

Hemp-Derived Furan Nanothreads: An Experimental Manual

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Status: TRL 1–2 research hypothesis. Every link in this chain is documented in the literature on its own, but no published work yet runs hemp biomass all the way to crystalline nanothreads. All yields and performance figures below are either cited literature values for the individual steps or projected design targets, never validated results for the integrated route.

Landry Industries Inc. · Hempoxies research platform · Moncton, New Brunswick

1. Introduction

The goal of this manual is a crystalline carbon nanothread whose every carbon atom traces back to a hemp stalk. The route is hemp hurd to furfural, furfural to furan, and furan to nanothreads by slow compression at room temperature.

Carbon nanothreads are one-dimensional, sp^3 -rich polymers with sub-nanometre widths and diamond-like cores. They are predicted to combine very high tensile strength and stiffness with polymer-like flexibility ([Fitzgibbons et al. 2015](#); [Huss et al. 2021](#)).

Why furan. Benzene-derived threads need onset pressures of 17 GPa or more, which limits scale. Furan is less aromatic, so it starts to polymerize at about 10 GPa, the lowest onset recorded for crystalline nanothreads ([HPCAT/APS](#)).

Why hemp. Furan is made industrially from furfural, and furfural comes from the pentose sugars in hemicellulose. Hemp hurd (shives) is a low-value by-product of fibre processing, and published work shows it yields furfural while keeping its cellulose intact for other products ([Brazdausks, RTU](#)). This makes a fully bio-based nanothread a plausible, testable hypothesis.

2. Background science

Furan reacts at lower pressure because it is barely aromatic, so its rings open more readily into a chain of $[4 + 2]$ (Diels–Alder) cycloadditions. All values below are from [Huss et al., ACS Nano 2021](#) unless noted.

Precursor	Resonance energy (eV per π electron)	Calculated initiation barrier (kcal/mol)	Onset of ordered polymerization
Benzene	~0.060	> 46	~18 GPa (HPCAT)
Pyridine	~0.060	> 46	23–30 GPa range reported for aromatics
Furan	0.007	35	~10 GPa

Mechanism. Two stacked furan rings undergo a [4 + 2] cycloaddition, creating sp³ carbons while leaving further polymerizable sp² segments. Repeating this along a column of stacked molecules builds a thread with a polar ether oxygen in each repeat unit.

Three thread types. Depending on which face each ring adds from, successive cycloadditions give syn, anti or mixed syn/anti threads. Simulated packings matched the experimental diffraction best for anti threads, but the real product is probably a mixture, either of thread types or of segments within one thread.

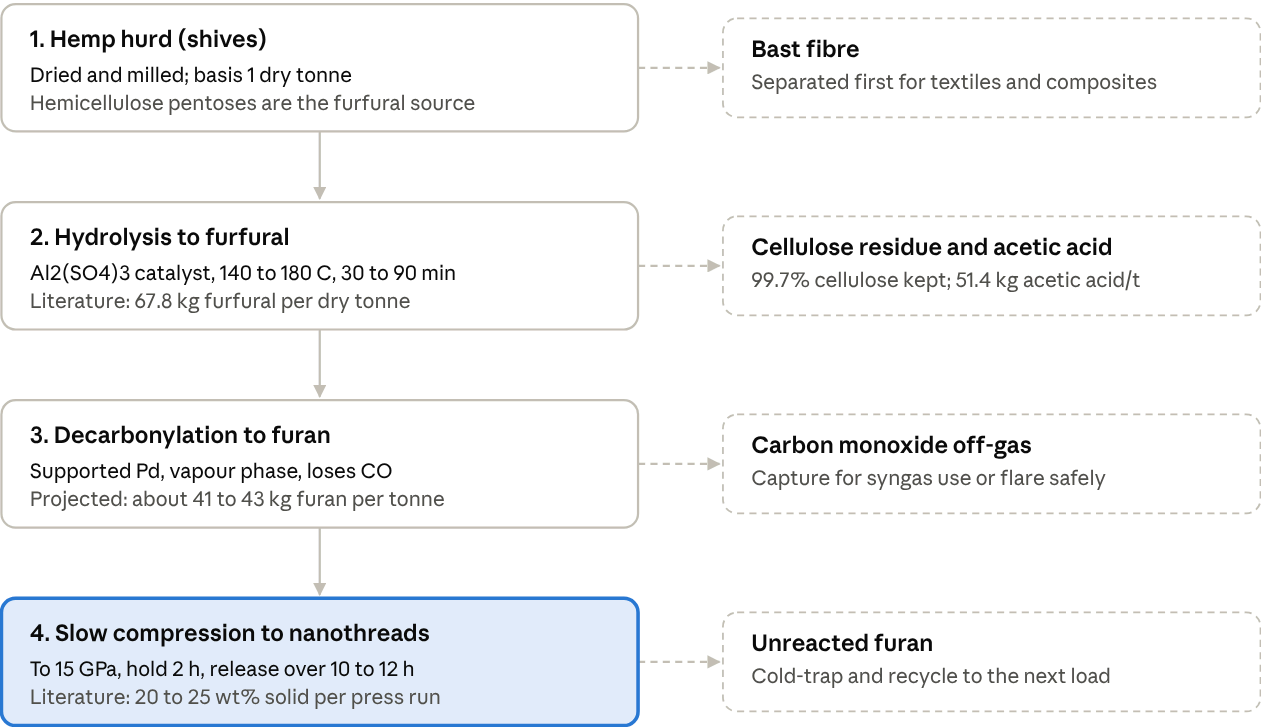
Why slow matters. An earlier study compressed furan quickly to 47 GPa and got an amorphous, heavily cross-linked solid. Slow compression to only 15 GPa gave the first crystalline furan threads. Rate control is therefore a core experimental variable, not a detail.

