

HEMPOXIES 44 “GENESIS”

Laboratory-Ready Standard Operating Procedure Package

Landry Industries Inc. · Hempoxies Materials Science Platform
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Design Targets	Document Scope
Tensile Strength ≥ 1500 MPa	SOP-H44-01 Safety & Hazards
Tensile Modulus ≥ 120 GPa	SOP-H44-02 Equipment & Materials
Mode-I GIc > 1400 J/m ²	SOP-H44-03 Batch Calculations
Bio-Based Content ≥ 85 wt%	SOP-H44-04 Fabrication Procedures
	SOP-H44-05 Curing Protocol
	SOP-H44-06 Characterization & Data Sheets

⚠ All performance figures are pre-experimental design targets. No experimental data exist at time of publication (OSML-2026).



SOP-H44-01 — Safety, Hazards & Emergency Procedures

1.1 Minimum PPE Requirements (ALL procedures)

PPE Item	Specification	When Required	Check ☐
Nitrile gloves (inner)	Chemical-resistant, 0.15 mm min	Always	✓
Neoprene over-gloves (outer)	Solvent & acid resistant	Steps 1–3	✓
Lab coat / Tyvek suit	Full-length, flame-resistant preferred	Always	✓
Safety goggles (splash)	ANSI Z87.1 rated	Always	✓
Face shield	Steps involving sonication, acids	Steps 2–3	✓
Respiratory protection (P100/OV)	Sonication, resin blending	Step 2	✓
Closed-toe shoes / boot covers	Chemical resistant	Always	✓

1.2 Chemical Hazard Summary

Chemical / Process	GHS Hazard	GHS Pictogram	SDS Ref.	Control Measure
Epoxidized Hempseed Oil (EHO)	Skin/eye irritant	GHS07	SDS-EHO	Ventilated hood
Hemp-Furanic Benzoxazine (HF-BX)	Irritant; thermal decomp. fumes	GHS07/GHS02	SDS-HFBX	Hood + OV respirator
Sebacic Acid	Mild irritant	GHS07	SDS-SA	Dust mask if powder

DMF (CQD synthesis)	Reproductive toxin (Repro. Cat.1B)	GHS08/GHS06	SDS-DMF	Closed system; glove box if possible
Citric Acid	Irritant	GHS07	SDS-CA	Gloves + goggles
HCTP (hexachlorotriphosphonitrile)	Toxic; corrosive	GHS06/GHS05	SDS-HCTP	Glove box; HCl gas risk on hydrolysis
KOH (HDCNS activation)	Corrosive; severe burns	GHS05/GHS06	SDS-KOH	Face shield + neoprene gloves
N ₂ gas (pyrolysis/inert atmosphere)	Asphyxiant	GHS04	SDS-N2	O ₂ monitor in room; ventilation
Autoclave (200°C, 12 h)	Pressure vessel; thermal burn risk	—	—	Certified autoclave operator; pressure log
Tip sonicator (400 W, 20 kHz)	Acoustic hazard; aerosol generation	—	—	Acoustic enclosure; closed vessel

⚠ CRITICAL — DMF Handling

- DMF is a Category 1B reproductive toxin. Use only in a certified chemical fume hood.
- Wear double nitrile gloves; change after every 30 min of contact.
- Never use DMF near open flames — flash point 58°C.
- Dispose of DMF waste as halogenated/non-halogenated organic waste per institutional SOP.
- Pregnancy: inform supervisor; re-assign DMF steps where possible.

⚠ CRITICAL — HCTP Handling

- HCTP hydrolyses to release HCl gas on contact with moisture. Keep strictly anhydrous.
- Weigh inside dry glove bag or dry glove box.
- Immediately seal waste in labelled secondary container with desiccant.
- Emergency: HCl gas — evacuate, activate fume alarm, administer fresh air; seek medical attention.

