

Standard Operating Procedure

Research Framework

Hemp-Derived Graphene Oxide (HGO) & Hemp-Derived Reduced Graphene Oxide (HrGO)

Landry Hemp Carbon Materials Platform

Hempoxies Division

Landry Industries

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This document establishes a standardized research workflow for investigating hemp-derived

graphene oxide (HGO) and hemp-derived reduced graphene oxide (HrGO) from sustainable hemp biomass. All experimental procedures must be performed under approved institutional laboratory safety protocols with appropriate engineering controls and PPE.

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1 Purpose

This Standard Operating Procedure (SOP) establishes a comprehensive, standardized research framework for the investigation, synthesis, characterization, and application of **hemp-derived graphene oxide (HGO)** and **hemp-derived reduced graphene oxide (HrGO)** produced from sustainable hemp (*Cannabis sativa L.*) biomass.

The primary objectives of this SOP are to:

1. Ensure **reproducibility** across all batches and experimental campaigns.
2. Maintain rigorous **documentation** and chain-of-custody for all materials.
3. Enforce **quality assurance** protocols at every stage of the workflow.
4. Guarantee **safe laboratory practice** during all synthesis, handling, and characterization activities.
5. Provide a **publication-ready** methodological foundation for peer-reviewed dissemination.

NOTE: This SOP is designed as a research planning and documentation framework. Specific experimental parameters (temperatures, reagent ratios, durations) must be selected from peer-reviewed literature or institutionally approved protocols and validated prior to implementation. This document does not constitute a prescriptive chemical synthesis manual.

2 Scope

This SOP is applicable to all research activities involving the following hemp-derived carbon precursors and intermediates:

- **Hemp bast fiber** – primary structural cellulose-lignin composite; highest graphitization potential
- **Hemp hurd** (shiv/core) – secondary lignocellulosic feedstock; lower crystallinity
- **Hemp biochar** – pyrolyzed carbon residue; amorphous to partially graphitic
- **Hemp carbon nanosheets** – exfoliated graphitic carbon from bast fiber
- **Hemp-derived graphitic carbon** – thermally ordered carbon with turbostratic structure

2.1 Exclusions

This SOP does **not** cover:

- Pharmaceutical or nutraceutical extraction from hemp
- Cannabis (high-THC) biomass processing
- Large-scale industrial manufacturing (>1 kg/batch)
- Any procedure not explicitly approved by institutional safety committees

3 Definitions and Abbreviations

Term	Definition
HGO	Hemp-Derived Graphene Oxide – oxidized graphitic carbon from hemp with oxygen-containing functional groups
HrGO	Hemp-Derived Reduced Graphene Oxide – partially de-oxygenated HGO with restored sp^2 domains
GO	Graphene Oxide (general, non-hemp-specific)
rGO	Reduced Graphene Oxide (general, non-hemp-specific)
Bast Fiber	The long, strong fibers from the phloem of the hemp stalk; primary source for graphitic carbon
Hurd	The woody inner core (shiv) of the hemp stalk; secondary carbon source
Biochar	Carbonaceous solid produced by pyrolysis of biomass under limited oxygen
Carbonization	Thermal decomposition of organic material in an inert atmosphere to produce carbon
Graphitization	Thermal ordering of amorphous carbon into layered graphitic structures
Exfoliation	Separation of stacked graphitic layers into individual or few-layer sheets
Oxidation	Introduction of oxygen functional groups (epoxy, hydroxyl, carboxyl) into graphitic carbon
Reduction	Removal of oxygen functional groups to restore electrical conductivity
TGA	Thermogravimetric Analysis
DSC	Differential Scanning Calorimetry
XRD	X-Ray Diffraction
XPS	X-Ray Photoelectron Spectroscopy
FTIR	Fourier-Transform Infrared Spectroscopy
Raman	Raman Spectroscopy
SEM	Scanning Electron Microscopy
TEM	Transmission Electron Microscopy
AFM	Atomic Force Microscopy
BET	Brunauer-Emmett-Teller surface area analysis
I_D/I_G	Ratio of D-band to G-band intensities in Raman spectroscopy; indicator of disorder
I_{2D}/I_G	Ratio of 2D-band to G-band intensities; indicator of layer number
SOP	Standard Operating Procedure
PPE	Personal Protective Equipment

