-functionalized GNP	0.5 wt%	Toughness +82%, strength +38%, modulus +15%	Supplier summary of Ahmadi-Moghadam et al. 2014
GO grafted with poly(oxypropylene)diamine	0.3 wt%	Tensile +20%, flexural +40%, elongation +90%, toughness +145%	Zhou et al., RSC Advances 2015
Diethylenetriamine-functionalized GO	0.3 wt%	Tensile +67%, flexural +51%, impact +152%; Tg 88.4 to 97.3 °C	Paraskar et al., Iran. Polym. J. 2019
Coal-derived flash graphene	20–50 wt%	Modulus +92% (1:2); hardness +140%, toughness +496% (1:3)	Advincula et al. 2023

All [R]. The pattern across many studies is [E]: small loadings, good dispersion and a reactive interface matter more than filler quantity. A review of graphene-epoxy work found toughness gains up to 131% and traced the usual decline at higher loadings to worsening dispersion ([Polymers 2016, 8:281](#)).

8.2 How sheets toughen epoxy

Rafiee and colleagues attributed graphene's advantage over nanotubes to high surface area, a wrinkled surface that interlocks with the matrix, and planar geometry ([PubMed abstract](#)). The usual mechanisms are crack deflection, crack pinning, platelet pull-out and

plastic void growth. Fractography (Experiment F-08) identifies which ones operate in each formulation.

8.3 Dispersion toolbox

- **Three-roll milling** is repeatable and scalable for viscous epoxies. In one comparison it gave 1.8×10^{-3} S/m at 1.0 wt%, three orders above sonication with high-shear mixing ([TUHH](#)). [R]
- **Probe sonication** breaks agglomerates but also breaks sheets; keep it short and cooled.
- **Solvent-assisted mixing** with acetone or ethanol, then evaporation, helps GO and functionalized fillers; residual solvent must be removed and verified by TGA.
- **Chemical coupling** with amines or silanes improves both dispersion and load transfer ([Univ. Oklahoma thesis using ASTM D5045](#)). [R]

8.4 Electrical percolation

Chemically modified graphene in polystyrene percolated at about 0.1 vol% and reached about 0.1 S/m at 1 vol% ([Stankovich et al., Nature 2006, 442:282](#)). [E] Randomly oriented disks with aspect ratio 1000 have a geometric threshold near 0.1 vol% ([US Patent 7,914,844](#)). Commercial GNP in epoxy percolated at 1.0 wt% in the three-roll-mill study above.

Hemp nanosheets conduct far less than rGO and their aspect ratio is unknown, so their threshold is likely higher. Their interconnected morphology might partly offset that. [H] Experiment F-05 measures it.

8.5 Thermoplastics

Melt-blending alone dispersed exfoliated graphene in PET: at 0.07 wt%, modulus rose more than 10% and tensile strength more than 40% ([Istrate et al., Carbon 2014, 78:243](#)). [R] Twin-screw compounding into polypropylene, polyamide, PLA or PHA is therefore realistic for HCNS once a dry, free-flowing powder exists. Moisture and residual potassium must be controlled to avoid hydrolysis of polyesters. [H]

8.6 Hierarchical hemp composites

Hemp-fibre/epoxy composites already benefit from nanofillers. Adding 1.5 wt% alumina raised tensile strength from 52 to 64 MPa and flexural strength from 78 to 93 MPa ([J. Met. Mater. Miner.](#)), and graphene at 0.3 wt% has been used in hemp/epoxy nano-

biocomposites ([SPE Polymers 2025 review](#)). [R]

The hemp-native version would use hemp fibre as the primary reinforcement and HCNS to stiffen and toughen the matrix and the fibre-matrix interphase. Everything in it would come from one plant. [H] Experiment F-09 tests it.

8.7 What to expect from HCNS in epoxy

- **Toughness gains are the likeliest win**, because crack deflection and pinning need sheets, not perfect crystals. [H]
- **Stiffness gains will be smaller** than with GNP, because the sheets are porous and less ordered. [H]
- **Viscosity will limit loading** sooner than with GNP, because of the very high surface area. [H]
- **Oxygen at 4–6 at%** may bond to amine hardeners without extra chemistry. [H]

Chapter 15 converts these into design targets with go/no-go thresholds. [T]

Chapter 9 — Bio-based epoxies, vitrimers and Hempoxies

A hemp-oil epoxy vitrimer reinforced with hemp nanosheets is chemically plausible and could be made entirely from one plant. Every performance claim for it is still a TRL 1–2 hypothesis. The nearest published analogues are graphene oxide in petroleum-based vitrimers and waste-derived nanosheets in a thiol-epoxy vitrimer.

9.1 Bio-based epoxy matrices

- **Epoxidized hemp oil (EHO)**. Blended with a DGEBA epoxy cured by triethylenetetramine and isophorone diamine, EHO performed marginally better than epoxidized soybean oil, but both were limited to under 30% bioresin, and the all-synthetic control remained strongest ([Manthey et al., J. Reinf. Plast. Compos. 2013, 32:1444](#)). [R]
- **Acrylated EHO** with jute fibre bonded better to the fibres than vinyl ester resin, at the cost of lower Tg and crosslink density ([Manthey et al., J. Compos. Mater.](#)). [R]
- **Commercial hemp-seed-oil epoxy**. ZILA BioWorks makes a hemp-seed-oil bio-epoxy sold as Hempoxy Resin and reports roughly double the bio-content of BPA-based bio-epoxies (about 30%), with potential to halve embodied carbon ([ISC3 profile; 425 Business](#)). Its US EPA-funded development set a target above 50% bio-based content and screened about 45 formulations ([EPA final report](#)). [R], company-reported.

The common weakness of oil-based epoxies is the long, flexible fatty chain, which lowers T_g and modulus. Stiff nanosheets are a logical way to compensate. [H]

9.2 Vitrimers in brief

In 2011 Leibler and colleagues added a transesterification catalyst to epoxy/acid and epoxy/anhydride networks. The networks stayed permanently crosslinked but their viscosity fell gradually on heating, like silica glass, so they were named vitrimers ([Chemical Science minireview](#)). [E] Their original system used zinc acetate at about 5 mol% ([Polymer Innovation Blog](#)). The temperature at which exchange freezes is the topology-freezing temperature, T_v ([Polymer Innovation Blog](#)). Other exchange chemistries include disulfide metathesis, imine, silyl ether and acetal exchange ([IISc thesis record](#)).

9.3 Carbon nanomaterials in vitrimers

System	Key result	Source
Epoxy vitrimer (disulfide exchange) + GO	0.2 wt% GO optimal; 89% flexural strength kept after remoulding; 48 µm scratch healed in 60 min; glass fibres recovered by thiol degradation	Sharma et al., J. Reinf. Plast. Compos. 2024
Vitrimeric thiol-epoxy + tyre-waste carbon nanosheets	Flexible and strong, tunable conductivity, self-healing and shape memory	Polymers, September 2026
Epoxy vitrimer + biomass activated carbon	Self-healing at 70 °C in 5 min versus 80 °C for the unfilled vitrimer	UPES PhD thesis
Soft epoxy vitrimer + 5 wt% magnetic nanoparticles	Induction heating to 240–250 °C in 5–10 min healed scratches; oven heating did not	Frontiers in Materials 2025

All [R]. A 2026 review of epoxy-vitrimer nanocomposites covers graphene, silica and magnetite fillers adding conductivity, thermal conductivity, healing and stimulus response ([Nano Research 2026](#)).

Two effects compete in any filled vitrimer. Fillers can hinder chain mobility and slow stress relaxation. Fillers with surface hydroxyl or carboxyl groups, such as GO and HCNS, may instead join the exchange reactions and speed it up. **[H]** Experiment G-02 measures which effect wins for hemp nanosheets.

9.4 Hempoxies: status, hypotheses and targets

Status. Hempoxies is Landry Industries' research initiative for bio-based vitrimer composites built from hemp inputs. It is at TRL 1–2, and every figure attached to it is a projected design target, not a measured result.

Working matrix. The program's current formulation is epoxidized hemp oil cured with maleinized hemp oil at 1:1 stoichiometry, as set out in its September 2026 technical report. Anhydride-cured epoxy networks are the chemical class Leibler made into vitrimers by adding a transesterification catalyst, so the formulation has a credible route to vitrimer behaviour. **[H]** for this specific system. Commercial hemp-seed-oil epoxies, such as ZILA's Hempoxy Resin, are a natural benchmark and a possible partner matrix, subject to agreement.

Reinforcement concept. HCNS (the author's "Mitlinite") as the nanophase in the matrix, and hemp fibre as the microphase reinforcement.

ID	Hypothesis	Design target	Tested by
H9.1	HCNS at 1 wt% or less raises the modulus and Tg of the EHO/MHO network	Storage modulus at 25 °C up 20% or more; Tg up 5 °C or more	F-01, F-04
H9.2	Surface oxygen on HCNS joins or catalyses transesterification	Stress-relaxation time at 160 °C cut by 25% or more	G-02
H9.3	Above percolation, HCNS allow Joule-heated healing and reshaping	Scratch healed at 30 V or less within 10 min	G-04
H9.4	Filled vitrimer survives reprocessing	80% or more of tensile strength kept after 3 cycles	G-03
H9.5	The matrix can be dissolved chemically and the HCNS recovered	90% or more of HCNS mass recovered; Raman unchanged	G-05

All targets are **[T]**: chosen to be meaningful and testable, not predicted.

9.5 Matrices for the experimental program

Code	Matrix	Role
M1	DGEBA cured with an amine hardener	Conventional control
M2	DGEBA cured with anhydride plus zinc acetate	Petroleum-based vitrimer control
M3	EHO/MHO 1:1, with and without transesterification catalyst	Hempoxies working matrix
M4	Commercial hemp-seed-oil bio-epoxy	Bio-based commercial benchmark
M5	Disulfide-exchange epoxy vitrimer	Benchmark matching the published GO-vitrimer study

Chapter 10 — Cementitious, ceramic, metal, coating and energy composites

Outside polymers, graphene materials have their strongest record in cement, at a few hundredths of a percent of GO, and in toughened ceramics. In metals and protective coatings the chemistry pushes back, through brittle carbides and galvanic corrosion.

10.1 Cement and concrete

Filler	Dose (by mass of cement)	Reported change	Source
GO in Portland cement paste	0.05%	Compressive +15–33%, flexural +41–59%; more tortuous cracks, less sudden failure	Pan et al., Cem. Concr. Compos. 2015
GO in cement paste	0.04%	Compressive +46.6%, flexural +75% at 28 days; lower permeability; higher viscosity, shorter setting	Australian J. Civil Eng.

Filler	Dose (by mass of cement)	Reported change	Source
GO in mortar with recycled fine aggregate	0.20%	Flexural +22–41.3%, compressive about +16%; workability down 7.5–18.8%	PMC5746897
GNP in cement	0.05%	Flexural +27.4% at 7 days and +25.2% at 28 days; compressive about +4%; finer pores	Dalian Univ. of Technology record

All **[R]**; the direction of the effect is consistent enough across groups to treat as **[E]**. One proposed mechanism is covalent bonding between GO carboxyl groups and calcium silicate hydrate gel ([Bol. Soc. Esp. Cerám. Vidrio 2024](#)). Workability loss is the recurring penalty and must be offset with superplasticizer.

Hemp angle. Lime–hemp binders are an established bio-based building material. Whether HCNS or hemp-derived GO strengthen lime–hemp or Portland binders the way graphite GO does is untested (Experiments H-01 and H-02). **[H]**

10.2 Ceramic matrix composites

About 1.5 vol% graphene platelets in silicon nitride, densified by spark plasma sintering near 1650 °C, raised fracture toughness from about 2.8 to 6.6 MPa·m^{1/2} (+235%); the platelets wrapped and anchored around grains ([Walker et al., ACS Nano 2011, via US Patent 8,962,504](#)). **[R]** Silicon carbide with graphene reached about +162% toughness ([CSIC repository](#)), and alumina/graphene composites have reached 5709 S/m ([Osendi, Graphene 2013 abstract](#)).