1.3 High-Temperature Process Safety

Process	Pre-Check	Monitoring	Post-Process	Sign ☐
Autoclave (200°C, 12 h)	Certified vessel inspection ≤ 12 months	Pressure log sheet completed	Slow depressurize; cool to <50°C before opening	✓
Tube furnace — pyrolysis 600°C	N ₂ purge >10 min before ramp	Exhaust scrubber operational	Allow furnace to cool <100°C before removing samples	✓
Tube furnace — KOH 800°C	Separate furnace from CQD synthesis	K ₂ O fume capture	HF-rated tongs; face shield	✓
Carbonization — hemp bast 900°C	N ₂ purge verified; tube integrity checked	CO alarm active in room	Never open hot tube	✓
Oven cure — 80/120/180°C	Calibrated oven; thermocouple verified	Ventilation to atmosphere	Cool to RT before removing; use oven mitts	✓

1.4 Emergency Response

Emergency	Immediate Action	Secondary Action	Reviewed ☐
Chemical splash	Eyewash station — 15 min	Call campus EHS	✓

(eyes)	continuous flush	immediately	
Chemical spill (large)	Evacuate; activate spill kit; call EHS	Do not re-enter without PPE assessment	✓
Fire (resin/solvent)	CO ₂ or dry chemical extinguisher — NO WATER on solvent	Evacuate; pull alarm	✓
HCl gas release	Evacuate immediately; activate alarm; fresh air	EHS + HAZMAT if uncontrolled	✓
Autoclave overpressure	Do not approach; evacuate room; contact facilities	Wait for licensed engineer	✓
N ₂ asphyxia	Remove victim to fresh air; CPR if unresponsive; call 911	O ₂ monitor alarm response protocol	✓
Burn (thermal)	Cool running water 20 min; do not remove clothing stuck to burn	Seek medical attention >2 cm ²	✓



SOP-H44-02 — Equipment Checklist & Materials Procurement

2.1 Major Equipment — Pre-Experiment Verification

Equipment	Spec / Notes	Pre-Use Check	OK ☐
Tip sonicator	400 W, 20 kHz, with ice bath fixture	Calibrate amplitude; check probe tip integrity	✓
Tube furnace #1	≥ 650°C; N ₂ inlet + exhaust scrubber	Verify thermocouple calibration; purge cycle test	✓
Tube furnace #2	≥ 850°C; KOH-resistant tube (Inconel/Al ₂ O ₃)	Separate from furnace #1; base fume capture	✓
Autoclave / pressure vessel	≥ 500 mL, rated ≥ 10 MPa at 200°C	Pressure cert. current; PTFE liner clean	✓
Vacuum oven	80°C capable; 50 mbar vacuum	Calibrate gauge; vacuum pump oil fresh	✓
Analytical balance	0.0001 g resolution	4-point calibration; tare verified	✓
Brookfield DV-II+ viscometer	With appropriate spindle for 100–1000 mPa·s	Spindle calibration fluid check	✓
RTM press / hand lay-up mold	≥ 0.6 MPa capability; 40°C-rated platens	Mold release agent applied; pressure gauge cal.	✓
Programmable cure oven / press	RT → 200°C; ±2°C control; 2°C/min ramp	Temperature profile programmed; thermocouple in mold	✓
DEA (dielectric analysis) system	In-mold fiber-optic or embedded sensor	Sensor placement plan ready; baseline verified	✓
Dialysis membrane	MWCO 1 kDa, 1 m length	Check for pinholes; pre-wetted in DI water	✓
Ball mill / vibratory mill	For HICF D ₅₀ ≈ 2 μm	Mill media (ZrO ₂); clean; dry	✓
Fume hood (primary)	≥ 1.0 m width; sash velocity ≥ 0.5 m/s verified	Airflow alarm functional	✓
DSC — TA Q2000	Calibrated with In + Zn standards	Hermetic pan press; N ₂ purge active	✓
DMA — TA Q800	3-point bend fixture	Clamp torque spec; calibration weight verified	✓
TGA — TA Q500	N ₂ and air gas lines connected	Platinum pan tared; gas flow rates set	✓