4 Research Objectives

- RO-1: Precursor Standardization** – Produce reproducible, well-characterized carbon precursors from hemp biomass with consistent carbon yield, ash content, and structural properties.
- RO-2: Oxidation Route Development** – Investigate and optimize oxidation routes from hemp-derived graphitic carbon to HGO, evaluating degree of oxidation, layer number, and defect density.
- RO-3: Reduction Route Development** – Investigate and optimize reduction routes from HGO to HrGO, evaluating restoration of sp^2 domains, electrical conductivity, and dispersibility.
- RO-4: Material Characterization** – Conduct comprehensive structural, chemical, morphological, and functional characterization of HGO and HrGO at every stage.
- RO-5: Composite Performance Evaluation** – Evaluate HGO and HrGO as functional additives and reinforcements in bio-based polymer matrices, particularly epoxidized hempseed oil (EHO) systems and vitrimer chemistries.
- RO-6: Scalability Assessment** – Assess process parameters for potential scale-up from laboratory (~ 1 g) to pilot (~ 1 kg) production.

5 Theoretical Background

5.1 Hemp as a Carbon Precursor

Hemp (*Cannabis sativa L.*) is a lignocellulosic biomass with a unique multilayered structure. The **bast fiber** consists of crystalline cellulose microfibrils embedded in a lignin-hemicellulose matrix, with natural alignment that facilitates graphitic ordering upon thermal treatment. Unlike conventional bulk carbons requiring temperatures $> 2000^\circ\text{C}$ for graphitization, selected biomass precursors with inherent molecular alignment can form turbostratic graphitic structures at temperatures near 1000°C .

5.2 From Hemp to Graphitic Carbon

The conversion pathway involves:

1. **Deconstruction** – Removal of hemicellulose and partial lignin degradation via hydrothermal or chemical pretreatment
2. **Carbonization** – Thermal decomposition in inert atmosphere ($\sim 300^\circ\text{C}$ – 1000°C) to produce amorphous carbon
3. **Activation/Graphitization** – High-temperature treatment to promote layer ordering and porosity development
4. **Exfoliation** – Mechanical, chemical, or thermal separation of stacked layers

5.3 Graphene Oxide Formation

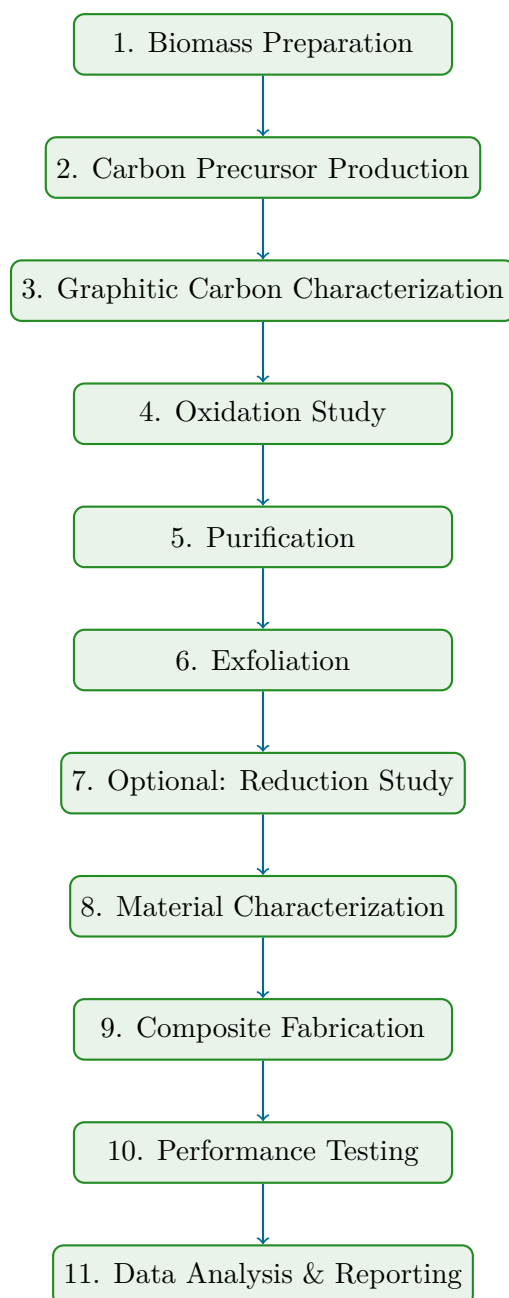
Graphitic carbon is oxidized using strong oxidants (typically KMnO_4 in concentrated H_2SO_4) to introduce oxygen functional groups (epoxy, hydroxyl, carboxyl) that increase interlayer spacing and hydrophilicity, enabling aqueous exfoliation into individual or few-layer sheets.