HCNS carry oxygen and fine pores, so they may react with oxide powders during sintering. They are a low-priority, high-risk candidate here (Experiment H-03). **[H]**

10.3 Metal matrix composites

Aluminium with 0.1–1 wt% GNP, made by milling, pressing at 450 MPa and sintering at 590 °C, formed aluminium carbide; at 1 wt% hardness reached about 1.31 GPa and compressive strength rose from about 70 to 162 MPa ([Süzer et al., Diamond Relat. Mater.](#)

[2024](#)). [R] Aluminium carbide is brittle and unstable in moisture, and its net effect on strength is still argued ([Metals 2022](#)).

Defective, high-area carbon should form carbide even faster. HCNS are a poor first choice for aluminium; copper, which forms no stable carbide, is the better screening matrix (Experiment H-04). [H]

10.4 Coatings and barrier films

Graphene on metals protects briefly, then accelerates corrosion: it is conductive and more noble than the metal, so any crack sets up a galvanic cell ([Schriver et al., ACS Nano 2013, listed at Peeref; R&D World](#)). Insulating boron nitride performed better over 160 days for that reason ([J. Mater. Chem. A 2016](#)). [E]

The rule for this program: in anticorrosion coatings, keep conductive carbon (including HCNS) below percolation, isolate it in a polymer, or pair it with sacrificial zinc. Experiment H-05 tests HCNS in an epoxy primer by salt-spray exposure. [H]

10.5 Energy composites

Energy storage is where hemp nanosheets started (Chapter 5), and peat-moss nanosheet frameworks serve sodium-ion anodes ([Ding et al., ACS Nano 2013](#)). One multifunctional idea links this to composites: a hemp-fibre/Hempoxy laminate whose matrix holds HCNS could store charge as a structural supercapacitor. [H] It is listed as an exploratory extension, not a core experiment.

10.6 Priority across matrix families

Matrix family	Evidence for graphene materials	Fit for HCNS	Priority here
Epoxy and bio-epoxy, including vitrimers	Strong	Good	1
Cement and lime-hemp binders	Strong for GO	Plausible	2
Thermoplastics	Moderate	Plausible once powder is dry	3
Protective coatings	Mixed; galvanic risk	Conditional	4

Matrix family	Evidence for graphene materials	Fit for HCNS	Priority here
Ceramics	Strong for platelets	Risky (reactivity)	5
Aluminium	Mixed (carbides)	Poor	6

Chapter 11 — Modelling nanosheet composites

Simple micromechanics says stiffness gains depend far more on aspect ratio and effective in-plane modulus than on loading. For hemp sheets, whose modulus has never been measured, the model's job is to bound expectations and tell the lab exactly what to measure.

11.1 Weight fraction to volume fraction

Every model works in volume fraction:

$$V_f = \frac{w_f/\rho_f}{w_f/\rho_f + (1-w_f)/\rho_m}$$

For example, 6 wt% GNP in an aerospace epoxy corresponded to 3.7 vol% ([King et al., J. Appl. Polym. Sci. 2013](#)). For HCNS there is a catch: resin can fill the pores, so the sheet's skeletal density (helium pycnometry) and its envelope density both matter. Experiment C-06 measures both. [H]

11.2 Halpin–Tsai for randomly oriented platelets

For platelets of diameter D and thickness h , with filler modulus E_f and matrix modulus E_m :

$$\xi = \frac{2D}{3h}, \quad \eta_L = \frac{E_f/E_m - 1}{E_f/E_m + \xi}, \quad \eta_T = \frac{E_f/E_m - 1}{E_f/E_m + 2}$$

$$\frac{E_L}{E_m} = \frac{1 + \xi \eta_L V_f}{1 - \eta_L V_f}, \quad \frac{E_T}{E_m} = \frac{1 + 2 \eta_T V_f}{1 - \eta_T V_f}, \quad E_c = 0.49 E_L + 0.51 E_T$$

The 3D-random combination and the shape factor are Van Es's; with them Halpin–Tsai stays within a few percent of Mori–Tanaka and of finite-element results up to about 0.5 vol%. From about 1 vol%, platelets start forming clusters and the analytical models

overpredict ([Tarasovs and Aniskevich, Mech. Mater. 2022](#)). [E] In practice, 2D- and 3D-random Halpin-Tsai variants predicted measured GNP/epoxy moduli well, from 2.72 GPa neat to 3.35 GPa at 6 wt% ([King et al., J. Compos. Mater. 2015](#)).

11.3 What the model says about hemp nanosheets

Predicted stiffness gain over the matrix, using the equations above with $E_m = 3.0$ GPa (a typical cured epoxy), computed for this book:

Filler modulus E_f	Aspect ratio 30	100	300	1000
At 0.5 vol%				
20 GPa	+1.6%	+1.8%	+1.9%	+1.9%
100 GPa	+3.8%	+6.1%	+7.5%	+8.3%
250 GPa	+4.9%	+9.9%	+15.1%	+18.7%
1000 GPa	+5.6%	+14.6%	+31.5%	+55.2%
At 1.0 vol%				
20 GPa	+3.2%	+3.6%	+3.7%	+3.8%
100 GPa	+7.7%	+12.2%	+15.1%	+16.5%
250 GPa	+9.8%	+19.8%	+30.2%	+37.4%
1000 GPa	+11.3%	+29.3%	+63.3%	+110.6%

Three conclusions follow. **[Model output]**

1. If ungraphitized HCNS behave like a porous carbon with an effective modulus near 20 GPa, no aspect ratio rescues stiffness: gains stay under 4% at 1 vol%. Their value would have to come from toughness, conductivity or interfacial effects instead.
2. If graphitization lifts the effective modulus toward 250 GPa (reduced-GO territory, Chapter 7.1) and sheets reach aspect ratio 300, about +30% at 1 vol% becomes possible.
3. Measuring E_f (Experiment C-08) and lateral size (C-03) is therefore not optional. Together they decide which of these rows the hemp material lives in.

Two more effects push real values below the table. Multilayer flakes transfer stress poorly between their own layers, so their effective modulus falls with thickness; and in monolayer studies, flakes needed to be several micrometres long for good reinforcement ([Kinloch et al., ICCM 2015](#)). Molecular dynamics puts the critical length near 500 nm for graphene and 300 nm for GO, and shows that waviness sharply reduces reinforcement ([CORE record](#)). [R]

11.4 Stress transfer and critical length

For fibres, the Kelly–Tyson relation links critical length to filler strength σ_f , thickness d and interfacial shear strength τ_i ; the same logic applies to platelets through shear-lag theory:

$$l_c = \frac{\sigma_f d}{\tau_i}$$

[E] The Hemproxies technical work already uses Kelly–Tyson for hemp fibres and Halpin–Tsai for the matrix; the same pair extends naturally to the HCNS-in-matrix level.

The tool for measuring stress transfer is Raman spectroscopy. Under strain, the graphene 2D (G') band shifts by more than -50 cm^{-1} per % strain, consistent with a modulus above 1 TPa, and a universal calibration converts band-shift rate into effective modulus ([Gong et al., arXiv:1007.1953](#)). [E] Experiment F-06 applies this to HCNS in epoxy.

11.5 Electrical percolation

Above the threshold Φ_c , composite conductivity follows a power law in filler volume fraction ([US Patent 7,914,844](#)):

$$\sigma_c = \sigma_f \left[\frac{\Phi - \Phi_c}{1 - \Phi_c} \right]^t$$

Randomly oriented disks of aspect ratio 1000 have a geometric threshold near 0.1 vol%. Experiment F-05 fits Φ_c and t for each filler; a high threshold with a low exponent would signal agglomeration.

11.6 Two-scale (hierarchical) models

For hemp-fibre laminates with an HCNS-modified matrix, model in two steps. First compute the nanosheet-modified matrix with Halpin–Tsai; then feed that matrix into a short-fibre model or Kelly–Tyson for the hemp fibres. Calibrate each step against its own experiment (F-01 for step one, F-09 for step two) rather than fitting both to the laminate alone.

11.7 Inputs the models need from the lab

Input	Symbol	How to measure	Experiment
Matrix modulus	E_m	Tensile test of neat resin	F-01
Matrix and filler density	ρ_m , ρ_f	Archimedes; helium pycnometry	C-06
Lateral size distribution	D	SEM or AFM on dispersed flakes, 200 or more counted	C-03
Thickness distribution	h	AFM	C-02
Effective filler modulus	E_f	Raman band-shift in a strained beam; AFM indentation	C-08, F-06
Interfacial shear strength	τ_i	Raman strain mapping along flakes	F-06
Dispersion and clustering	—	TEM, micro-CT	F-07
Orientation	—	SEM of polished sections; X-ray scattering	F-07

Chapter 12 — Characterization of standalone nanosheets, GO and rGO

Run every batch through the same core panel, follow ISO/TS 21356-1 for structure and ISO/TS 23359 for chemistry, and report distributions rather than single numbers. Without that discipline, no hemp result can be compared with a graphite result.

12.1 The standards spine

Document	Covers	Source
ISO/TS 80004-13:2024	Vocabulary	SCC record

Document	Covers	Source
ISO/TS 21356-1:2021	Structure of graphene, bilayer graphene and GNP from powders and dispersions: layers or thickness, lateral size, disorder, layer alignment, specific surface area; method sequence after isolating flakes on a substrate	SCC record
ISO/TS 23359:2025	Chemistry of graphene-related 2D materials from powders and dispersions: elemental composition, O/C ratio, trace metals, functional groups, by XPS, TGA, ICP-MS and FTIR	SFS record
ISO 9277:2022	BET specific surface area by gas adsorption	StatNano record
VAMAS TWA 41	Interlaboratory comparisons of SEM and AFM lateral size and thickness for GNP, led by NPL	VAMAS project sheet
EMPIR GRACE guide	Electrical measurement of graphene films	arXiv:2007.14047

12.2 The core panel

Technique	What it tells you	Report	Hemp-specific note
Raman (532 and 633 nm)	Disorder, crystallite size, layer stacking	I _D /I _G , FWHM of G and D, 2D shape; map 100 or more spectra	Fluorescence from residual organics; use FWHM(G) as well as I _D /I _G (see 12.3)
X-ray diffraction	Interlayer spacing and stacking order	d002, Lc, La, degree of graphitization g	Broad, asymmetric (002) expected before graphitization
XPS	Surface chemistry	C/O, N, C1s components (sp ² , sp ³ , C-O, C=O, O-C=O)	Track nitrogen (about 1-1.5 at% in HCNS)

Technique	What it tells you	Report	Hemp-specific note
TGA, air and nitrogen	Oxidation onset, volatiles, ash	Onset and peak temperatures; residue at 900 °C	Residue reveals potassium and silicon carry-over
ICP-OES or ICP-MS after digestion	Trace metals	K, Ca, Si, Fe, Mn, S, I	Mandatory after KOH, iron catalysis or Hummers
CHNS-O combustion	Bulk composition	wt% C, H, N, S, O	Compare with XPS to see surface versus bulk
Gas sorption (N ₂ at 77 K, CO ₂ at 273 K)	Area and pore size	BET per ISO 9277; DFT pore-size distribution; micropore fraction	CO ₂ needed for ultramicropores
Helium pycnometry	Skeletal density	g/cm ³	Feeds weight-to-volume conversion (Chapter 11.1)
SEM with EDS	Morphology, lateral size, residues	Lateral-size histogram, 200 or more flakes	Sheets may stay interconnected; report aggregate size too
AFM	Thickness	Thickness histogram, 100 or more flakes	Porosity can make apparent thickness ambiguous
TEM, HRTEM, SAED, EELS	Layer number, stacking, sp ² fraction	Layer counts at edges; diffraction rings or spots	Confirms partial order claimed by the patent
Powder conductivity	Electrical quality	Four-probe on a compact at 10, 50 and 100 MPa, with compact density	The common yardstick for all materials (C-05)
Zeta potential, UV-vis stability	Dispersion behaviour	ζ versus pH; absorbance over 7 days	Essential for GO and cement work

12.3 Reading Raman and XRD correctly

Crystallite size from Raman. For nanocrystalline graphite, in-plane crystallite size follows the Cançado relation, with λ the laser wavelength in nm ([formula as used in arXiv:1208.2439](#)):

$$L_a \text{ (nm)} = 2.4 \times 10^{-10} \lambda^4 \left(\frac{I_D}{I_G} \right)^{-1}$$

[E] The relation fails for very disordered carbon. Below roughly 2 nm the trend reverses and I_D/I_G falls as order is lost ([Ferrari and Robertson, Phys. Rev. B 2000](#)). Hemp carbons may sit near that boundary, so a single I_D/I_G value is ambiguous. Report the G-band width and use two laser wavelengths to tell the regimes apart.