2.2 Materials Procurement List — Trial Batch (~500 g matrix resin + 2× 300×300 mm panels)

Material	Supplier / Source	Qty (trial batch)	QC Notes	Received ☐

Epoxidized Hempseed Oil (EHO)	Naturally Splendid Enterprises (BC) / Hemptown Organics	125 g	OOC $\geq$ 5.9 wt%; EEW $\approx$ 340 g/mol	☐
Hemp-Furanic Benzoxazine (HF-BX)	Custom synthesis (furfurylamine + hemp-phenol)	100 g	DSC ring-opening onset 110–130°C; confirm by DSC before use	☐
Sebacic Acid ($\geq 99\%$)	Bioamber, Samia ON / Croda Canada	85 g	Dry 80°C/12h before use; MW = 202.25 g/mol	☐
Citric Acid (anhydrous, $\geq 99\%$)	Sigma-Aldrich / Fisher Sci.	5 g	CQD precursor; store desiccated	☐
HCTP (hexachlorotripropyl phosphonitrile)	Sigma-Aldrich 377295 or equiv.	2.5 g	Anhydrous; store in glove bag with desiccant	☐
DMF (anhydrous, $\geq 99.8\%$)	Sigma-Aldrich 227056	100 mL	Molecular sieves 4Å; reproductive toxin protocol	☐
Hemp Hurd (dried, milled)	Canadian hemp supplier (SK/AB)	50 g	Moisture <5%; for HDCNS Gen II pyrolysis	☐
Hemp Bast Fiber (long)	Prairie Hemp Council growers (SK)	30 g	For HICF carbonization at 900°C	☐
HCNFs (hemp cellulose nanofibers)	Alberta Innovates pilot / Canfor BC	15 g	Dry 80°C/24h before dispersion; confirm aspect ratio >100 by TEM	☐
KOH ($\geq 85\%$ pellets)	Fisher Sci. / Sigma-Aldrich	150 g	HDCNS activation; corrosive; store in sealed bottle	☐
PAN Carbon Fiber — UD prepreg or dry	Toray Composites Canada / SGL Carbon Montreal	~600 g (2 panels)	T300 or T700 grade; 0°/90° $\pm$ 45° ply schedule	☐
Hemp Bast Woven Fabric (2×2 twill)	Prairie Hemp Council (SK)	~400 g (2 panels)	Washed + dried 80°C/12h; areal weight ~200 g/m ²	☐
Mold release agent	Frekote 700-NC or equiv.	50 mL	Apply 3 coats + cure per TDS before first panel	☐
Dialysis tubing — MWCO 1 kDa	Sigma-Aldrich D9652 or equiv.	1 m	Pre-wet; check integrity before CQD purification	☐
N₂ gas (99.999% purity)	Praxair / Air Liquide Canada	1× K-cylinder	Pyrolysis + resin blending inert atmosphere	☐
DI water (18.2 MΩ·cm)	In-house Millipore / Barnstead	5 L	CQD purification dialysis bath	☐



SOP-H44-03 — Batch Calculation Worksheet

Instructions: Fill in the yellow-shaded input fields. Verify all calculated fields match before proceeding to resin blending. Keep this sheet with the batch record.