5.4 Reduction Mechanisms

Reduction of HGO to HrGO involves removal of oxygen functional groups to restore the conjugated sp^2 carbon network. Methods include thermal reduction, chemical reduction (e.g., hydrazine, ascorbic acid), electrochemical reduction, and green reduction pathways (e.g., plant extracts, microbial reduction).

6 Research Workflow

The following workflow represents the complete research pipeline from raw biomass to characterized composite materials. Each stage is described in detail in subsequent sections.



6.1 Stage 1: Biomass Preparation

6.1.1 Raw Material Selection

- Source certified industrial hemp ($\text{THC} \leq 0.3\%$ dry weight)
- Record cultivar, harvest date, geographic origin, and growth conditions
- Prefer bast fiber for highest graphitization potential

6.1.2 Pre-treatment Protocol

1. Wash raw hemp biomass with deionized water to remove soil and debris
2. Dry at 60°C in a convection oven until moisture content $< 5\%$

3. Record initial mass and moisture content
4. Separate bast fiber from hurd via mechanical decortication
5. Mill to uniform particle size (target: < 1 mm)
6. Store in airtight, desiccated containers at room temperature

SAFETY WARNING: Ensure adequate ventilation during drying. Hemp dust is combustible; avoid ignition sources. Use dust collection systems during milling.

6.2 Stage 2: Carbon Precursor Production

6.2.1 Hydrothermal Carbonization (Optional Pre-treatment)

- Treat hemp biomass in aqueous medium at 180°C – 200°C for 12–24 h
- Deconstructs lignocellulosic matrix; facilitates subsequent graphitization
- Wash and dry product before thermal carbonization

6.2.2 Thermal Carbonization

1. Load dried hemp biomass into alumina or quartz boat
2. Place in tube furnace under continuous inert gas flow (N_2 or Ar, $\geq 99.998\%$ purity)
3. Ramp at $5^{\circ}\text{C}/\text{min}$ to 300°C ; hold for 1 h–3 h (tar decomposition)
4. Continue ramp to target carbonization temperature (800°C – 1000°C)
5. Hold at target temperature for 2 h–4 h
6. Cool to room temperature under inert atmosphere
7. Record carbon yield (typically 15–25% of dry biomass mass)

6.2.3 Chemical Activation (Optional)

For enhanced porosity and surface area:

- Mix carbonized material with activating agent (KOH , ZnCl_2 , or H_3PO_4) at mass ratio 1:3 to 1:5
- Heat in inert atmosphere to 700°C – 900°C for 1 h–3 h
- Wash thoroughly with dilute HCl and deionized water until neutral pH
- Dry at 100°C – 120°C

SAFETY WARNING: Chemical activation uses corrosive reagents. Perform in fume hood with acid-resistant PPE. Neutralize waste streams before disposal. KOH is highly caustic; ZnCl_2 is toxic.

6.3 Stage 3: Graphitic Carbon Characterization

Before proceeding to oxidation, characterize the carbon precursor:

Technique	Parameter	Target/Acceptance
TGA/DSC	Thermal stability, carbon content	Residual mass > 70% at 800 °C
XRD	Graphitic ordering	(002) peak at $2\theta \approx 22-26^\circ$; FWHM indicates crystallite size
Raman	I_D/I_G ratio	< 1.5 for well-ordered carbon; > 1.5 indicates high disorder
BET	Specific surface area	> 500 m ² /g for activated carbons
SEM/TEM	Morphology, layer structure	Sheet-like morphology preferred
Elemental Analysis	C, H, N, O, ash	Carbon > 80%; ash < 5%

6.4 Stage 4: Oxidation Study

6.4.1 Modified Hummers Method (Reference Protocol)

SAFETY WARNING: This section describes a **reference framework** based on published methods. All oxidation procedures involving concentrated acids and strong oxidants are **hazardous** and must be performed only under approved safety protocols with appropriate engineering controls. The specific reagent ratios, temperatures, and durations must be validated and approved by institutional safety committees before implementation.