Degree of graphitization from XRD. With d_{002} in nm, taking 0.3440 nm for disordered carbon and 0.3354 nm for graphite ([Malvern Panalytical application note](#)):

$$g = \frac{0.3440 - d_{002}}{0.3440 - 0.3354}$$

Stack height L_c comes from the (002) peak width with the Scherrer constant 0.9, and L_a from the (100) or (110) peak with 1.84. **[E]** A value of g only means something when the (002) peak is sharp enough to fit; for turbostratic hemp carbon, report the fit residual too.

After reduction. Chemical reduction of GO often raises I_D/I_G , because many small new sp^2 domains appear. That is not extra damage. **[E]** Pair Raman with XPS C/O and conductivity before drawing conclusions.

12.4 Sampling and statistics

- Three independent batches for every material, each measured in triplicate where the method allows.
- Report mean, standard deviation and the full distribution for size, thickness and Raman metrics.
- A commercial GNP and a commercial GO, characterized in-house on arrival, serve as running controls. Section 1.1 explains why a supplier's data sheet is not enough.
- Store raw spectra, images and isotherms with the data sheet in Appendix B. Re-analysis must be possible without re-measuring.

Chapter 13 — Characterization of composites

A composite result is only credible when it travels with evidence of dispersion, degree of cure and void content. Every mechanical number in this program is reported alongside a micrograph, a Tg and a density.

13.1 Process quality before mechanics

- **Dispersion.** Optical microscopy of the uncured resin for agglomerates over 10 μm ; TEM of ultramicrotomed sections; micro-CT for agglomerates and voids.
- **Rheology.** Viscosity against shear rate and loading on a parallel-plate rheometer. The loading where a low-shear yield stress appears marks network formation and usually the practical processing limit.
- **Cure state.** DSC on cured specimens; target 95% conversion or more. Fillers can change cure, especially GO, whose epoxides and hydroxyls react with amines (Chapter 6.7).
- **Density and voids.** Archimedes density compared with the theoretical value from the rule of mixtures. **[T]** Under 1% voids for structural specimens.

13.2 Mechanical tests

Property	Standard	Specimen	Why it matters here
Tensile, neat and filled resin	ASTM D638 or ISO 527	Dog-bone	Modulus input for Chapter 11 models
Flexural	ASTM D790	Bar	Used in the hemp-fibre benchmarks of Chapter 8 (J. Met. Mater. Miner.)
Plane-strain fracture toughness, K _{IC} and G _{IC}	ASTM D5045	Single-edge-notch bend	Toughness is the likeliest HCNS benefit (Univ. Oklahoma thesis)
Laminate tensile	ASTM D3039	Straight-sided coupon	Hemp-fibre laminates (F-09)
Interlaminar shear	ASTM D2344	Short beam	Matrix and interface effects in laminates (ASTM D2344 text)

Property	Standard	Specimen	Why it matters here
Mode I interlaminar fracture	ASTM D5528	Double cantilever beam	Delamination resistance of nano-modified matrices (ANSI listing)
Impact	ISO 179 (Charpy) or ASTM D256 (Izod)	Notched bar	Energy absorption
Fatigue crack growth	ASTM E647	Compact tension	Graphene's reported fatigue advantage (Chapter 8.1)
Hardness, local modulus	Shore D; nanoindentation	Polished block	Maps local stiffness around agglomerates

Use at least five valid specimens per condition, as most of these standards require, and report Weibull parameters for strength.

13.3 Thermal and thermomechanical tests

Property	Standard or method	Note
T _g and storage modulus	DMA per ASTM D7028 (ASTM store)	Rubbery-plateau modulus gives crosslink density
T _g by calorimetry	DSC per ASTM E1356	Cross-check against DMA
Thermal stability	TGA in air and nitrogen	Onset of degradation
Thermal conductivity	Laser-flash diffusivity (ASTM E1461) × heat capacity × density	Only worth running for graphitized fillers
Thermal expansion	TMA	Dimensional stability

13.4 Electrical tests

- Volume resistivity of insulating samples by ASTM D257; four-probe for samples above percolation.
- Percolation curve from at least eight loadings, fitted with the power law of Chapter 11.5.
- Optional: shielding effectiveness for EMI applications.

13.5 Interface diagnostics

- **Raman strain mapping** of embedded flakes under load gives effective filler modulus and interfacial shear strength (Chapter 11.4; Experiment F-06).
- **Fractography** by SEM identifies crack deflection, pinning, pull-out and void growth (F-08). The person scoring the images should not know which formulation they are looking at.
- **XPS of fracture surfaces** shows whether cracks ran through the matrix or along the filler surface.

13.6 Durability tests

Exposure	Standard	Applies to
Moisture uptake and diffusivity	ASTM D5229 (standard record)	All polymer composites; critical for oil-based epoxies
UV and condensation	ASTM G154	Outdoor bio-composites
Salt spray	ASTM B117	Coatings (H-05)
Freeze-thaw and thermal cycling	Program protocol, -30 to +60 °C	Atlantic Canadian service conditions
Creep	Tensile creep at service temperature	Essential for vitrimers, which can creep

13.7 Vitrimer-specific tests

1. **Stress relaxation** at four or more temperatures; fit relaxation times to an Arrhenius law to get the activation energy and extrapolate T_v .
2. **Swelling** in an inert solvent: a vitrimer swells but does not dissolve ([Chemical Science minireview](#)). Dissolution means the network has broken.
3. **Reprocessing**: grind, hot-press and retest, for three cycles; report property retention.
4. **Self-healing**: cut a standard scratch, heal, and measure closure by optical profilometry.
5. **Chemical recycling**: dissolve the matrix by a chemistry suited to its exchange bonds and recover the filler; re-characterize the filler with the Chapter 12 panel.

13.8 Cementitious tests

Property	Standard	Note
Compressive strength	ASTM C109 cubes (ANSI listing) or ASTM C349 on broken prisms (ASTM store)	7 and 28 days
Flexural strength	ASTM C348 (Nimonik listing)	Prisms
Workability	Flow table	Watch the loss reported for GO (Chapter 10.1)
Pore structure	Mercury intrusion porosimetry	Pore refinement mechanism
Hydration	Isothermal calorimetry; TGA for portlandite	Does the filler change hydration?

13.9 Statistics and pre-registration

Write each experiment's hypothesis, primary outcome and go/no-go threshold into the lab record before testing. Analyse with ANOVA or a mixed model with batch as a random effect, and report effect sizes with 95% confidence intervals, not only p-values. A single best specimen is never a result.

Chapter 14 — Knowledge-gap registry

Twenty-two gaps stand between the hemp nanosheet literature and a defensible composite product. Nine are priority 1: if any of them closes badly, the hemp case weakens sharply, so they are scheduled first.

Priority: **P1** decides whether the hemp route is viable; **P2** decides how good it can be; **P3** extends it to more markets.

ID	Gap	Why it matters	Evidence today	Closed by	Priority
G-01	How fast from Canadian grain-crop straw behaves in the Mitlin route, and how much it varies	Over 80% of Canadian hemp area is grain (Ch. 2.2)	None	A-01, A-06	P1

ID	Gap	Why it matters	Evidence today	Closed by	Priority
G-02	Unreported process parameters: acid strength, liquid ratio, activation hold time, yield	No one can reproduce or cost the process without them	Partial (Ch. 5.1)	A-02, A-03, A-06	P1
G-03	Whether hemp bast is uniquely suited, compared with flax, kenaf, jute or hurd	Decides whether "hemp" is a technical or only a marketing claim	None	A-04	P2
G-04	Residual potassium and ash, and their effect on cure and ageing	Residues can upset cure, corrode metals and seed defects	None	A-05, C-07, E-05	P1
G-05	Whether HCNS can be graphitized while keeping sheet shape	Prerequisite for stiffness and for GO sheets rather than dots	None for hemp; [R] for sawdust and flash routes	B-01 to B-04, B-06, B-07	P1
G-06	Whether graphitized HCNS exfoliate into thinner sheets	Thinner sheets raise aspect ratio per gram	None	B-05, C-02	P2
G-07	Thickness, lateral size and aspect ratio distributions measured to ISO/TS 21356-1	Model inputs; the patents give ranges only	Partial	C-02, C-03	P1
G-08	Whether the very high area comes from pores inside the sheets	Governs resin uptake, viscosity and void risk	[H] (Ch. 1.4)	C-04	P2
G-09	The in-plane effective modulus of HCNS	Decides which row of Table 11.3 applies	None	C-08, F-06	P1
G-10	Comparable conductivity and density data across all materials	Fair comparison with GNP, GO and rGO	Inconsistent methods	C-05, C-06	P2
G-11	Whether oxidizing hemp carbon gives GO sheets or quantum dots; heat release and yield	The hemp-to-GO/rGO pathway hinges on it	None for HCNS; [R] coal gives dots	D-02, D-06	P1
G-12	Purity of hemp-derived GO and how it reduces	Residues and reducibility set composite performance	None	D-03, D-04, D-05	P2
G-13	Dispersion and rheology of HCNS in epoxies	Sets the processable loading	None	E-01, E-04, F-07	P1

ID	Gap	Why it matters	Evidence today	Closed by	Priority
G-14	Interface chemistry with amine and anhydride cures	Load transfer and toughening depend on it	None	E-02, E-03, E-05, F-06	P2
G-15	Mechanical and toughening performance against GNP and GO at equal loading	The headline comparison	None	F-01 to F-04, F-08	P2
G-16	Electrical percolation of HCNS composites	Antistatic, sensing and heating uses	None	F-05	P2
G-17	Performance of hemp-fibre laminates with HCNS-modified matrix	The all-hemp product concept	None	F-09	P2
G-18	How HCNS affect vitrimer relaxation, reprocessing, healing and recycling	The Hempoxies proposition (Ch. 9.4)	[R] for GO and tyre-waste sheets only	G-01 to G-05	P2
G-19	Behaviour in cement, lime-hemp, thermoplastics, coatings, ceramics and metals	Market breadth	[R] for GO and GNP only	H-01 to H-06	P3
G-20	Durability of filled bio-composites in Atlantic Canadian service conditions	Building and outdoor uses	None	F-10	P3
G-21	Worker exposure during handling, and particle release from finished parts	Licence to operate; NIOSH's preliminary graphene benchmark is under 10 $\mu\text{g}/\text{m}^3$ (Ch. 16.2)	None for HCNS	I-01, I-02	P1
G-22	Life-cycle impact and cost per unit of performance against graphite routes	The sustainability claim must be measured, not assumed	None for HCNS	I-03, I-04, I-05	P2

The nine P1 gaps (G-01, G-02, G-04, G-05, G-07, G-09, G-11, G-13 and the safety gap G-21) define the first phase of work in Chapter 16. Twenty-two gaps map to 58 experiments in Chapter 15.

Chapter 15 — The experimental program

Fifty-eight experiments in nine modules close the 22 gaps of Chapter 14. Each card states the gap it closes, a falsifiable hypothesis, materials, method, design, measurements and a go/no-go gate, so a lab can execute it without further design work.

15.0 How to read the cards, and the standard conditions

Every gate is a **[T] design target**: a threshold chosen to make the decision clear, not a prediction. Parameters marked *program default* are starting points that Module A refines; they are not taken from the literature unless cited.

SC-1 · Standard HCNS route (program default, after Chapter 5.1)

1. Cut bast fibre to 5–10 mm; dry at 105 °C to constant mass.
2. Hydrothermal step: fibre in 0.5 wt% H₂SO₄, 1:20 w/v, in a PTFE-lined steel autoclave filled to no more than two-thirds; 180 °C for 24 h; cool naturally.
3. Filter, wash to neutral pH, dry at 105 °C. This is the biochar.
4. Grind biochar with KOH at 1:1 by mass in a glovebox or dry room; load into a nickel boat.
5. Activate under argon at 200 mL/min: ramp 5 °C/min to 800 °C, hold 1 h, cool under argon.
6. Quench the cooled product slowly into water in a fume hood; wash with 1 M HCl, then deionized water to pH 7; dry at 105 °C for 12 h.