What the oxygen adds. The ether oxygens may help with post-processing such as intercalation with metals or Lewis acids and exfoliation. Mass spectrometry also showed the threads bind sodium strongly, which the authors suggest could open new processing routes.

3. Process overview

One dry tonne of hemp hurd could in principle supply about 41–43 kg of furan, but today's presses convert only milligrams per run. The chemistry scales; the final pressure step does not yet.

Hemp hurd becomes furan nanothreads in three chemical steps



process flow · 4 stages, co-products at right

Steps 2 and 3 are established industrial chemistry; step 4 is a specialist high-pressure laboratory technique.

Projected mass balance (per 1 dry tonne of hurd). Projected values are calculated from the cited literature figures, not measured for this route.

Stream	Amount	Basis
Furfural	67.8 kg	Measured for Al2(SO4)3 hydrolysis of hemp shives (RTU)
Acetic acid co-product	51.4 kg	Same study
Cellulose retained in residue	99.7% of initial	Same study
Furan, theoretical	48.0 kg	Projected: 67.8 kg × (68.07 / 96.08 g/mol)
Furan at 85–90% yield	40.8–43.2 kg	Projected, using yields claimed for vapour-phase decarbonylation (US9650354)

Stream	Amount	Basis
Nanothread solid at 20–25 wt%	8.2–10.8 kg	Projected, using the Paris–Edinburgh press yield (Huss 2021)
Press runs needed	~2.2 million	Projected: $43 \text{ kg} \div 0.936 \text{ g/mL} \div 21 \text{ }\mu\text{L per run}$

The last row is the honest bottleneck. Converting one tonne's worth of furan would take millions of press runs, so near-term work should target milligram-to-gram quantities for characterization and proof of bio-origin.

4. Experiment 1: Feedstock preparation and characterization

Objective: produce dry, milled, fully characterized hemp hurd, because hemicellulose content sets the ceiling on furfural yield.

Reference composition. Published hurd analyses fall in a narrow band, with hemicellulose the variable that matters most here.

Source	Cellulose	Hemicellulose	Lignin	Other
Review range (Materials 2014)	40–48%	18–24%	21–24%	—
Same study, measured sample	44.5%	32.78%	21.03%	Ash 3.04%
Carmagnola hurd (BioResources 2013)	44.0% (alpha)	25.0%	23.0%	Extractives 4.0%, ash 1.2%
Decorticated hurd (Penn State)	45.66%	24.57%	21.67%	—

Procedure

1. Source decorticated hurd from a single, documented cultivar and harvest; record variety, field location and harvest date.
2. Remove residual bast fibre and dust by sieving.
3. Dry at 105 °C to constant mass and record moisture content.
4. Mill and sieve to a defined particle-size fraction; record the fraction used, because particle size affects hydrolysis kinetics.