General Procedure Framework:

1. Dispersion: Suspend hemp-derived graphitic carbon in concentrated H₂SO₄ (~98%) under ice bath cooling (0 °C–5 °C)
2. Oxidant Addition: Slowly add KMnO₄ (strong oxidant) with continuous stirring, maintaining temperature < 20 °C to prevent runaway exotherm
3. Oxidation: Remove ice bath; heat to 35 °C–50 °C and stir for 30 min–2 h
4. Dilution: Slowly add deionized water (exothermic); maintain temperature < 100 °C
5. Termination: Add H₂O₂ (3–30%) until solution turns bright yellow (indicates complete oxidation)
6. Washing: Centrifuge and wash repeatedly with dilute HCl (5%) and deionized water until neutral pH
7. Drying: Dry at 60 °C–80 °C to obtain graphite oxide

6.4.2 Modified Tour Method (Safer Alternative)

The Tour method replaces NaNO_3 with H_3PO_4 and operates at lower temperatures, eliminating toxic gas evolution:

- Mix $\text{H}_2\text{SO}_4\text{:H}_3\text{PO}_4$ (typically 9:1 v/v)
- Add graphitic carbon and KMnO_4 at room temperature
- Stir at $35\text{ }^\circ\text{C}$ – $50\text{ }^\circ\text{C}$ for 12–24 h
- Quench with ice, add H_2O_2 , wash, and dry as above

6.4.3 Green Oxidation Routes (Under Investigation)

- Electrochemical oxidation in neutral/electrolyte solutions
- Plasma-assisted oxidation at atmospheric pressure
- Enzymatic oxidation using laccase or peroxidase enzymes
- Microwave-assisted oxidation with reduced reagent loadings

6.5 Stage 5: Purification

1. Suspend graphite oxide in deionized water
2. Centrifuge at 3000–4000 rpm for 10–15 min to remove unexfoliated material
3. Decant supernatant; repeat washing until neutral pH and clear supernatant
4. Optional: Dialysis against deionized water for 48–72 h to remove ionic residues
5. Record yield and store as aqueous dispersion or dry powder

6.6 Stage 6: Exfoliation

6.6.1 Sonication-Assisted Exfoliation

- Disperse purified graphite oxide in deionized water (typically 1–5 mg/mL)
- Sonicate using bath or probe sonicator
- Bath sonication: 1–3 h at moderate power
- Probe sonication: 30 min–1 h at low power (avoid over-oxidation and fragmentation)
- Monitor by UV-Vis spectroscopy (characteristic peak at $\sim 230\text{ nm}$)

6.6.2 Thermal Exfoliation

- Rapid heating of dry graphite oxide to $300\text{ }^\circ\text{C}$ – $1050\text{ }^\circ\text{C}$ in inert atmosphere or vacuum
- Thermal shock causes gas evolution (CO , CO_2 , H_2O) forcing layer separation
- Produces thermally reduced graphene oxide (TrGO) simultaneously

6.6.3 Mechanical Exfoliation

- High-shear mixing, ball milling, or three-roll milling
- Combine with surfactants or stabilizers for aqueous dispersion

6.7 Stage 7: Optional Reduction Study

6.7.1 Thermal Reduction

- Heat HGO to 300 °C–1100 °C under inert atmosphere or vacuum
- Higher temperatures restore more sp^2 character but may cause layer restacking
- Monitor by Raman (I_D/I_G), XPS (C/O ratio), and conductivity measurements

6.7.2 Chemical Reduction

- Hydrazine hydrate: Highly effective but toxic; use in fume hood with full PPE
- Sodium borohydride (NaBH_4): Moderate reducing agent; aqueous compatible
- Ascorbic acid (Vitamin C): Green reducing agent; biocompatible
- Hydroiodic acid (HI): Strong reduction; produces high-quality rGO

6.7.3 Green Reduction Methods

- Plant extract reduction (e.g., green tea polyphenols, citrus extracts)
- Microbial reduction using bacteria or fungi
- Electrochemical reduction at controlled potential
- Photochemical reduction using UV or solar irradiation

6.8 Stage 8: Material Characterization

Comprehensive characterization must be performed on HGO and HrGO at each developmental stage.