SC-2 · Core panel: every technique in Table 12.2, on three batches.

SC-3 · Statistics: five or more specimens per mechanical condition; mixed model with batch as a random effect; effect sizes with 95% confidence intervals; pre-registered gate (Chapter 13.9).

Materials codes: HCNS = hemp nanosheets by SC-1; G-HCNS = graphitized HCNS selected at B-07; H-GO and H-rGO = hemp-derived GO and rGO; GNP-R and GO-R = commercial reference materials from C-01; M1–M5 = matrices of Table 9.5.

Module A — Feedstock and HCNS synthesis

A-01 · Feedstock survey and baseline reproduction

Closes G-01 · **Priority** P1 · **Effort** about 6 weeks

Hypothesis. Bast from Canadian grain-crop straw yields CNS-800-type sheets within $\pm 20\%$ of the reported area (1505 m²/g) and inside the reported 10–30 nm thickness range.

Materials. Three bast sources: decorticated bast from grain-hemp straw (2025 or 2026 harvest), fibre-hemp bast, and a commercial technical hemp fibre. H₂SO₄, KOH (85% or better), argon, 1 M HCl.

Method.

1. Analyse each feedstock: cellulose, hemicellulose and lignin by standard wet chemistry; ash at 575 °C; K, Ca and Si by ICP-OES; moisture.
2. Run SC-1 on each source, three independent batches each, with the activation at 800 °C to target CNS-800.
3. Record mass yield after each step.

Measurements. SC-2 panel, plus mass yields.

Gate. At least one grain-straw source lands within $\pm 20\%$ of CNS-800 area and inside 10–30 nm median thickness. If none does, add a fibre pre-treatment work item before Module B.

A-02 · Hydrothermal-step design of experiments

Closes G-02 · **Priority** P1 · **Effort** about 8 weeks

Hypothesis. Acid strength and time, not temperature, control how far microfibrils separate, and a higher-yield setting exists that keeps sheet morphology.

Design. Central composite design over H₂SO₄ at 0, 0.25, 0.5 and 1.0 wt%; temperature 160, 180 and 200 °C; time 12, 24 and 36 h. About 20 runs, with three repeated centre points. Feedstock: best source from A-01.

Method. Each hydrothermal product is activated by the fixed SC-1 activation.

Measurements. Biochar yield and O/C; SEM of fibril separation; final sheet thickness and area.

Gate. A setting gives sheet thickness equal to or better than the A-01 baseline with at least 20% more biochar yield.

A-03 · Activation design of experiments: composite grade versus electrode grade

Closes G-02 · **Priority** P1 · **Effort** about 8 weeks

Hypothesis. A lower KOH ratio gives thin sheets with much lower area, which suit composites better (Chapter 5.4).

Design. KOH:biochar at 0.5, 1, 2 and 3; temperature 700, 750, 800 and 850 °C; hold 0.5, 1 and 2 h. A D-optimal subset of about 24 runs.

Measurements. Thickness (AFM), BET area and micropore fraction, conductivity (C-05 method), oxygen content, yield.

Gate. A *composite grade* recipe with median thickness of 30 nm or less and BET area of 900 m²/g or less. It becomes the HCNS used in Modules E to H.

A-04 · Is hemp bast special?

Closes G-03 · **Priority** P2 · **Effort** about 5 weeks

Method. Run flax, kenaf and jute bast, and hemp hurd, through the A-03 composite-grade recipe, three batches each.

Measurements. Thickness, lateral size and aspect ratio (C-02, C-03); area; yield.

Gate. Hemp bast counts as distinct if its median aspect ratio is at least 1.5 times every comparator's. If not, the book's language shifts from "hemp-derived" to "bast-derived" nanosheets.

A-05 · Washing and residue limits

Closes G-04 · **Priority** P1 · **Effort** about 3 weeks

Design. Four wash sequences on one HCNS batch: water only; 1 M HCl then water; 1 M HCl at 60 °C then water; two cycles of the third.

Measurements. K, Ca, Si and Fe by ICP-OES after digestion; ash by TGA at 900 °C in air; potassium leached into water at 60 °C over 24 h.

Gate. Potassium under 1000 ppm and ash under 0.5 wt%. The cheapest sequence that passes becomes part of SC-1.

A-06 · Reproducibility and tenfold scale-up

Closes G-01, G-02 · **Priority** P1 · **Effort** about 6 weeks

Method. Run the A-03 composite-grade recipe at ten times the A-01 scale, three times. Log electricity, water, acid, KOH and argon for I-03.

Gate. Batch-to-batch coefficient of variation of 10% or less for median thickness and BET area, and yield within 15% of the small-scale value.

Module B — Graphitization and exfoliation of hemp nanosheets

B-01 · Iron-catalysed graphitization before activation

Closes G-05 · **Priority** P1 · **Effort** about 6 weeks

Hypothesis. A light iron loading on the hydrothermal biochar raises graphitic order without converting sheets to tubules (risk noted in Chapter 4.4).

Design. $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ impregnation at 1, 3 and 5 wt% Fe; heat-treatment at 800 and 1000 °C under argon; then with and without SC-1 activation.

Method. Leach iron with 2 M HCl at 60 °C until ICP shows under 500 ppm Fe.

Measurements. d002, Lc and La by XRD; Raman at two wavelengths; SEM and TEM morphology; residual Fe; area.

Gate. A resolvable (002) peak with d002 of 0.340 nm or less **and** sheet morphology retained in SEM.

B-02 · Catalytic graphitization after activation

Closes G-05 · **Priority** P1 · **Effort** about 5 weeks

Design. Composite-grade HCNS impregnated with Fe or Ni at 1, 3 and 5 wt%; heat at 900, 1000 and 1100 °C; leach as in B-01.

Measurements and gate. As B-01.

B-03 · Flash Joule heating of hemp nanosheets

Closes G-05 · **Priority** P1 · **Effort** about 8 weeks, including rig commissioning

Hypothesis. Because HCNS already conduct, flash heating converts them to turbostratic graphene-like sheets without additives (Chapter 4.2).

Method.

1. Load 0.1–0.5 g HCNS in a quartz tube between graphite or copper electrodes; compress to a reproducible bed resistance found in pilot runs.
2. Discharge a capacitor bank in a series of increasing energies; vent through a filter.
3. Repeat each energy three times.

Measurements. Raman 2D/G and D/G ratios and 2D shape; XRD; SEM; area; mass loss.

Gate. A turbostratic signature (sharp, symmetric 2D band and weak D band) with sheet morphology retained.

Safety. High voltage and arc flash: interlocked enclosure, discharge stick, two-person rule.

B-04 · Thermal annealing series (upper bound)

Closes G-05 · **Priority** P2 · **Effort** about 4 weeks (partner furnace)

Design. Composite-grade HCNS at 1500, 2000 and 2500 °C under argon for 1 h; plus a 1000 °C "closing anneal" to test micropore collapse.

Measurements. XRD, Raman, area and micropore fraction, conductivity.

Output. The order ceiling reachable by heat alone; the benchmark for B-01 to B-03.

B-05 · Exfoliating graphitized hemp nanosheets

Closes G-06 · **Priority** P2 · **Effort** about 5 weeks

Method. Two routes on the best B-01 to B-04 products:

1. Shear mixing in aqueous sodium cholate at a local shear rate above 10^4 s^{-1} (Chapter 3.2); centrifuge to remove unexfoliated material.
2. Electrochemical: powder pressed in a porous holder as anode in 0.1 M $(\text{NH}_4)_2\text{SO}_4$ (Chapter 3.3).

Gate. Median thickness halved with lateral size kept above 70% of the starting value.

B-06 · Graphitizable coating

Closes G-05 · **Priority** P3 · **Effort** about 5 weeks

Method. Coat HCNS with 10 or 30 wt% lignin from hemp hurd (or mesophase pitch as control), carbonize at 1000 °C, anneal at 2000 °C.

Gate. Better order than B-04 at the same temperature.

B-07 · Order gate: choose graphitized candidates

Closes G-05 · **Priority** P1 · **Effort** about 2 weeks

Method. Run the SC-2 panel side by side on all B outputs.

Gate. Up to three G-HCNS candidates proceed to Module D, each meeting all of: d_{002} of 0.340 nm or less; a two-wavelength Raman picture consistent with L_a of 10 nm or more; sheet morphology retained; carbon yield of at least 50% from the HCNS input. If none passes, Module D still runs on HCNS as a deliberate negative control, and the hemp-to-GO claim is reported as not supported.

Module C — Characterization of the standalone materials

C-01 · Reference materials on arrival

Closes G-10 · **Priority** P1 · **Effort** about 3 weeks

Method. Buy two commercial GNP grades and one commercial single-layer GO; run SC-2 on each.

Output. GNP-R and GO-R, the program's controls. Any property more than 20% off the supplier's data sheet is logged and reported; Kauling's survey (Chapter 1.1) says to expect this.

C-02 · Thickness and layer metrology to ISO/TS 21356-1

Closes G-07 · **Priority** P1 · **Effort** about 4 weeks for the first set, then ongoing

Method.

1. Disperse at 0.05 mg/mL in water with 1 wt% sodium cholate by bath sonication for 10 min; centrifuge at low speed to remove aggregates.
2. Spin-coat on Si wafers with 290 nm oxide.
3. AFM tapping mode: 100 or more flakes per sample; TEM edge counts on 30 flakes.

Output. Thickness histograms and layer-number distributions for every material.

C-03 · Lateral size and aspect ratio

Closes G-07 · **Priority** P1 · **Effort** with C-02

Method. SEM of the C-02 substrates, 200 or more flakes; pair SEM and AFM on the same flakes where possible to get aspect ratio per flake.

Output. Aspect-ratio distribution, the main input to Table 11.3.

C-04 · Where does the surface area come from?

Closes G-08 · **Priority** P2 · **Effort** about 4 weeks

Method. N₂ at 77 K and CO₂ at 273 K isotherms with DFT pore-size distributions; repeat after gentle milling to see whether area tracks particle size or not; HRTEM of cross-sections.

Output. The share of area in pores under 2 nm. If over 70%, resin uptake and void risk are flagged for Module E.

C-05 · Powder conductivity at fixed pressure

Closes G-10 · **Priority** P2 · **Effort** about 2 weeks

Method. Insulated die with four in-line probes; 10, 50 and 100 MPa; record compact density at each; three repeats per material.

Output. One comparable conductivity table for HCNS, G-HCNS, GNP-R, H-rGO and graphite rGO.

C-06 · Skeletal and envelope density

Closes G-10 · **Priority** P2 · **Effort** about 1 week

Method. Helium pycnometry for skeletal density; envelope density by mercury intrusion or a powder envelope analyser.

Output. Densities and open porosity for Chapter 11.1.

C-07 · Chemistry to ISO/TS 23359

Closes G-04 · **Priority** P1 · **Effort** about 3 weeks

Method. XPS, TGA, ICP-MS and FTIR as the standard specifies, plus CHNS-O combustion; on every candidate material.

Output. O/C ratio, functional-group shares, trace metals.

C-08 · Effective in-plane modulus of hemp nanosheets

Closes G-09 · **Priority** P1 · **Effort** about 8 weeks

Method. Two independent routes:

1. **Raman band-shift.** Deposit isolated flakes on an SU-8-coated PMMA beam; strain in four-point bending to 0.5%; record the G and 2D band positions against strain; convert the shift rate to effective modulus with the calibration of Chapter 11.4.
2. **AFM indentation** of flakes suspended over 1–2 μm holes, where flakes are large enough.

Gate. If the effective modulus is below 50 GPa, stiffness claims are dropped for that material and Module F emphasizes toughness and conductivity.

Module D — Graphene oxide and reduced graphene oxide

D-01 · Benchmark the improved Hummers method on graphite

Closes G-11 (calibration) · **Priority** P1 · **Effort** about 4 weeks

Purpose. Prove the lab can reproduce a published GO synthesis before it touches hemp carbon.