5. Store sealed and dry; label each lot with a batch ID that follows it through every later experiment.

Characterization (per lot)

- Cellulose, hemicellulose and Klason lignin by standard wet-chemical methods.
- Xylose content of the hemicellulose fraction, which is the direct furfural precursor.
- Ash and extractives.
- Retain a sealed reference sample of each lot for later radiocarbon comparison (Experiment 4).

Record: lot ID, cultivar, moisture %, particle size, composition, mass in and out.

Open question: confirm current Health Canada requirements for possessing and processing hemp stalk material before sourcing.

5. Experiment 2: Furfural from hemp hurd

Objective: convert the hurd's pentosans to furfural by steam hydrolysis with aluminum sulfate, choosing between a furfural-maximizing mode and a cellulose-saving cascade mode. Method and numbers follow [Brazdausks, RTU doctoral thesis 2019](#) on Bialobrzeskie hemp shives.

Theoretical ceiling. Those shives held 17.6% pentosans (14.6% xylan + 3.0% arabinan), giving a theoretical 125 kg furfural per dry tonne. Furfural yield (% of theoretical) = furfural obtained ÷ [(arabinose + xylose) ÷ 1.563], where 1.563 is the pentose-to-furfural molecular weight ratio (150.13 / 96.08).

Two validated operating modes

Mode	Temperature	Al ₂ (SO ₄) ₃ (% of dry mass)	Time	Furfural yield	Cellulose
A. Furfural maximum	180 °C	5.95%	60 min	71.8% of theoretical (9.0% of dry mass)	Heavily degraded
B. Cascade (cellulose kept)	164 °C	7%	30 min	54.3% of theoretical (6.8% of dry mass)	99.7% retained

For a nanothread-focused run, Mode A gives about a third more furfural. For a biorefinery that also wants cellulose (for example, Hempoxies fibre or ethanol), Mode B is the default and is the basis of the mass balance in Section 3.

Apparatus (as published): vertical steam-jacketed hydrolysis reactor, 110 mm internal diameter, 13.7 L working volume, automatic temperature control, condenser on the steam outlet. Catalyst is mixed into the shives in a paddle mixer before loading.

Procedure

1. Fractionate shives to 3–12 mm at about $20\% \pm 2\%$ moisture.
2. Mix aluminum sulfate octadecahydrate onto the shives at the chosen loading; the published constant catalyst solution concentration was 16%.
3. Load about 1,200 g dry-basis shives into the pre-heated reactor; reach set conditions within 2 minutes and start the clock.
4. Pass continuous steam at about 200 mL/min (condensate basis) through the bed.
5. Collect condensate in 10-minute fractions; record each volume.
6. Discharge the solid residue with steam, cool, weigh and dry to $10\% \pm 1\%$ moisture.

Analysis

- Furfural in each fraction by potassium bromide–bromate titration (as published) or by HPLC.
- By-products in the hydrolyzate: formic, acetic and levulinic acids and HMF.
- Residue: cellulose, xylan and residual catalyst. The thesis estimates 8.2–8.8% aluminum sulfate remains in the residue, so wash it before using the residue elsewhere.

Furfural recovery. The condensate is dilute (2.3–4.6 g/L furfural in the published runs). Recover furfural by distillation and phase separation from water, then dry and redistill before Experiment 3. Record final purity by GC.

Watch points. The thesis notes furfural decomposes markedly in the reactor during the first 30 minutes, so steam velocity in that window is an optimization target. Seasonal storage of hurd may also shift composition.

6. Experiment 3: Furfural decarbonylation to furan

Objective: remove CO from hemp-derived furfural over supported palladium to give furan ($\text{C}_5\text{H}_4\text{O}_2 \rightarrow \text{C}_4\text{H}_4\text{O} + \text{CO}$). The bench protocol below follows the vapour-phase examples in [US8653287 \(DuPont, now Covation\)](#), which name hemp among suitable furfural sources.