3.1 Matrix Resin — Stoichiometry (500 g total matrix target)

Parameter	Formula / Basis	Value	Your Measured Value
Total matrix batch mass	Target	500 g	_____ g
EHO (25 wt%)	0.25×500	125.0 g	_____ g
HF-BX (20 wt%)	0.20×500	100.0 g	_____ g
Sebacic Acid (17 wt%)	Stoich. + EHO-EEW calc.	85.0 g	_____ g
	125 / 340 g/mol $\times$		

→ EHO oxirane mol (nox)	1000	0.368 mol	_____ mol
→ Sebacic mol required	$\text{nox} \times 1.05 / 2$	0.193 mol	_____ mol
→ Sebacic mass required	0.193×202.25 g/mol	39.0 g ⚠ check	_____ g
⚠ NOTE: Remaining 46 g sebacic from '17 wt%' accounts for HF-BX co-reaction excess. Confirm with PI before use.			
N,P-CQDs (1.5 wt%)	0.015×500	7.5 g	_____ g
HCNFs (3 wt%)	0.03×500	15.0 g	_____ g
HDCNS Gen II (3 wt%)	0.03×500	15.0 g	_____ g
HICF (3 wt%)	0.03×500	15.0 g	_____ g
TOTAL (should sum to 500 g)	Check sum	500.0 g ✓	_____ g

3.2 CQD Synthesis — Solvothermal (per batch, ~0.5 g product yield estimated)

Parameter	Prescribed	Actual	Notes
Citric Acid	1.000 g	_____ g	MW = 192.12 g/mol
HCTP	0.500 g	_____ g	Weigh in glove bag; transfer quickly
DMF (anhydrous)	20.00 mL (20.00 g at 0.944 g/mL)	_____ mL	Measure in graduated cylinder in hood
Autoclave PTFE liner	≤ 70% fill (liner vol. ≥ 30 mL)	Liner vol: _____ mL	Never exceed 70% fill
Autoclave setpoint	200°C / 12 h	Actual: _____°C / _____ h	Log pressure relief if triggered
Dialysis MWCO	1 kDa; 48 h DI water bath; change water ×4	Date started: _____	Confirm by DLS: target 3–7 nm

3.3 HDCNS Gen II Synthesis — Pyrolysis + KOH Activation

Parameter	Prescribed	Actual / Measured	Notes
Hemp hurd feedstock	Dry at 110°C / 2h first; record initial mass	_____ g	Moisture loss: _____ %
Pyrolysis: Temp / Time / Gas	600°C / 2 h / N ₂ (100 mL/min)	_____°C / _____ h	Ramp: 5°C/min; char yield ~25–35%
Char mass (after pyrolysis)	Calculated: ~12–17 g from 50 g hurd	_____ g	Record; cool in N ₂
KOH:char ratio	3:1 by mass	KOH: _____ g	Char mass × 3
Activation: Temp / Time / Gas	800°C / 1 h / N ₂ (100 mL/min)	_____°C / _____ h	Separate furnace from CQD batch
Wash post-activation	DI water wash ×5 until neutral pH	Final pH: _____	Target pH 6–7; dry 110°C
BET surface area target	> 400 m ² /g	Measured: _____ m ² /g	Run BET before use in formulation

3.4 HICF Synthesis — Hemp Bast Carbonization

Parameter	Prescribed	Actual / Measured	Notes
Hemp bast feedstock	Washed; dried 80°C / 24 h	_____ g feed	Pre-weigh before carbonization
Carbonization: Temp / Time / Gas	900°C / 2 h / N ₂ (100 mL/min)	_____°C / _____ h	CO alarm active in room
Carbon yield	~20–30% of dry mass	_____ g yield	Record yield for batch record
	D ₅₀ ≈ 2 μm (laser)	D ₅₀ measured:	Use ZrO ₂ media; dry

Milling target	diffraction)	_____ μm	mill 2 h
Post-mill storage	Sealed vial; N ₂ backfill; dry $\leq$ 5% RH	Storage date: _____	Use within 30 days