Method. Follow the documented run exactly (Chapter 6.1): 1 g graphite flake in 130 mL 9:1 H₂SO₄/H₃PO₄; 6.0 g KMnO₄; 50 °C for 12 h; quench on about 120 mL ice water with 1 mL 30% H₂O₂; sieve, centrifuge, wash with water/HCl/ethanol; coagulate, filter on PTFE, vacuum-dry. Replace the diethyl-ether coagulation with dialysis if the lab's solvent rules require it, and record the change.

Measurements. Yield; XPS C/O; XRD interlayer spacing; AFM thickness and lateral size; UV-vis.

Gate. Yield within ±15% of the reported 1.8 g, and at least 50% monolayers by AFM count.

Safety. Add KMnO₄ slowly with stirring; keep the concentrated-acid stage well below 55 °C (the decomposition threshold of manganese heptoxide); blast shield; never let organic solvents meet the acid–permanganate mixture.

D-02 · Oxidize hemp carbons: sheets or dots?

Closes G-11 · **Priority** P1 · **Effort** about 8 weeks

Hypothesis. Graphitized hemp nanosheets oxidize to GO sheets; ungraphitized ones fragment into dots (Chapter 6.3).

Inputs. Composite-grade HCNS; up to three G-HCNS from B-07; GNP-R as control.

Method.

1. Scale D-01 to 0.25 g carbon: 32.5 mL acid mixture, 1.5 g KMnO₄.
2. Add KMnO₄ in six portions over 30 min in an ice bath, logging temperature every second (or in a reaction calorimeter).
3. Hold at 50 °C for 12 h; quench and purify as D-01.
4. Repeat with half the oxidant (3 weight equivalents), since porous carbon may over-oxidize.

Measurements. Heat-release profile; yield; lateral size and thickness (AFM, SEM); photoluminescence (dots fluoresce); XPS C/O.

Classification. *Sheets* if median lateral size is 500 nm or more and at least 50% are monolayers. *Dots* if median size is under 20 nm with photoluminescence.

Gate. At least one hemp input gives sheets, then H-GO proceeds to D-03 to D-05 and Modules E to H. If none does, the result is published as a negative finding, and the dot product moves to an exploratory track.

D-03 · Purification and residue limits

Closes G-12 · **Priority** P2 · **Effort** about 4 weeks

Design. Compare dialysis (12–14 kDa membrane, 7 days, daily water change) with repeated HCl washing and centrifugation, on H-GO and graphite GO.

Measurements. Mn, S and K by ICP-MS; final pH; zeta potential; yield loss.

Gate. Manganese under 100 ppm and sulfur under 0.5 wt%, at the lower-cost method. The passing method becomes the purification SOP.

D-04 · Reduction matrix

Closes G-12 · **Priority** P2 · **Effort** about 6 weeks

Design. Four reductions (Chapter 6.6) on H-GO and graphite GO: L-ascorbic acid in water at 95 °C for 1 and 12 h; 55% HI at 100 °C for 1 h; thiourea dioxide; thermal at 300 and 1000 °C under argon.

Measurements. XPS C/O; Raman; XRD; conductivity of powder compacts (C-05) and of vacuum-filtered films (four-probe).

Gate. For at least one method, H-rGO conductivity reaches 50% or more of graphite rGO made the same way.

D-05 · Reduction during cure or hot-pressing

Closes G-12 · **Priority** P2 · **Effort** about 5 weeks

Hypothesis. A cure or hot-press schedule can partly reduce GO in place, giving conductivity without a separate reduction step (Chapter 6.6).

Design. GO and H-GO at 0.5 wt% in M1 and M2; standard cure versus an extended post-cure at 180 °C; hot-pressed plaques at 200 °C.

Measurements. Composite volume conductivity; Raman of embedded sheets (G position, D/G); DMA T_g.

Gate. Conductivity rises by two orders of magnitude or more over the standard-cure control, with T_g loss of 5 °C or less.

D-06 · Scale-up and process safety for oxidation

Closes G-11 · **Priority** P2 · **Effort** about 8 weeks

Method.

1. Hazard study (HAZOP) of the oxidation using D-02 heat-release data.
2. Run a 5 g batch in a jacketed 1 L reactor with dosing-controlled permanganate addition and a high-temperature trip at 45 °C.
3. Design the quench and the waste train: neutralize spent acid and recover or precipitate manganese.

Output. A scale SOP; a waste inventory per kilogram of GO for I-03 and I-04.

Gate. Calculated adiabatic temperature rise stays within the safety margin set by the hazard study, and the product matches D-02 within 15% on C/O and lateral size.

Module E — Composite processing

E-01 · Dispersion method screen

Closes G-13 · **Priority** P1 · **Effort** about 6 weeks

Design. Fillers HCNS, G-HCNS, GNP-R and H-GO (or GO-R) at 0.5 wt% in M1; four methods:

1. Three-roll mill: three passes, final gap 5 µm.
2. Probe sonication in acetone, 30 min pulsed in an ice bath, then solvent removal under vacuum to under 0.5 wt% residual (TGA).
3. High-shear mixer, 30 min.
4. Sonication followed by three-roll milling.

Measurements. Agglomerate size distribution by optical microscopy (10 fields); viscosity; a quick flexural test.

Gate. Agglomerate D₉₀ under 10 µm. The best method per filler is fixed for Module F.

E-02 · Surface oxygen and amine grafting

Closes G-14 · **Priority** P2 · **Effort** about 6 weeks

Design. Composite-grade HCNS as made (4–6 at% O); air-oxidized at 350 °C for 1 and 4 h; deoxygenated in H₂/Ar at 900 °C; and amine-grafted with a polyetheramine at 80 °C.

Measurements. XPS O and N; dispersion (E-01 method); K_{IC} by ASTM D5045 at 0.5 wt% in M1.

Gate. A treatment improves K_{IC} by 15% or more over untreated HCNS at the same loading.

E-03 · Edge carboxylation by ball milling

Closes G-14 · **Priority** P3 · **Effort** about 4 weeks

Method. Apply the dry-ice ball-milling route (Chapter 3.4) to HCNS and the best G-HCNS: planetary mill, pressure-rated jars with relief, 12, 24 and 48 h.

Measurements. O/C; dispersibility in polar solvents; lateral size and aspect ratio.

Gate. Dispersibility improves with aspect-ratio loss of 30% or less.

Safety. Dry ice sublimates and pressurizes sealed jars; use only rated jars and vent before opening.

E-04 · Rheology and the processing window

Closes G-13 · **Priority** P1 · **Effort** about 4 weeks

Design. HCNS, G-HCNS, GNP-R and GO at 0, 0.1, 0.25, 0.5, 1, 2 and 5 wt% in M1 and M3.

Measurements. Viscosity from 0.1 to 100 s⁻¹ at 25 °C and at processing temperature; low-shear yield stress; pot life.

Output. The maximum loading for infusion (viscosity under 0.5 Pa·s at processing temperature) and for casting (under 10 Pa·s). [T]

E-05 · Cure kinetics with fillers

Closes G-04, G-14 · **Priority** P2 · **Effort** about 4 weeks

Design. M1, M2 and M3, each neat and with each filler at 0.5 and 2 wt%.

Method. DSC at 5, 10, 15 and 20 °C/min; apparent activation energy by Kissinger and Ozawa analyses; isothermal conversion at the cure temperature.

Gate. Activation-energy change of 10% or less, and final conversion of 95% or more. A larger shift for HCNS than for GNP-R points to residues and sends the batch back to A-05.

Module F — Composite performance

Unless stated otherwise, Module F uses the best dispersion method from E-01, the cure schedule fixed by E-05, and SC-3 statistics.

F-01 · Loading series: stiffness and strength

Closes G-15 · **Priority** P2 · **Effort** about 8 weeks

Design. HCNS, best G-HCNS, GNP-R and GO-R (plus H-GO if D-02 passed) at 0, 0.05, 0.1, 0.25, 0.5 and 1.0 wt%, in M1 and M3. Convert every loading to vol% with C-06 densities.

Tests. Tensile (ASTM D638) and flexural (ASTM D790), five or more specimens each.

Gate. At equal vol%, the best hemp filler delivers 70% or more of GNP-R's modulus gain with tensile-strength loss of 5% or less. Compare results with Table 11.3 using the measured E_f and aspect ratio.

F-02 · Fracture toughness

Closes G-15 · **Priority** P2 · **Effort** about 5 weeks

Design. Single-edge-notch bend specimens per ASTM D5045 at 0.1, 0.25, 0.5 and 1.0 wt%, M1 and M3; razor-tapped pre-cracks.

Gate. K_{IC} up 30% or more at 0.5 wt% or less in M1 for at least one hemp filler.

F-03 · Fatigue crack growth

Closes G-15 · **Priority** P3 · **Effort** about 6 weeks

Design. Compact-tension specimens (ASTM E647) for neat M1, the best hemp formulation and the GNP-R formulation; constant-amplitude loading at $R = 0.1$.

Gate. Crack-growth rate cut by half or more at mid-range ΔK compared with neat resin.

F-04 · Thermomechanics and crosslink density

Closes G-15 (and H9.1) · **Priority** P2 · **Effort** with F-01

Tests. DMA per ASTM D7028 on every F-01 formulation; DSC T_g ; crosslink density from the rubbery modulus.

Gate (M3 only). Storage modulus at 25 °C up 20% or more and T_g up 5 °C or more at 1 wt% or less (hypothesis H9.1).

F-05 · Electrical percolation

Closes G-16 · **Priority** P2 · **Effort** about 5 weeks

Design. HCNS, G-HCNS, GNP-R and H-rGO at eight or more loadings up to 5 wt% or the E-04 processing limit, in M1.

Tests. ASTM D257 below percolation; four-probe above; fit Φ_c and t (Chapter 11.5).

Gate. Percolation at 1.5 vol% or less for G-HCNS.

F-06 · Interface by Raman strain mapping

Closes G-09, G-14 · **Priority** P1 · **Effort** about 8 weeks

Method.

1. Model composite: isolated flakes in a thin epoxy layer on a beam; map 2D and G band shifts along flakes at 0, 0.2, 0.4 and 0.6% strain.
2. Bulk composite at 1 wt% strained in situ under the Raman microscope.

Output. Effective filler modulus and interfacial shear strength for HCNS, G-HCNS and GNP-R. These feed C-08 and Chapter 11.

F-07 · Dispersion quantified

Closes G-13 · **Priority** P1 · **Effort** about 4 weeks

Method. TEM of ultramicrotomed sections (10 or more images per sample); micro-CT at 1 μm voxel size or finer for agglomerates and voids.

Output. Agglomerate volume fraction and size distribution, regressed against F-01 and F-02 results.

F-08 · Blind fractography

Closes G-15 · **Priority** P2 · **Effort** about 3 weeks

Method. SEM of F-02 fracture surfaces; two scorers who do not know the formulation classify crack deflection, pinning, pull-out and void growth; profilometry for surface roughness.

Output. Which mechanisms operate for each filler; inter-scorer agreement reported.

F-09 · Hierarchical hemp laminates

Closes G-17 · **Priority** P2 · **Effort** about 10 weeks

Design. Hemp fabric or non-woven mat with M1 and M3, each unmodified and with the best HCNS loading from F-02. Vacuum infusion where E-04 allows; otherwise hand lay-up and press.

Tests. ASTM D3039, D2344 and D5528; fibre volume fraction by burn-off or image analysis.

Gate. Interlaminar shear strength up 15% or more and mode I interlaminar toughness up 25% or more over the unmodified matrix.

F-10 · Durability

Closes G-20 · **Priority** P3 · **Effort** about 16 weeks (exposure time)

Design. The two best formulations plus neat controls, in M1 and M3.

Exposures. Moisture uptake (ASTM D5229) at 50% RH and in immersion; UV and condensation (ASTM G154) for 1000 h; 100 freeze–thaw cycles from –30 to +60 °C.

Gate. 85% or more property retention, and no worse than the neat matrix under the same exposure.

Module G — Vitrimers and Hempoxies

G-01 · Hempoxies matrix baseline

Closes G-18 · **Priority** P2 · **Effort** about 10 weeks

Materials. Epoxidized and maleinized hemp oil, made in-house (peracid epoxidation; thermal maleinization) or sourced; M2 and M4 as comparators.