Reported bench results (290–310 °C, H₂:furfural = 2:1)

Catalyst (0.5% Pd, 3 mm pellets)	Initial conversion	Furan selectivity	Productivity reached
Pd/alumina, unpromoted	99%	83% at start	7.87 kg furan/g Pd at 126 h, conversion falling
Pd/alumina + Cs ₂ CO ₃	99.2%	94.1% rising to 98.6%	26.3 kg furan/g Pd at 171 h
Pd/lithium aluminate + Cs ₂ CO ₃	100%	95–97.6%	42.7 kg furan/g Pd at 287 h, still 92% conversion

The cesium-promoted lithium aluminate catalyst is the recommended starting point: it fouled least and suppressed hydrogenation side products such as THF.

Catalyst promotion (from the patent)

1. Immerse the Pd catalyst pellets in 7.5% aqueous Cs₂CO₃ (example: 20.3 g catalyst in 20 mL) and agitate gently for 5 h at room temperature.
2. Filter, rinse three times with deionized water and air dry.
3. Oven dry at 120 °C for 2 h, then calcine at 300 °C for 4 h.

Reactor and run

1. Load about 2 g catalyst on a mesh support in a 45.7 cm × 1.3 cm o.d. 316 stainless tube reactor with an internal thermocouple, at atmospheric pressure.
2. Condition under N₂, ramp to 290 °C over 2 h while flowing H₂ at 15 mL/min.
3. Feed freshly distilled hemp furfural at 2.0 mL/h with 3 wt% water and 0.5 wt% dodecane as internal standard; set H₂ to 17 mL/min (2:1 molar).
4. Condense product gas in a chilled (–10 °C) trap; vent CO through a monitored, ventilated line.
5. Analyze by GC-FID/MS for conversion and selectivity to furan, THF, 2-methylfuran and furfuryl alcohol.
6. When conversion drops, raise the temperature in 20 °C steps (310 then 330 °C), as in the patent examples.

Critical inputs from Experiment 2. The patent stresses that furfural must be freshly distilled, because it oxidizes to high-boiling products. Hemp furfural will also carry traces of acetic and formic acids and HMF; test whether these accelerate catalyst fouling by running a commercial-furfural control side by side.

Record: furfural lot ID, catalyst lot, hours on stream, conversion, selectivity, kg furan per g Pd, crude furan mass.

7. Experiment 4: Furan purification and bio-origin verification

Objective: deliver furan of at least 99% purity, matching the reagent grade used in the published nanothread work, and prove by radiocarbon that its carbon is hemp-derived rather than fossil.

Purification

1. Fractionally distill the cold-trapped crude product. Furan boils at 31.3 °C ([ICSC 1257](#)), well below THF, 2-methylfuran and unreacted furfural, so a short column separates it cleanly.
2. Collect into a chilled, dry, inert-gas-flushed receiver.
3. Confirm purity by GC-FID and identity by GC-MS and ¹H NMR; record water content.
4. Store cold, dark and under inert gas. Furan can form unstable peroxides on standing in air ([ChemicalBook](#)), so use fresh material and test for peroxides before any concentration or loading step.
5. If a stabilizer is added for storage, remove it before Experiment 5. The published method filtered commercial furan through basic alumina immediately before loading to strip its BHT inhibitor ([Huss 2021](#)).

Bio-origin verification (ASTM D6866)

[ASTM D6866](#) measures biobased carbon content by radiocarbon analysis; Method B (AMS with IRMS) has a stated maximum total error of ±3% absolute. Fossil carbon contains no ¹⁴C, so a hemp-derived furan should read near 100% biobased, while petrochemical furan reads near 0%.

Sample	Why test it	Expected result
Hurd reference (from Experiment 1)	Baseline for this lot	~100% biobased
Purified furan	Proves the precursor is hemp-derived	~100% biobased (hypothesis)
Nanothread solid (Experiment 5)	The headline claim; a solid is easier to ship than volatile furan	~100% biobased (hypothesis)

Sample	Why test it	Expected result
Commercial furan control	Confirms the method distinguishes sources	Measured value reported by lab

Use a laboratory that keeps biobased samples isolated from artificial ^{14}C ; the standard warns that contamination from tracer work can inflate results.

Record: furan lot ID, distillation cut temperatures, GC purity, peroxide test result, D6866 certificate numbers.

8. Experiment 5: Pressure-induced polymerization to nanothreads

Objective: reproduce the published furan-nanowire synthesis using hemp-derived furan, first in a diamond anvil cell (DAC) for in situ proof, then in a Paris–Edinburgh (PE) press for milligram samples. All conditions are from [Huss et al., ACS Nano 2021](#).