SOP-H44-04 — Detailed Fabrication SOPs

STEP 1 — Component Preparation

#	Procedure — STEP 1	Done
1.1	Weigh HCNFs: _____ g (target 15.0 g). Place in vacuum oven at exactly 80°C. Apply vacuum to $\leq$ 50 mbar. Hold 24 h. Record start time: _____. Record end time: _____.	✓ ☐
1.2	Remove HCNFs. Mass after drying: _____ g. Expected mass loss $<3\%$. If $>3\%$, return to oven for additional 12 h.	✓ ☐
1.3	Verify CQD batch is available: _____ g (need 7.5 g). Confirm DLS size 3–7 nm. DLS result: _____ nm. If not within spec, re-synthesize.	✓ ☐
1.4	Weigh HDCNS Gen II: _____ g (target 15.0 g). Confirm BET $> 400 \text{ m}^2/\text{g}$. BET result: _____ m^2/g .	✓ ☐
1.5	Weigh HICF: _____ g (target 15.0 g). Confirm $D_{50} \approx 2 \mu\text{m}$. Laser diffraction D_{50} : _____ μm .	✓ ☐
1.6	Dry sebacic acid: 80°C / 12 h vacuum (50 mbar). Weigh after drying: _____ g (target 85.0 g). Confirm white powder; no visible clumps.	✓ ☐
1.7	Place all dried components in labelled, sealed vials at room temperature. Log: DATE _____ TIME _____ OPERATOR _____.	✓ ☐

STEP 2 — Resin Blending

⚠ Conduct ALL blending inside fume hood. N₂ blanket required. Ice bath must be prepared BEFORE starting sonication.

- Set up ice bath: 50% ice / 50% water in large plastic tub. Ensure sonicator probe tip is submerged in resin, not ice.
- Prepare: 3-neck round-bottom flask ($\geq 500 \text{ mL}$); vacuum port; N₂ inlet; thermocouple.
- Double-check PPE: neoprene gloves + goggles + face shield + P100/OV respirator BEFORE opening any chemicals.

#	Procedure — STEP 2	Done
2.1	Tare 3-neck flask. Add EHO: _____ g (target 125.0 g). Record mass: _____ g.	✓ ☐
2.2	Add HF-BX: _____ g (target 100.0 g). Record mass: _____ g. Stir gently with glass rod.	✓ ☐
2.3	Add Sebacic Acid: _____ g (target 85.0 g). Record mass: _____ g. Mixture should be translucent/amber.	✓ ☐
2.4	Apply N ₂ blanket (0.1 L/min) via inlet. Maintain throughout blending.	✓ ☐
2.5	Add CQDs: _____ g. Then HCNFs: _____ g. Then HDCNS Gen II: _____ g. Then HICF: _____ g. Add sequentially, not all at once.	✓ ☐
2.6	Place flask in ice bath. Insert sonicator probe (NOT touching flask wall). Set: 400 W, 20 kHz, PULSED (10 s on / 5 s off). Sonicate 30 min total ON time.	✓ ☐
2.7	Monitor mixture temperature with thermocouple during sonication. MUST remain $< 35^\circ\text{C}$. If temperature exceeds 35°C , pause until $<25^\circ\text{C}$ before resuming.	✓ ☐
2.8	Temperature log during sonication: 10 min: _____ $^\circ\text{C}$ 20 min: _____ $^\circ\text{C}$ 30 min: _____ $^\circ\text{C}$	✓ ☐
2.9	Connect vacuum (50 mbar). Degas for 15 min. Bubble count should approach zero by end.	✓ ☐
2.10	Measure viscosity with Brookfield DV-II+ at 25°C . Spindle: _____ Speed: _____ rpm. Result: _____ mPa·s. ACCEPTANCE CRITERIA: $< 600 \text{ mPa}\cdot\text{s}$. If $> 600 \text{ mPa}\cdot\text{s}$: investigate (check temperature, incomplete mixing). Do NOT proceed until $<600 \text{ mPa}\cdot\text{s}$.	✓ ☐
2.11	Record TOTAL BATCH MASS: _____ g. Expected: $\sim 500 \text{ g} \pm 5 \text{ g}$. Batch ID: _____.	✓ ☐