6.8.1 Structural Characterization

Technique	Information Obtained	Key Parameters
XRD	Layer spacing, crystallinity	(001) peak for GO at $2\theta \approx 10^\circ$ – 12° ; (002) for rGO at $2\theta \approx 24^\circ$ – 26°
Raman	Defect density, layer number	I_D/I_G : disorder; I_{2D}/I_G : layer count; L_a from Tuinstra-Koenig relation

Technique	Information Obtained	Key Parameters
TEM/SAED	Morphology, crystallinity	Layer thickness, SAED rings confirm graphitic structure
AFM	Exact layer thickness	Monolayer ~ 1 nm; few-layer ~ 2 nm–5 nm

6.8.2 Chemical Characterization

Technique	Information Obtained	Key Parameters
XPS	Elemental composition, oxidation states	C/O ratio: GO ~ 2 –4; rGO > 8 ; C 1s deconvolution
FTIR	Functional groups	Epoxy (~ 1220 cm^{-1}), hydroxyl (~ 3400 cm^{-1}), carboxyl (~ 1720 cm^{-1})
TGA	Thermal stability, functional group content	Mass loss steps indicate decomposition of oxygen groups
Elemental Analysis	Bulk C, H, N, O, S	Carbon purity; sulfur residue from sulfonation

6.8.3 Physical Properties

Technique	Information Obtained	Key Parameters
BET	Surface area, pore structure	GO: 400–800 m^2/g ; rGO: 200–600 m^2/g
Four-Point Probe	Electrical conductivity	rGO: 10^0 – 10^3 S/m; GO: insulating
Zeta Potential	Colloidal stability	$ \zeta > 30$ mV indicates stable dispersion
UV-Vis	Dispersion quality, concentration	GO peak at ~ 230 nm; rGO shoulder at ~ 270 nm

6.9 Stage 9: Composite Fabrication

Evaluate HGO and HrGO as functional additives in the following matrix systems:

- **Epoxidized hempseed oil (EHO)** thermosets – bio-based epoxy systems
- **Bioepoxies** – plant oil-derived epoxy resins
- **Thermosets** – conventional epoxy, phenolic, and polyester resins
- **Vitrimers** – dynamic covalent networks with exchangeable bonds

- **Bioplastics** – PLA, PHA, starch-based, and cellulose-derived polymers

General Composite Preparation Protocol:

1. Disperse HGO/HrGO in solvent or monomer using sonication (30–60 min)
2. Add curing agent/catalyst and mix thoroughly
3. Degas under vacuum to remove trapped air
4. Cast into molds and cure according to resin-specific protocol
5. Post-cure if required
6. Machine test specimens per ASTM/ISO standards

6.10 Stage 10: Performance Testing

6.10.1 Mechanical Testing

- Tensile strength and modulus (ASTM D638, ISO 527)
- Flexural strength and modulus (ASTM D790, ISO 178)
- Fracture toughness (K_{IC} , G_{IC}) (ASTM D5045, ISO 13586)
- Impact strength (Izod/Charpy) (ASTM D256, ISO 180)

6.10.2 Thermal Analysis

- Dynamic Mechanical Analysis (DMA) – storage modulus, $\tan \delta$, T_g
- TGA – thermal stability, decomposition temperature
- DSC – glass transition, curing behavior
- Thermal conductivity (laser flash or guarded hot plate)

6.10.3 Electrical and Functional Testing

- Electrical conductivity (four-point probe, van der Pauw method)
- Dielectric properties (impedance spectroscopy)
- Electromagnetic interference (EMI) shielding effectiveness
- Water absorption (ASTM D570, ISO 62)
- Chemical resistance (immersion tests in acids, bases, solvents)