Hypothesis (TRL 1–2): hemp-derived furan of $\geq 99\%$ purity will polymerize at the same ~ 10 GPa onset and give the same near-hexagonal diffraction as reagent furan. A measurable difference would point to trace impurities carried through from hydrolysis.

Design: run hemp furan and commercial $\geq 99\%$ furan side by side under identical programs. The commercial run is the positive control.

Track A — Diamond anvil cell (nanogram scale, in situ)

Equipment as published: symmetric DAC with 500 μm culets or spherical-seat DAC with 250 μm culets; stainless steel gaskets indented to 45–55 μm with a 100–160 μm sample hole; double-stage gas membrane drive with digital controllers; ruby chips for pressure calibration by fluorescence.

1. Filter furan through basic alumina immediately before loading.
2. Load liquid furan into the gasket hole at room temperature with a ruby chip. For a powder sample, freeze a small amount, place it over the hole and crush it in with the anvils. For single crystals, freeze under pressure, melt back to a few crystallites, then slowly re-compress to grow them.
3. Run the pressure program below at 298 K.
4. Monitor in situ by Raman (633 nm laser, 100 μW or less to avoid side reactions) and, where beamtime allows, synchrotron XRD.

5. Open the cell carefully; the published product was an off-white solid filling the chamber.

Pressure program (compression; decompression mirrors it)

Stage	Pressure range	Rate
1	Ambient to 8 GPa	4.2 GPa/h
2	8 to 11 GPa	1.8–2.4 GPa/h
3	11 to 15 GPa	0.6–1.2 GPa/h
Hold	15 GPa	2 h
Release	15 GPa to ambient	Same rates in reverse, over 10–12 h

What to expect: furan freezes near 1.4 GPa. A new diffraction peak appears at about 10 GPa and grows to 15 GPa. Raman fluorescence rises around 11.4 GPa, and furan Raman modes vanish by about 15.1 GPa.

Track B — Paris-Edinburgh press (milligram scale)

1. Load 21 μ L of filtered furan into an encapsulated stainless steel gasket, freezing it with liquid nitrogen so the gasket fills with no trapped air; the boil-off nitrogen also excludes oxygen and water.
2. Mount in a V7 PE press with double-toroid polycrystalline diamond anvils, driven by an automatic oil syringe pump.
3. Ramp to about 15 GPa sample pressure (published: 692 bar oil), slowing to 1 bar/min above 547 bar oil, on both compression and decompression.
4. Recover 4–5 mg solid per run (20–25% mass yield).

The PE press gives about 5,000 times more product than a DAC, but its pressure is less uniaxial, which may favour side reactions and more disorder.

Variables worth exploring (each is a forward-looking hypothesis): hold time at 15 GPa; slower final-stage rates; the 1.5 GPa-to-ambient release, where long-range order was lost in the published work; and mild heat or light to lower onset pressure, which the original authors suggested as future work.

Record: furan lot, gasket and anvil IDs, full pressure–time log, ruby readings, Raman and XRD files, recovered mass, appearance.

9. Characterization: what counts as a nanofiber

A product counts as furan nanofibers only if it matches the published reference signatures on diffraction, vibrational spectra and mass spectrometry, and is 100% biobased by radiocarbon. Reference values are from [Huss et al. 2021](#).

Method	Reference signature for furan nanofibers	Pass criterion
Single-crystal XRD, in situ at 1.5 GPa	Near-6-fold pattern; d-spacings 4.75, 4.85 and 4.90 Å	Sharp near-hexagonal spots at matching d-spacings
XRD after full release to ambient	Lumpy ring at ~5.05 Å; vestigial spots at 5.09–5.24 Å	Ring present at ~5.0 Å
XRD / WAXS of PE-press solid	Sharp d-spacing at 4.96 Å with second order at 2.48 Å	Same ring positions
In situ Raman	Fluorescence rise ~11.4 GPa; furan modes gone by ~15.1 GPa	Same onset window
FTIR of recovered solid	Strong sp ³ C-H stretch 2800–3060 cm ⁻¹ ; C-O-C wag and breathing bands 450–760 cm ⁻¹	sp ³ C-H dominant; low-frequency thread bands present
MALDI FT-ICR MS	Series spaced 68.02 Da (one furan unit); signal to ~6–7 kDa (~100 rings); sodiated ions dominant	68.02 Da ladder observed
XPS	Largely saturated carbon; C/O ratio 6.2:1	Report C/O; compare
Polarized light (PE samples)	Birefringence, consistent with 1D structure	Birefringence observed
ASTM D6866 (Experiment 4)	n/a (new to this route)	~100% biobased carbon