STEP 3 — Preform Lay-Up

#	Procedure — STEP 3	Done
3.1	Apply mold release: 3 coats Frekote 700-NC (or equiv.) to mold surfaces. Allow 10 min cure between coats. Final coat: 30 min before infusion. Record: coats applied: _____	✓ ☐
3.2	Cut PAN-CF UD plies to 300×300 mm: total plies needed: _____ (calc: target Vf 45%, ply thickness ≈ 0.25 mm → ~18 plies for 4.5 mm laminate). Record actual ply count: _____.	✓ ☐
3.3	Cut hemp bast woven plies to 300×300 mm. Areal weight: _____ g/m ² . Plies cut: _____.	✓ ☐
3.4	Stack sequence: [0°/90°/±45°]s — alternating PAN-CF (UD) and hemp bast woven (2×2 twill). Document exact stacking order below:	
3.4 a	Layer 1 (bottom): _____ Layer 2: _____ Layer 3: _____ Layer 4: _____ Layer 5: _____ Layer 6: _____ (mirror for symmetric layup)	✓ ☐
3.5	For RTM: close mold; connect resin injection port. Preheat mold to 40°C. Verify temperature: _____ °C. Inject resin at 0.4 MPa. Fill time: _____ min. Vent resin flow confirmed: Y/N _____	✓ ☐
3.6	For hand lay-up: wet out each ply individually; consolidate with roller; apply 0.6 MPa bladder pressure. Confirm 0.6 MPa applied: Y/N _____	✓ ☐
3.7	Target fiber volume fraction Vf = 45 ± 2 vol%. Estimate from panel mass: Vf = [(panel mass - matrix mass) / (PAN-CF density × panel volume)]. Calculated Vf: _____ vol%.	✓ ☐



SOP-H44-05 — Curing Protocol & Quality Control

5.1 Cure Schedule — Four-Stage Temperature Program

Stage	Temperature	Ramp / Hold	Duration	QC Checkpoint
Stage 0 — Ramp to gelation	25°C → 80°C	2°C/min	~28 min	Record start: _____
Stage 1 — Gelation	80°C	Hold 2 h	120 min	DEA monitoring ACTIVE; record gel-point time: _____ min
Stage 2 — Ramp to advance cure	80°C → 120°C	2°C/min	~20 min	—
Stage 3 — Advance Cure	120°C	Hold 2 h	120 min	DSC conversion target >95%; benzoxazine initiates
Stage 4 — Ramp to post-cure	120°C → 180°C	2°C/min	~30 min	—
Stage 5 — Post-Cure	180°C	Hold 1 h	60 min	Complete benzoxazine ring-opening; target Tg ≥ 175°C
Stage 6 — Controlled cool-down	180°C → 60°C	~2°C/min	~60 min	Slow cool to minimize residual stress in PAN-CF
Stage 7 — Free cool	60°C → RT	Free (oven door ajar)	~30 min	Do NOT force-cool
TOTAL CYCLE TIME	—	—	~7.5 h (programmed) + prep	Record actual end time: _____

5.2 In-Process Temperature Log (record every 30 min)

Time (min)	Stage	Setpoint	Actual Temp	DEA Reading	Operator Initials
0	Gelation start	80°C	_____°C	_____	_____
30	Gelation	80°C	_____°C	_____	_____
60	Gelation	80°C	_____°C	_____	_____
90	Gelation	80°C	_____°C	_____	_____

120	Advance cure start	120°C	_____°C	_____	_____
150	Advance cure	120°C	_____°C	_____	_____
180	Advance cure	120°C	_____°C	_____	_____
210	Advance cure	120°C	_____°C	_____	_____
240	Post-cure start	180°C	_____°C	_____	_____
270	Post-cure	180°C	_____°C	_____	_____
300	Post-cure end	Cooling	_____°C	_____	_____
360	Cooldown	<100°C	_____°C	_____	_____
420	Cooldown	<60°C	_____°C	_____	_____