Design. EHO/MHO at 1:1 epoxy-to-anhydride equivalents, with no catalyst, zinc acetate at 5 mol%, or an organic transesterification catalyst.

Tests. DSC cure profile; DMA Tg; tensile; insoluble fraction after 48 h in an inert solvent; stress relaxation at 140–200 °C.

Gate. The catalysed M3 relaxes stress to below 1/e of its initial value within 1 h at 200 °C or below, while keeping an insoluble fraction of 90% or more.

Safety. Peracid epoxidation is exothermic and peroxides can accumulate; follow the reagent supplier's guidance and keep batches small.

G-02 · Stress relaxation with fillers

Closes G-18 (H9.2) · **Priority** P2 · **Effort** about 6 weeks

Design. Catalysed M2 and M3 with HCNS, G-HCNS, GO-R and GNP-R at 0.5 and 2 wt%; relaxation at four temperatures.

Output. Relaxation times and Arrhenius activation energies.

Gate (H9.2). For HCNS, the relaxation time at 160 °C falls by 25% or more against the unfilled vitrimer.

G-03 · Reprocessing cycles

Closes G-18 (H9.4) · **Priority** P2 · **Effort** about 6 weeks

Method. Grind cured plaques to under 2 mm; hot-press (program default 180–200 °C, 5–10 MPa, 1–2 h); repeat for three cycles.

Tests. Tensile, DMA, and Raman of the filler after each cycle.

Gate (H9.4). 80% or more of tensile strength kept after three cycles.

G-04 · Joule-heated healing and reshaping

Closes G-18 (H9.3) · **Priority** P3 · **Effort** about 5 weeks

Design. Above-percolation formulations from F-05 in catalysed M3; electrodes painted on; DC voltage steps; infrared thermography.

Gate (H9.3). A standard scratch heals at 30 V or less within 10 min.

G-05 · Chemical recycling and filler recovery

Closes G-18 (H9.5) · **Priority** P2 · **Effort** about 6 weeks

Method. Dissolve the catalysed vitrimer by alcoholysis (program default: ethylene glycol with the same catalyst, at elevated temperature); filter, wash and dry the filler; run the SC-2 panel; reuse it in a fresh F-02 batch.

Gate (H9.5). 90% or more of filler mass recovered; I_D/I_G within 10% of the original; reused filler gives 90% or more of its original K_{IC} gain.

Module H — Other matrices

H-01 · Portland cement paste and mortar

Closes G-19 · **Priority** P2 · **Effort** about 10 weeks (28-day cures)

Design. GO-R, H-GO and HCNS at 0.01, 0.02, 0.05 and 0.1% by mass of cement; filler dispersed by sonication in the mixing water with a polycarboxylate superplasticizer.

Tests. Flow table; compressive (ASTM C109 or C349) and flexural (ASTM C348) at 7 and 28 days; mercury porosimetry; isothermal calorimetry.

Gate. H-GO reaches 70% or more of GO-R's flexural gain at equal dose.

H-02 · Lime-hemp binder

Closes G-19 · **Priority** P2 · **Effort** about 16 weeks (90-day cures)

Design. Hydrated-lime binder with HCNS or H-GO at 0.05–0.5% by binder mass; hemp shiv aggregate.

Tests. Compressive strength at 28 and 90 days; thermal conductivity; carbonation depth.

Gate. Compressive strength up 20% or more with thermal conductivity up no more than 10%.

H-03 · Ceramic screening (partner laboratory)

Closes G-19 · **Priority** P3 · **Effort** about 8 weeks

Design. Alumina with 1–3 vol% G-HCNS or GNP-R, spark plasma sintered.

Tests. Raman to confirm the filler survived; density; fracture toughness.

Gate. Filler survives sintering and toughness rises 20% or more.

H-04 · Copper-matrix screening

Closes G-19 · **Priority** P3 · **Effort** about 8 weeks

Design. Copper powder with 0.5–2 vol% G-HCNS or GNP-R; milled, pressed and sintered in hydrogen-argon.

Tests. Density, hardness, electrical conductivity (%IACS), micro-tensile.

Gate. Hardness up 15% or more with conductivity loss of 10 %IACS or less.

H-05 · Anticorrosion primer

Closes G-19 · **Priority** P3 · **Effort** about 12 weeks

Design. M1 epoxy primer with HCNS at 0.1, 0.5 and 1 wt% (spanning percolation), with and without zinc dust, on blasted steel panels.

Tests. Salt spray (ASTM B117) for 1000 h; scribe creep; electrochemical impedance.

Gate. Below-percolation HCNS reduce scribe creep against the neat primer. Conductive formulations without zinc are expected to do worse (Chapter 10.4); confirming that is itself a result.

H-06 · Thermoplastics

Closes G-19 · **Priority** P3 · **Effort** about 8 weeks

Design. PLA and polypropylene with HCNS, G-HCNS and GNP-R at 0.5, 1, 2 and 5 wt%; twin-screw compounding; injection-moulded bars. Dry PLA and fillers thoroughly before compounding.

Tests. Tensile (ISO 527); impact; heat deflection; melt flow; molecular-weight retention for PLA.

Gate. Modulus up 15% or more at 2 wt% or less, with PLA molecular-weight loss under 10%.

Module I — Safety, life cycle and techno-economics

I-01 · Worker exposure and controls

Closes G-21 · **Priority** P1 · **Effort** about 4 weeks, then repeated per new task

Method. During weighing, milling, three-roll milling, and cutting or sanding cured parts: real-time particle counting near the breathing zone, plus filter sampling for elemental carbon, the metric NIOSH uses for carbon nanotubes ([NIOSH CIB 65](#)).

Benchmarks. NIOSH's preliminary graphene benchmark of under $10 \mu\text{g}/\text{m}^3$ ([CompositesWorld](#)) and, as the conservative reference, the $1 \mu\text{g}/\text{m}^3$ respirable elemental-carbon limit for nanotubes (8-hour TWA).

Gate. Every task below $1 \mu\text{g}/\text{m}^3$ respirable elemental carbon with the controls in place; otherwise add engineering controls and re-measure before the task continues.

I-02 · Particle release from finished parts

Closes G-21 · **Priority** P2 · **Effort** about 6 weeks

Method. Abrade F- and H-series specimens with a standard abrader, before and after UV ageing (ASTM G154); collect released particles on filters; SEM to classify free sheets versus matrix-bound fragments.

Gate. No free sheets detected above the GNP-R composite's release level.

I-03 · Mass and energy balance

Closes G-22 · **Priority** P1 (data capture starts in A-01) · **Effort** about 4 weeks of analysis

Method. From A-06, B-series and D-06 logs, compute per kilogram of HCNS, G-HCNS and H-GO: biomass, water, acids, KOH, argon, electricity, heat, yield, and waste streams; propagate uncertainty.

Output. The inventory for I-04 and I-05. Yield is the number the hemp literature does not report (Chapter 5.6).

I-04 · Life-cycle assessment

Closes G-22 · **Priority** P2 · **Effort** about 8 weeks

Method. Cradle-to-gate assessment following ISO 14040 and 14044, with two functional units: 1 kg of filler, and the filler mass needed to raise K_IC of 1 kg of epoxy by 30% (from F-02). Use the New Brunswick grid mix. Comparators come from published graphite-route inventories (Chapter 17.3) and from the biomass flash-graphene result of 1.9 kg CO₂-eq per kg.

Gate. The best hemp route shows at least 30% lower global-warming impact than the graphite-GNP route per functional unit two. If not, the sustainability claim is withdrawn or narrowed.

I-05 · Techno-economics

Closes G-22 · **Priority** P2 · **Effort** about 6 weeks

Method. Process model for a 10 t/yr plant from I-03; capital and operating cost; sensitivity to yield, KOH recovery, electricity price and feedstock cost.

Gate. Cost per unit of performance within twice that of commercial GNP at pilot scale.

15.10 Decision points

After	If the result is...	Then
A-01	No Canadian grain-straw bast gives sheets	Add a pre-treatment work item; source fibre-hemp bast in the meantime

After	If the result is...	Then
B-07	No graphitized candidate passes	Run D-02 on HCNS as a negative control; report the hemp-to-GO pathway as not supported; HCNS continue as tougheners and conductive fillers
C-08 and F-06	Effective modulus under 50 GPa	Drop stiffness claims; focus Module F on toughness, conductivity and vitrimer function
D-02	Dots, not sheets, from every hemp input	Publish the negative result; open an exploratory dots track
F-02	No K _{IC} gain of 30% at 0.5 wt% or less	Revisit E-02 and E-03 interface chemistry once, then stop the epoxy toughening claim
G-01	EHO/MHO does not behave as a vitrimer	Hempoxies uses a disulfide-exchange hemp-oil route (M5-type chemistry) or partners on a proven vitrimer
I-04	Hemp route not at least 30% better per functional unit	Narrow the sustainability claim to what was measured

15.11 Master index of the 58 experiments

ID	Experiment	Closes	Priority	Weeks
A-01	Feedstock survey and baseline	G-01	P1	6
A-02	Hydrothermal-step DoE	G-02	P1	8
A-03	Activation DoE, composite grade	G-02	P1	8
A-04	Is hemp bast special?	G-03	P2	5
A-05	Washing and residue limits	G-04	P1	3
A-06	Reproducibility and scale-up	G-01, G-02	P1	6
B-01	Fe graphitization before activation	G-05	P1	6
B-02	Catalytic graphitization after activation	G-05	P1	5

ID	Experiment	Closes	Priority	Weeks
B-03	Flash Joule heating of HCNS	G-05	P1	8
B-04	Thermal annealing series	G-05	P2	4
B-05	Exfoliating graphitized HCNS	G-06	P2	5
B-06	Graphitizable coating	G-05	P3	5
B-07	Order gate	G-05	P1	2
C-01	Reference materials	G-10	P1	3
C-02	Thickness metrology	G-07	P1	4
C-03	Lateral size and aspect ratio	G-07	P1	with C-02
C-04	Origin of surface area	G-08	P2	4
C-05	Powder conductivity	G-10	P2	2
C-06	Densities	G-10	P2	1
C-07	Chemistry to ISO/TS 23359	G-04	P1	3
C-08	Effective modulus	G-09	P1	8
D-01	Hummers benchmark on graphite	G-11	P1	4
D-02	Oxidizing hemp carbons	G-11	P1	8
D-03	Purification and residues	G-12	P2	4
D-04	Reduction matrix	G-12	P2	6
D-05	Reduction during cure	G-12	P2	5
D-06	Oxidation scale-up and safety	G-11	P2	8
E-01	Dispersion screen	G-13	P1	6
E-02	Surface oxygen and amine grafting	G-14	P2	6
E-03	Edge carboxylation	G-14	P3	4
E-04	Rheology and processing window	G-13	P1	4
E-05	Cure kinetics	G-04, G-14	P2	4
F-01	Stiffness and strength series	G-15	P2	8

ID	Experiment	Closes	Priority	Weeks
F-02	Fracture toughness	G-15	P2	5
F-03	Fatigue crack growth	G-15	P3	6
F-04	Thermomechanics	G-15	P2	with F-01
F-05	Electrical percolation	G-16	P2	5
F-06	Raman interface mapping	G-09, G-14	P1	8
F-07	Dispersion quantified	G-13	P1	4
F-08	Blind fractography	G-15	P2	3
F-09	Hierarchical hemp laminates	G-17	P2	10
F-10	Durability	G-20	P3	16
G-01	Hempoxies matrix baseline	G-18	P2	10
G-02	Stress relaxation with fillers	G-18	P2	6
G-03	Reprocessing cycles	G-18	P2	6
G-04	Joule-heated healing	G-18	P3	5
G-05	Recycling and filler recovery	G-18	P2	6
H-01	Portland cement	G-19	P2	10
H-02	Lime-hemp binder	G-19	P2	16
H-03	Ceramics (partner)	G-19	P3	8
H-04	Copper matrix	G-19	P3	8
H-05	Anticorrosion primer	G-19	P3	12
H-06	Thermoplastics	G-19	P3	8
I-01	Worker exposure	G-21	P1	4
I-02	Particle release	G-21	P2	6
I-03	Mass and energy balance	G-22	P1	4
I-04	Life-cycle assessment	G-22	P2	8
I-05	Techno-economics	G-22	P2	6

The weeks sum to roughly 340 experiment-weeks; with three parallel workstreams the program fits in about 30 months (Chapter 16.4).