Side-product flags. Weak sp² C-H (3060–3180 cm⁻¹), C=C near 1610 cm⁻¹, O-H near 3470 cm⁻¹ and C=O near 1718 cm⁻¹ were attributed to unreacted furan, oligomers, defects or ring opening. Track their intensity relative to the control; an increase in the hemp run would point to impurities.

Structure assignment. Compare IR bands with the computed syn, anti and syn/anti spectra. The published data fit best with anti packings but are consistent with mixtures.

Mechanical testing is out of scope at this stage: the published furan work reports no tensile measurements, and any strength figures in Hempoxies documents must remain projected targets.

10. Safety

Furan is the highest-risk material in this chain: extremely flammable, near-boiling at room temperature and classed as possibly carcinogenic. Every step involving it needs a fume hood, cold handling and ignition control. Read the current SDS for every chemical before work; this section does not replace a site risk assessment.

Hazard	Key facts	Controls
Furan flammability	Boiling point 31.3 °C; flash point –35 °C; vapour/air mixtures explosive (ICSC 1257)	Closed systems, explosion-proof equipment, no ignition sources, non-sparking tools, chilled handling
Furan toxicity	IARC Group 2B; NTP "reasonably anticipated" human carcinogen; GHS Carc. 1B, acute toxic by ingestion and inhalation (SDS); German MAK 0.02 ppm with skin notation (ICSC)	Fume hood at all times, chemical-resistant gloves, avoid all skin contact, minimal quantities
Furan peroxides	May form unstable peroxides in air (ChemicalBook)	Store cold, dark, under inert gas; test before distillation; use fresh
Carbon monoxide (Experiment 3)	One mole CO released per mole furan made	Vented, monitored line; CO detector in the room
Hydrogen co-feed	Flammable gas at 290–330 °C reactor	Leak-checked lines, flow limits, inert purge before and after
Steam hydrolysis (Experiment 2)	140–180 °C pressurized steam	Pressure-rated reactor, relief valve, trained operators

Hazard	Key facts	Controls
Furfural	Toxic, oxidizes on standing	SDS controls; store sealed, distill fresh
High pressure (Experiment 5)	DAC and PE press operate to 15 GPa; anvils can fail	Trained high-pressure staff only, shielding, follow host facility rules
Cryogenics	Liquid nitrogen used in PE loading	Cryo gloves, face shield, ventilation