5.3 Post-Cure Quality Checks (before demold)

Check	Acceptance Criterion	Result	Sign ☐
Panel reached RT (≤25°C) before demold	Verify with thermocouple	Actual temp at demold: _____°C	✓ ☐
Panel edges / flash inspection	No dry spots; uniform color; no delamination visible	Notes: _____	✓ ☐
Panel thickness (4 corners + center)	Target: _____ mm ± 0.2 mm	C1:____ C2:____ C3:____ C4:____ Centre:____	✓ ☐
Void content (acid digestion or burn-off, optional pre-screening)	Target Void < 2 vol%	Result: _____%	☐ opt.
Tap test (acoustic)	No hollow sound across panel area	Pass / Fail: _____	✓ ☐
Dimensions (width × length)	Target: 300 × 300 mm ± 2 mm	Actual: _____ × _____ mm	✓ ☐
Batch label applied	Panel ID / Date / Operator / Cure cycle version	Panel ID: _____	✓ ☐

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SOP-H44-06 — Characterization SOPs & Data Recording Sheets

6.1 Mechanical Testing — Specimen Preparation & Setup

Test	Cutting Method	Specimen Dimensions	n	Setup Notes
Tensile (ASTM D3039)	Diamond blade saw; water-cooled	250 × 25 × 2 mm (±0.1 mm)	n = 5	0° / 90° clearly marked; tab bond verified
Flexural 3-pt bend (ASTM D7264)	Diamond blade saw	Span/depth = 32:1; depth = t _{panel}	n = 5	Span set before testing: _____ mm
Compression CLC (ASTM D6641)	Diamond blade saw; fixture aligned	Standard CLC coupon	n = 5	Combined loading fixture torque: _____ N·m
ILSS short-beam (ASTM D2344)	Diamond blade saw	Span/depth = 5:1	n = 5	Verify uniform thickness: ±0.05 mm
Mode-I Glc DCB (ASTM D5528)	Diamond blade saw; pre-crack insert film	Pre-crack 50 mm from insert tip	n = 6	PTFE insert 13 µm; pre-crack propagation confirmed
Charpy notched impact (ISO 179-1)	Notch cutter; notch depth 1/3 specimen width	80 × 10 × 4 mm; 45° V-notch	n = 10	Pendulum energy capacity appropriate for expected impact energy

6.2 Tensile Test Data Sheet (ASTM D3039)

Panel ID: _____ Test Date: _____ Operator: _____ Crosshead speed: 1 mm/min
Environment: 23 ± 2°C / 50 ± 5% RH

ID	Width (mm)	Thickness (mm)	Area (mm ²)	Failure Load (kN)	UTS (MPa)	Modulus (GPa)	ε _{fail} (%)	Failure Mode
T-01	—	—	—	—	—	—	—	—
T-02	—	—	—	—	—	—	—	—
T-03	—	—	—	—	—	—	—	—
T-04	—	—	—	—	—	—	—	—
T-05	—	—	—	—	—	—	—	—
MEAN	—	—	—	—	—	—	—	—
SD	—	—	—	—	—	—	—	—
CV (%)	—	—	—	—	—	—	—	—
TARGET	—	—	—	—	≥ 1500	≥ 120	—	—

6.3 Mode-I Fracture Toughness Data Sheet (ASTM D5528 — DCB)

Panel ID: _____ Test Date: _____ Operator: _____ Pre-crack length a_0 : _____ mm

ID	Width b (mm)	Thickness 2h (mm)	Load P _c (N)	δ at P _c (mm)	a _c (mm)	G _{Ic} (J/m ²)
DCB-01	—	—	—	—	—	—
DCB-02	—	—	—	—	—	—
DCB-03	—	—	—	—	—	—
DCB-04	—	—	—	—	—	—
DCB-05	—	—	—	—	—	—
DCB-06	—	—	—	—	—	—
MEAN	—	—	—	—	—	—
TARGET	—	—	—	—	—	> 1400