Chapter 16 — Running the program

Three people, a chemistry lab with good ventilation, a composites bench and access to university core facilities can run the whole program in about 30 months. The first nine months go to the P1 gaps; nothing downstream starts until Gate 1 is passed.

16.1 Team

Role	Time	Owns
Materials chemist (lead)	Full time	Modules A, B, D
Composites engineer	Full time	Modules E, F, G, H
Characterization specialist	Full time	Module C and the SC-2 panel for every batch
Safety and sustainability analyst	Part time	Module I; hazard reviews before each new procedure

16.2 Hazards and minimum controls

Hazard	Where	Minimum control
Concentrated H ₂ SO ₄ and H ₃ PO ₄ ; KMnO ₄ forming manganese heptoxide	D-01, D-02, D-06	Fume hood, blast shield, ice bath, portion-wise addition, temperature logging, no organics near the mixture (Chapter 6.4)
KOH melt at 700–800 °C; hydrogen release; possible metallic potassium	A-series activation	Nickel boats, argon purge, slow cooling, controlled water quench in a hood
Pressure vessels	Hydrothermal step	Rated autoclaves with burst discs; fill limit; shielded oven
High voltage and arc flash	B-03 flash heating	Interlocked enclosure, grounding stick, two-person rule

Hazard	Where	Minimum control
55% HI, H ₂ O ₂ , peracids	D-04, D-01, G-01	Hood, face shield, small batches, compatible storage
Pressurized milling jars	E-03	Pressure-rated jars, venting before opening
Dry nanocarbon powders	Weighing, milling, sanding	Enclosed weighing, HEPA local exhaust, wet methods, masterbatch handling; verify by I-01

For dry powders, NIOSH's 2026 study of 44 workers at 11 US companies found that handling dry powder and handling more than 100 kg raised exposure, and recommended that composite makers receive nanocarbons already embedded in masterbatch ([CompositesWorld](#)). **[R]** This program adopts that practice: once a filler passes Module C, it is converted to a resin or polymer masterbatch before Modules F to H.

16.3 Equipment

In-house	Core facility or partner
Fume hoods; PTFE-lined autoclaves; tube furnace to 1100 °C with argon	XPS; XRD; SEM with EDS; AFM; TEM
Glovebox or dry room for KOH handling	ICP-MS or ICP-OES
Centrifuge; freeze dryer; probe sonicator; high-shear mixer	Gas sorption (if not in-house); helium pycnometer
Three-roll mill; vacuum oven; hot press; vacuum-infusion kit	Micro-CT
DSC; TGA; rheometer; DMA	Graphitization furnace to 2500 °C
Universal test machine with extensometer	Spark plasma sintering (H-03)
Raman microscope, 532 and 633 nm; four-probe powder die	Salt-spray and UV chambers
Capacitor-bank flash rig (built in-house, B-03)	Twin-screw compounder and injection moulder (H-06)

16.4 Phases and gates

Phase	Months	Experiments	Gate at the end
1. Foundations and P1 gaps	0–9	A-01, A-02, A-03, A-05, A-06; B-01 to B-03; C-01 to C-03, C-07; D-01; E-01; I-01; I-03 data capture	Gate 1: composite-grade HCNS made reproducibly; graphitization route chosen or ruled out; exposure controls verified
2. Core comparison	9–20	B-04, B-07; C-04 to C-06, C-08; D-02 to D-05; E-02, E-04, E-05; F-01, F-02, F-04 to F-07; G-01	Gate 2: hemp versus GNP verdict in epoxy; GO sheets-or-dots verdict; Hempoxies matrix shows vitrimer behaviour or not
3. Extension and evidence	20–30	A-04; B-05, B-06; D-06; E-03; F-03, F-08 to F-10; G-02 to G-05; H-01 to H-06; I-02, I-04, I-05	Final: life-cycle and cost verdicts; publication of all results, positive and negative

A 30-month program in three phases, each closed by a go/no-go gate

Experiments by phase from Chapter 16.4. Accent: the go/no-go gate that closes each phase.

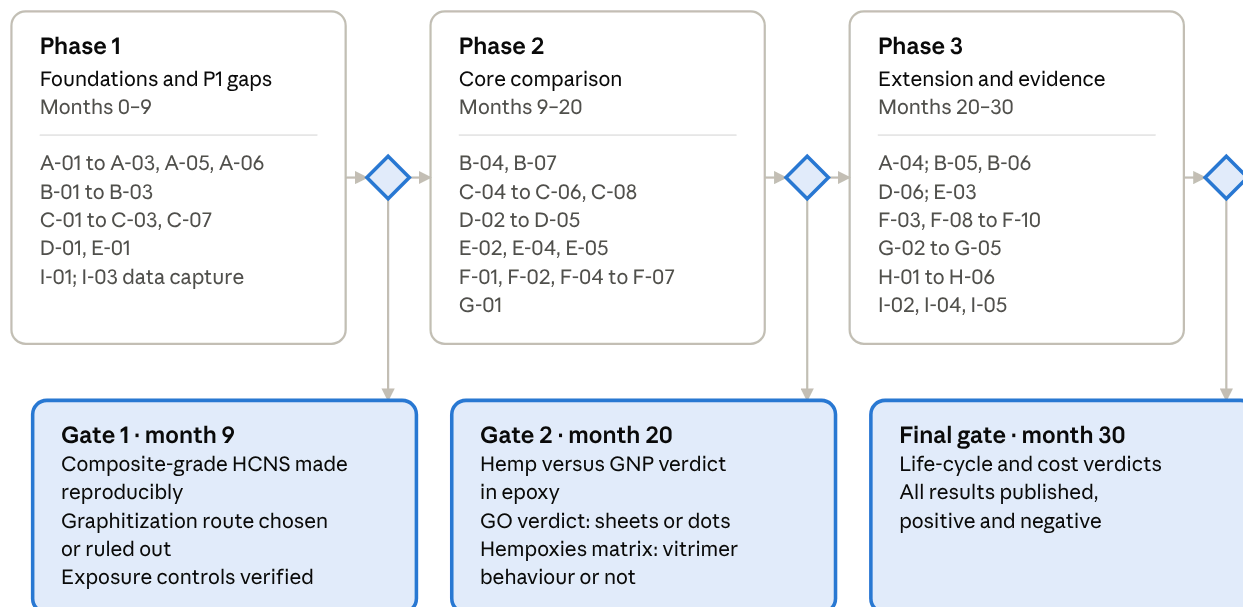


Figure 16.1 · program roadmap, three phases and three gates

Phase 1 spends nine months on the priority-1 gaps, and nothing downstream starts until Gate 1 is passed. Each later gate turns a body of results into a published verdict.

16.5 Data management

- **One sample ID** follows each material from feedstock lot through every process step, characterization, composite and test.
- **Raw data** (spectra, images, isotherms, load-displacement curves) are kept with the metadata needed to reprocess them, in an electronic lab notebook.
- **Every reported number links to its raw file and method.** This is the program's working form of Seshat's Axiom: retrieve, store, cite.
- **Datasets are deposited with DOIs** on Zenodo at each gate, including failed and negative results, so others can check or reuse them.
- **Pre-registration:** each experiment's hypothesis and gate are time-stamped before testing (SC-3).

Chapter 17 — The case for hemp: where it wins, where it loses

Hemp's strongest case is not "better than graphene". It is a renewable, domestically grown, permanganate-free route to a conductive, toughening nanosheet, and the only route that allows an all-hemp composite. Its weak points are low graphitic order, an unmeasured modulus, an unreported yield and an unmeasured footprint. Chapter 15 is built to settle each one.

17.1 Scorecard today

Criterion	Hemp nanosheets	Graphite-derived GNP, GO, rGO	Verdict now
Supply security	Domestic biomass; 15,380 ha in Canada in 2025, mostly grain (Ch. 2.2)	Natural graphite concentrated in China, with export controls (Ch. 2.1)	Hemp ahead [R]
Graphitic order and stiffness	Partial order; modulus never measured	High order; reduced-GO sheets 0.25 TPa	Graphite ahead

Criterion	Hemp nanosheets	Graphite-derived GNP, GO, rGO	Verdict now
Conductivity	211–226 S/m (compacts)	Up to 29,800 S/m for HI-reduced rGO	Graphite ahead unless B-series succeeds
Surface area and porosity	1505–2287 m ² /g	Low for GNP	Hemp ahead for energy and adsorption; unclear for composites
Built-in surface chemistry	4–6 at% oxygen	About 6 at% in the patent's GNP reference	Even
Process chemicals for the nanosheet	Dilute acid, KOH	Shear routes are mild; GO needs concentrated acid and permanganate	Hemp likely ahead of GO; even with shear-exfoliated GNP [H]
Measured footprint	None	Published inventories exist (17.3)	Unknown until I-04
All-hemp composite system	Fibre, oil-based epoxy and nanosheet from one plant	Not possible	Hemp unique
Proven composite performance	None	Many peer-reviewed results (Ch. 8)	Graphite ahead

17.2 Supply in practice

Canadian hemp is a grain crop first: in 2025, 12,859 of 15,380 hectares were grown for grain, fibre acreage kept falling, and licences fell to 585 ([HempToday](#)). For nanosheets, that is good and bad news. Straw is a by-product, so the feedstock is cheap and needs no new acreage. But the bast in grain straw has not been tested in the Mitlin route (A-01), and decortication of grain straw at the scale a carbon plant needs is a question for the I-05 cost model. [H]

17.3 Footprint benchmarks from the literature

Route	Result	Source
GO and rGO by Hummers and Marciano methods	Cumulative energy demand about 20.7–68.5 GJ/kg; Hummers lower per kg, Marciano better per unit conductivity for thin films	Spanish LCA, CORE record
rGO at commercial scale (comparative LCA)	0.046 kg CO ₂ -eq/g thermal, 0.081 kg CO ₂ -eq/g chemical reduction; oxidation hotspots: sulfuric acid and neutralizing lime	Nottingham LCA supporting information
Electrochemical exfoliation (same study)	0.28 kg CO ₂ -eq/g at commercial scale; CVD orders of magnitude higher	Same
Biomass flash graphene, pyrolysis then flash	1.9 g CO ₂ -eq per g, about 86% below flashing raw biomass	Zhu et al., Nature Communications 2024

All **[R]**. Scopes, scales and system boundaries differ, so these numbers orient the I-04 study rather than rank the routes. Two program rules follow. Compare routes per unit of delivered performance, not per kilogram. Report biogenic carbon stored in the nanosheets separately, never netted against process emissions.

17.4 Where hemp nanosheets are most likely to be the best choice

Ranked by fit between known HCNS strengths and application needs. **[H]** throughout.

- 1. Toughening and conductive modification of bio-epoxies and vitrimers (Hempoxies).** Uses toughening, moderate conductivity and built-in oxygen; the all-hemp story is a genuine differentiator.
- 2. Energy-storage electrodes.** The proven home of the material (Chapter 5.2).
- 3. Lime-hemp and cement binders.** Plausible if dispersion works; aligned with bio-based construction.
- 4. Antistatic and EMI thermoplastics.** Needs percolation at practical loadings (F-05, H-06).

Less likely to suit HCNS without successful graphitization: high-stiffness aerospace laminates, thermal management, aluminium matrix composites, and transparent conductors.

17.5 What would change the verdict

- **Toward hemp:** B-07 passes, C-08 measures an effective modulus of 200 GPa or more, F-02 shows a 30% toughness gain at 0.5 wt% or less, and I-04 shows a 30% or larger footprint advantage per performance unit. Hemp would then be a credible graphite alternative for composites.
- **Away from hemp:** B-07 fails and C-08 comes in under 50 GPa. HCNS would remain a valuable specialty porous carbon, mainly for energy storage and adsorption, rather than a graphene substitute.

Either outcome is publishable, and either is worth knowing. That is the point of running the program rather than asserting the answer.