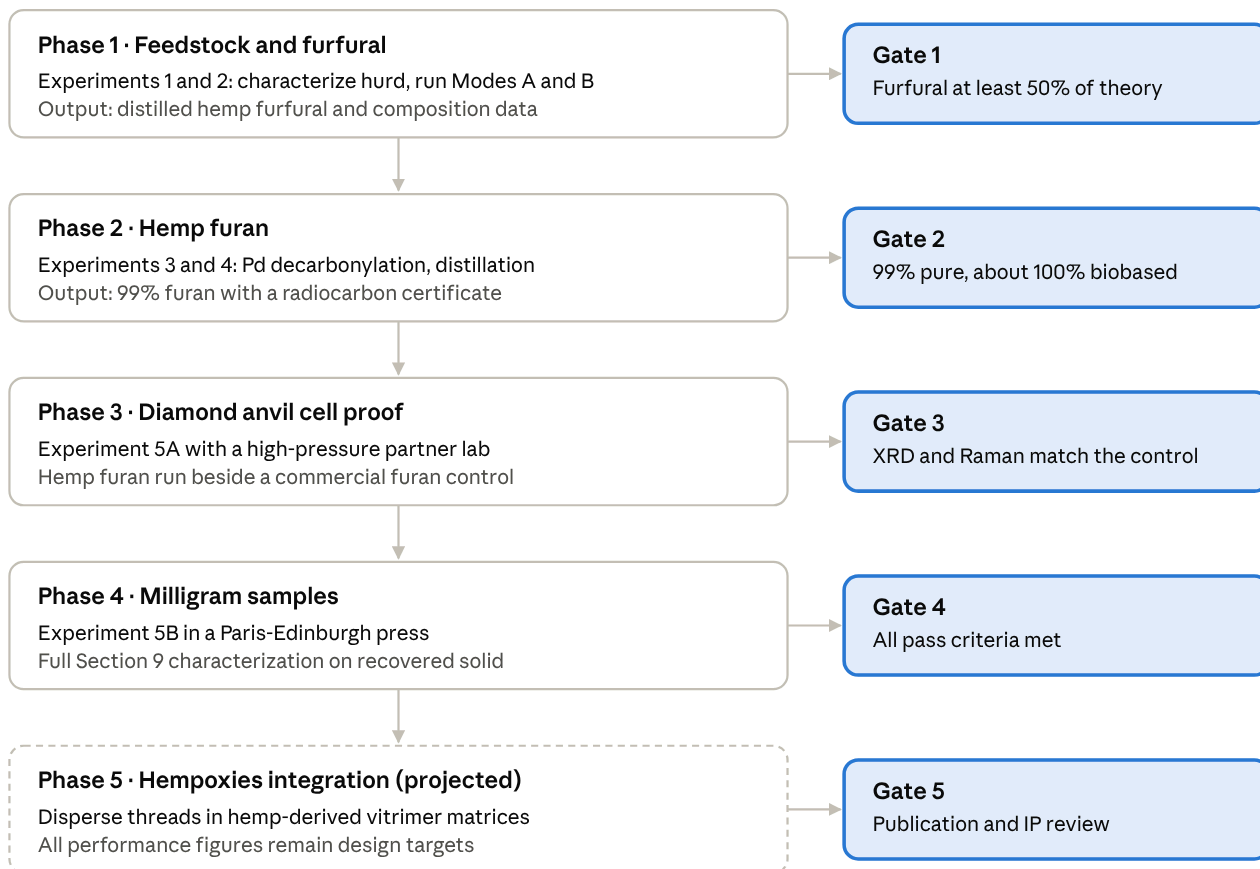
Waste. Collect furan- and furfural-contaminated waste as flammable, toxic organic waste. Wash aluminum-sulfate-bearing residues before reuse or disposal.

Facility recommendation. Experiments 1–4 fit a standard ventilated chemistry lab with the controls above. Experiment 5 should be run with an established high-pressure group rather than built in-house; the published work came from Penn State's NSF Center for Nanowire Chemistry with synchrotron work at HPCAT, Argonne.

11. Hypotheses, open questions and roadmap

The central claim to test is that a crystalline nanowire can be made entirely from hemp carbon. It stands or falls on four hypotheses, tested in order.

1. **H1 — Furfural.** Hemp hurd from a documented regional supply gives furfural yields within the published 54–72% of theoretical range.
2. **H2 — Furan.** Hemp furfural decarbonylates over Cs-promoted Pd at selectivities comparable to the 95–98% reported for commercial furfural, despite trace hydrolysis by-products.
3. **H3 — Threads.** Hemp furan of $\geq 99\%$ purity polymerizes with the same ~ 10 GPa onset and diffraction signature as reagent furan.
4. **H4 — Origin.** The recovered solid reads $\sim 100\%$ biobased by ASTM D6866, making it a verifiably bio-based carbon nanowire.

Each phase must pass its gate before the next begins

research roadmap · 5 phases, 5 gates

Phase 5 is drawn dashed because it is the most speculative: the ether oxygens in furan threads may aid dispersion and interfacial bonding in a matrix, but that is an untested idea.

Open questions

- Which high-pressure group would host Experiment 5, and on what terms?
- Do acetic acid, formic acid or HMF traces in hemp furfural shorten Pd catalyst life?
- Can photochemistry, heat or catalysis lower the 10 GPa onset, as the original authors proposed?
- How can the 1.5 GPa-to-ambient loss of order be reduced?
- Freedom to operate: US8653287 on Pd/metal aluminate decarbonylation is listed as active until 2030 on Google Patents, so any commercial use of that catalyst route needs a patent review.

Novelty position. Each step is published separately; the integrated hemp-to-nanowhread route, with radiocarbon proof of origin, does not appear in the sources reviewed for this manual. That is a claim to re-verify with a formal literature search before publication.

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