6.4 Thermal Analysis Data Sheet

Test	Instrument	Conditions	Sample	Results
DSC — Cure Kinetics	TA Q2000	5°C/min; N ₂ ; -40→300°C	Sample mass: _____ mg	T _g onset: _____°C T _g midpoint: _____°C ΔH _{cure} : _____ J/g Conversion: _____%
DSC — Post-cure T _g	TA Q2000	5°C/min; 2nd heat scan	Sample mass: _____ mg	T _g (2nd heat): _____°C TARGET: ≥ 175°C Pass ☐ Fail ☐
DMA — Storage Modulus	TA Q800	3-pt bend; 1 Hz; 2°C/min	Specimen: _____ × _____ × _____ mm	E' at 25°C: _____ GPa T _g (E'' peak): _____°C Tan δ peak: _____°C
TGA — Thermal Stability	TA Q500	10°C/min; N ₂ then air	Sample mass: _____ mg	T _{onset} (N ₂): _____°C T _{50%} (N ₂): _____°C Char yield at 800°C: _____%
TGA — Bio-based content (LOI)	TA Q500 (air)	Burn-off at 600°C in air	Use neat matrix disk	Inorganic residue: _____% Bio-content estimate: _____% TARGET: ≥ 85%

	burn)			
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6.5 Spectroscopic Characterization Data Sheet

Test	Instrument	Conditions	Data to Record
FTIR-ATR — Cure conversion	Bruker Vertex 70	4000–400 cm ⁻¹ ; 64 scans; 4 cm ⁻¹ res.	Oxazine ring peak at 943 cm ⁻¹ before cure: _____ (A.U.) After cure: _____ (A.U.) Conversion = (1 – A _{after} /A _{before}) × 100% = _____% Epoxide peak at 910 cm ⁻¹ before: _____ After: _____ Conv: _____%
Raman — CQD D/G ratio	Renishaw inVia	532 nm laser; 10% power; 10 accumulations	D band (1350 cm ⁻¹): _____ G band (1580 cm ⁻¹): _____ D/G ratio: _____ (Target D/G < 1.0 for good graphitization of HDCNS)
XRD — HCNF crystallinity	PANalytical Empyrean	Cu Kα; 2θ = 5–40°; step 0.02°	I ₀₀₂ peak: _____ lam: _____ CI = (I ₀₀₂ – lam)/I ₀₀₂ = _____ (Target CI > 0.75 for HCNFs)
SEM/EDX — Fracture surface	FEI Quanta 250	Au sputter-coat; 5 kV; secondary electron	Attach representative fracture SEM image to data package. Fiber pull-out length: _____ μm Void estimate: _____% Fiber/matrix adhesion: Good / Poor

6.6 Final Acceptance Summary

Property	Design Target	Measured Value	Disposition
Tensile Strength (MPa)	≥ 1500	_____ MPa	Pass ☐ Fail ☐
Tensile Modulus (GPa)	≥ 120	_____ GPa	Pass ☐ Fail ☐
Flexural Strength (MPa)	≥ 1100	_____ MPa	Pass ☐ Fail ☐
Mode-I G_{IC} (J/m²)	> 1400	_____ J/m ²	Pass ☐ Fail ☐
T_g — DSC 2nd heat (°C)	≥ 175	_____ °C	Pass ☐ Fail ☐
Bio-Based Content (wt%)	≥ 85	_____ %	Pass ☐ Fail ☐
Void Content (vol%)	< 2	_____ %	Pass ☐ Fail ☐
OVERALL BATCH DISPOSITION	ALL above = Pass	PASS ☐ FAIL ☐	PI Signature: _____

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All design targets are pre-experimental projections (OSML-2026). No experimental data exist at time of issue.