Appendices

Appendix A — Glossary

Term	Meaning in this book
1LG, 2LG, FLG	Monolayer, bilayer and few-layer (3–10 layers) graphene, per ISO/TS 80004-13
GNP	Graphene or graphite nanoplatelets: stacks thicker than FLG
GO, rGO	Graphene oxide; reduced graphene oxide
HCNS	Hemp-derived carbon nanosheets (the author's "Mitlinite" when made by the Wang–Mitlin route)
G-HCNS	HCNS that passed the graphitization gate B-07
H-GO, H-rGO	GO and rGO made from hemp carbon
GNP-R, GO-R	Commercial reference materials characterized in C-01

Term	Meaning in this book
Hydrothermal carbonization	Heating biomass in water under pressure (here 180 °C, 24 h)
KOH activation	Heating carbon with potassium hydroxide to open pores and, for hemp bast, separate sheets
Turbostratic	Graphene layers stacked with random rotation and wider spacing than graphite
d002, Lc, La	Interlayer spacing; crystallite stack height; in-plane crystallite size
I _D /I _G	Ratio of Raman D- and G-band intensities; a disorder indicator (read with care, Ch. 12.3)
BET area	Specific surface area from gas adsorption per ISO 9277
Percolation threshold	Filler volume fraction at which a conductive network first spans the composite
Halpin–Tsai	Empirical micromechanics equations for composite stiffness (Ch. 11.2)
Vitrimer	Permanent covalent network whose bonds exchange on heating, allowing reprocessing
T _v	Topology-freezing temperature of a vitrimer
EHO, MHO	Epoxidized and maleinized hemp oil
M1–M5	Matrix codes of Table 9.5
K _{IC} , G _{IC}	Plane-strain fracture toughness; critical energy release rate
SC-1, SC-2, SC-3	Standard conditions of Chapter 15.0
TRL	Technology readiness level
[E], [R], [H], [T]	Evidence labels: established, reported, hypothesis, design target

Appendix B — Batch data sheet

One sheet per batch, stored with the raw files.

```
# Batch data sheet
Sample ID:                Parent lot / feedstock ID:
Material code (HCNS, G-HCNS, H-GO, H-rGO, GNP-R, GO-R):
Experiment ID:            Operator:                Date:

## Process
Route (SC-1 or variant):
Hydrothermal: acid ___ wt%, ratio ___ w/v, ___ °C, ___ h
Activation: KOH:char ___, ___ °C, hold ___ h, gas ___ mL/min
Graphitization / oxidation / reduction step and conditions:
Wash sequence:
Mass in ___ g   Mass out ___ g   Yield ___ %
Energy and reagent log file:

## Structure (ISO/TS 21356-1)
Thickness median / IQR (AFM, n=___):
Lateral size median / IQR (SEM, n=___):
Aspect ratio median:
Raman I_D/I_G @532 ___ @633 ___   FWHM(G) ___
XRD d002 ___ nm   Lc ___ nm   La ___ nm   g ___
BET ___ m²/g   Micropore share ___ %
Skeletal density ___ g/cm³   Envelope density ___ g/cm³

## Chemistry (ISO/TS 23359)
XPS C / O / N (at%):
TGA onset in air ___ °C   Residue at 900 °C ___ %
ICP: K ___ ppm   Fe ___ ppm   Mn ___ ppm   S ___ ppm

## Function
Powder conductivity at 10 / 50 / 100 MPa (S/m):

## Raw files and links
Paths or DOIs:
```

Appendix C — Reporting checklist

Tick every item before a result leaves the lab.

- ☐ Hypothesis and gate were time-stamped before testing

- ☐ Material described by layers or thickness, lateral size, order and chemistry, not by the word "graphene" alone
- ☐ Distributions reported, with sample counts
- ☐ Weight fractions converted to volume fractions with measured densities
- ☐ Dispersion evidence (micrograph or micro-CT) attached to every mechanical result
- ☐ Degree of cure and T_g reported with every mechanical result
- ☐ Reference materials (GNP-R, GO-R) tested alongside, under the same conditions
- ☐ Effect sizes with 95% confidence intervals
- ☐ Evidence label ([E], [R], [H], [T]) on every claim
- ☐ Raw data deposited with a DOI, negative results included
- ☐ Hempoxies results stated as TRL 1-2 unless a gate has been passed and replicated

Sources

Every source below was opened or retrieved while writing this book, as of the date in the byline. Grouped by topic.

Standards, vocabulary and metrology

- [ISO/TS 80004-13:2024, graphene vocabulary \(SCC\)](#)
- [ISO/TS 21356-1:2021, structural characterization \(SCC\)](#)
- [ISO/TS 23359:2025, chemical characterization \(SFS\)](#)
- [ISO 9277:2022, BET surface area \(StatNano\)](#)
- [ASTM D7028, DMA T_g of polymer matrix composites](#)
- [ASTM D2344, short-beam strength](#)
- [ASTM D5528, mode I interlaminar fracture \(ANSI\)](#)
- [ASTM D5229, moisture absorption](#)
- [ASTM C109 \(ANSI\); ASTM C349; ASTM C348](#)
- [VAMAS TWA 41 SEM/AFM project sheet](#)
- [EMPIR GRACE good practice guide, arXiv:2007.14047](#)
- [Bianco et al., graphene-family nomenclature, Carbon 2013 \(Nanowerk\)](#)
- [Kauling et al., worldwide graphene flake production, Adv. Mater. 2018; Physics World summary](#)

Hemp and biomass carbons

- [Wang et al., hemp carbon nanosheets, ACS Nano 2013](#)
- [US Patent 9,478,365; Google Patents](#)
- [US Patent 10,170,251; Google Patents](#)
- [US Patent 12,300,438](#)
- [Nanowerk spotlight on hemp nanosheets](#)
- [ScienceDirect topic: hemp fiber](#)
- [High-surface-area carbon from hemp bast, C 2018](#)
- [Hemp-derived graphene synthesis preprint](#)
- [Ding et al., peat-moss nanosheet frameworks, ACS Nano 2013; Ding PhD thesis](#)
- [Kai Cui author profile \(peanut-shell carbon nanosheets\)](#)
- [Singh et al., lignocellulosic biomass-derived graphene review, 2024](#)
- [Kong et al., graphene-like carbon from biomass pyrolysis](#)
- [Lignin-derived laser-induced graphene review, Green Chem. 2026](#)
- [Hemp anatomy and composition review; Gandolfi et al., hemp hurd analysis; Bokhari et al., hemp hurd](#)
- [Franklin, graphitizing and non-graphitizing carbons, 1951](#)
- [Structural models of non-graphitising carbon, C 2025](#)
- [Chen et al., Ni-catalysed graphitization of cellulose, 2018](#)
- [Thompson et al., iron-catalysed graphitization of biomass, Green Chem. 2015](#)
- [Precursor effects in iron-catalysed graphitization, Mater. Adv. 2020; Hunter, PhD thesis 2023](#)

Top-down and bottom-up production

- [Hernandez et al., liquid-phase exfoliation, 2008](#)
- [Paton et al., shear exfoliation, 2014; ChemistryViews](#)
- [Parvez et al., electrochemical exfoliation, 2014](#)
- [Jeon et al., edge-carboxylated graphene by ball milling, 2012](#)
- [Li et al., CVD graphene on copper, 2009](#)
- [Luong et al., flash graphene, Nature 2020; RePEc record; behind the paper](#)
- [Zhu et al., continuous low-carbon biomass flash graphene, 2024](#)
- [Peanut-shell flash graphene \(Graphene-Info\)](#)

- [Chen et al., lignin flash graphene/Fe₃O₄, 2023](#)
- [Advincula et al., coal flash graphene in epoxy, 2023](#)
- [Lin et al., laser-induced graphene, 2014](#)

Graphene oxide and reduction

- [Improved Hummers method as documented in US Patent 10,989,681](#)
- [Safer modified Hummers method review, 2019](#)
- [Ferrate-assisted Hummers method, Sci. Rep. 2016](#)
- [Structure of graphite oxide, arXiv:1008.0937](#); [EELS of GO, arXiv:1612.03594](#); [Oxidative debris, PMC6724119](#)
- [Ye et al., graphene quantum dots from coal, 2013](#); [Rice University via Nanowerk](#)
- [GO from waste sources, review 2024](#); [US Patent 11,731,875, methods for production of graphene oxide](#); [US Patent 11,021,370, graphene and GO from carbon-rich natural materials](#)
- [Manganese heptoxide data \(chemeurope\)](#)
- [HI reduction, PMC7948250](#); [ascorbic acid then thermal reduction](#); [comparative chemical reduction, Nanoscale 2023](#); [thiourea dioxide reduction, 2012](#)
- [GO reactivity, Nanoscale 2016](#)

Intrinsic properties

- [Lee et al., graphene strength and modulus, 2008](#); [strength caveat, arXiv:1903.07695](#)
- [Balandin et al., thermal conductivity, arXiv:0802.1367](#); [laminar values](#)
- [Suk et al., GO modulus, 2010](#)
- [Gómez-Navarro et al., reduced GO modulus, 2008](#)
- [GO composition report](#)

Polymer composites

- [Rafiee et al., ACS Nano 2009](#)
- [Atif, Shyha, Inam](#)
- [Imran and Shivakumar, three-roll-milled GNP epoxy](#)
- [Amine-functionalized GNP summary](#)
- [Zhou et al., amine-rich GO epoxy, 2015](#); [Paraskar et al., 2019](#)

- [Graphene-epoxy review, Polymers 2016](#); [TUHH dispersion comparison](#); [Oklahoma thesis](#)
- [Stankovich et al., Nature 2006](#); [US Patent 7,914,844](#)
- [Istrate et al., melt-processed PET/graphene, 2014](#)
- [Hemp/epoxy hybrid nanofillers](#); [hemp-fibre composites review, 2025](#)

Bio-epoxies and vitrimers

- [Manthey et al., epoxidized hemp oil, 2013](#); [acrylated EHO](#)
- [ZILA BioWorks \(ISC3\)](#); [425 Business](#); [EPA final report](#)
- [Vitimer minireview, Chem. Sci.](#); [Polymer Innovation Blog part 1](#), [part 2](#); [IISc thesis](#)
- [Sharma et al., epoxy/GO vitrimers, 2024](#)
- [Tyre-waste nanosheets in thiol-epoxy vitrimer, Polymers 2026](#)
- [UPES thesis, biomass carbon in vitrimers](#); [induction-healed vitrimers, 2025](#); [epoxy-vitrimer nanocomposites review, 2026](#)

Cement, ceramic, metal and coatings

- [Pan et al., GO-cement, 2015](#); [GO paste, 0.04 wt%](#); [GO mortar with recycled aggregate](#); [GNP cement](#); [GO hydration, 2024](#)
- [Graphene-toughened silicon nitride \(up to ~235% K_{1c}\), as reported in US Patent 8,962,504](#); [SiC/graphene \(CSiC\)](#); [Osendi abstract](#)
- [Süzer et al., Al-GNP, 2024](#); [Al₄C₃ discussion, Metals 2022](#)
- [Schriver et al., ACS Nano 2013, as cited in a Peeref reference list](#); [R&D World](#); [BN versus graphene barriers, 2016](#)

Modelling and characterization

- [King et al., 2013](#); [King et al., 2015](#)
- [Tarasovs and Aniskevich, arXiv:2202.03891](#)
- [Kinloch et al., ICCM 2015](#); [critical flake length by MD \(CORE\)](#); [Gong et al., arXiv:1007.1953](#)
- [Raman La relation as used in arXiv:1208.2439](#); [Ferrari and Robertson, 2000](#); [Malvern note on graphitization degree](#)

Safety, sustainability, supply and policy

- [NIOSH CIB 65, carbon nanotubes](#); [NIOSH graphene exposure study \(CompositesWorld\)](#)
- [Spanish LCA of Hummers and Marcano GO \(CORE\)](#); [Nottingham comparative LCA supporting information](#)
- [HempToday, Canadian hemp 2025](#); [Health Canada hemp statistics page](#)
- [Crux Investor on graphite controls](#); [Meticulous Research](#); [SMM on Announcement No. 58](#)

Earlier work by the author

- [Mitlinit priority declaration and research proposal, Zenodo, June 2026](#)
- [Groundbreaking Integration of Hemp-Derived Carbon Nanosheets in Composite Materials, February 2025](#)