6.11 Stage 11: Data Analysis and Reporting

1. Compile all characterization data into structured database
2. Perform statistical analysis (mean, standard deviation, ANOVA where applicable)
3. Correlate processing parameters with material properties
4. Generate publication-quality figures and tables
5. Draft manuscript following target journal guidelines
6. Archive raw data, protocols, and analysis scripts per institutional data management policy

7 Documentation Requirements

Every batch must be documented with the following information:

Field	Description/Format
Batch ID	Unique identifier (e.g., HGO-YYYY-MM-DD-XXX)
Biomass Source	Cultivar, supplier, geographic origin
Harvest Date	Date of hemp harvest
Moisture Content	% moisture at time of processing (wet basis)
Pre-treatment	Washing, drying, milling parameters
Carbonization Temperature	Target and actual temperature (°C)
Carbonization Duration	Hold time at target temperature (h)
Atmosphere	Gas type, flow rate (L/min)
Carbon Yield	Mass of carbon / mass of dry biomass (%)
Activation Agent	Type, mass ratio, if applicable
Oxidation Method	Hummers, Tour, or alternative
Oxidation Temperature	Reaction temperature (°C)
Oxidation Duration	Total reaction time (h)
Exfoliation Method	Sonication, thermal, mechanical
Reduction Method	Thermal, chemical, green (specify)
Sample Mass	Final product mass (g)
Operator	Name and signature
Laboratory Notebook	Page/reference number
Date/Time	Start and end timestamps
Deviations	Any deviations from protocol with justification

8 Quality Control

Each batch must pass the following quality control checks:

QC Parameter	Method	Acceptance Criteria
Carbon Purity	Elemental Analysis	C > 80%; ash < 5%
Oxygen Functionality	XPS C/O Ratio	GO: 2–5; HrGO: > 8
Layer Morphology	TEM/AFM	Predominantly few-layer (< 5 layers)
Particle Size	DLS or SEM image analysis	Lateral dimension > 500 nm preferred
Surface Area	BET	> 200 m ² /g
Reproducibility	Inter-batch comparison	Key parameters within ±10%
Dispersion Quality	UV-Vis, visual inspection	Stable colloid for > 24 h without sedimentation

9 Safety Protocols

SAFETY WARNING: ALL synthesis, oxidation, reduction, purification, and waste handling shall be performed only under approved institutional laboratory safety procedures, with appropriate engineering controls, personal protective equipment (PPE), hazard assessments, and regulatory compliance. This SOP does not replace institutional safety training or hazard-specific SOPs.

9.1 Hazard Categories

Hazard	Source	Mitigation
Corrosive chemicals	Concentrated H ₂ SO ₄ , H ₃ PO ₄ , KOH, HCl	Acid-resistant gloves, face shield, fume hood, neutralization kit
Strong oxidants	KMnO ₄	Avoid contact with organics; store separately; use in fume hood
Toxic gases	NO _x , Cl ₂ (from NaNO ₃ + H ₂ SO ₄), SO ₂	Use modified Tour method to eliminate NaNO ₃ ; gas detection
Flammable materials	Organic solvents, hydrogen gas (from NaBH ₄)	Ground equipment; no open flames; explosion-proof equipment
Toxic reducers	Hydrazine hydrate, HI	Full-face respirator with organic vapor cartridge; dedicated fume hood
Nanomaterial exposure	Inhalation/dermal contact with HGO/HrGO powders	N95/P100 respirator; nitrile gloves; work in ventilated enclosure

Hazard	Source	Mitigation
Thermal hazards	High-temperature furnaces, exothermic reactions	Heat-resistant gloves; temperature monitoring; emergency cooling

9.2 Required PPE

- Lab coat (chemical-resistant for oxidation/reduction steps)
- Safety goggles or face shield
- Nitrile gloves (double-gloving recommended for concentrated acids)
- Closed-toe shoes
- N95 or P100 respirator when handling dry nanomaterial powders
- Acid-resistant apron for oxidation procedures

9.3 Emergency Procedures

1. **Chemical spill:** Evacuate area; alert others; use spill kit appropriate for acid/base/oxidant; do not attempt cleanup without proper PPE
2. **Fire:** Activate fire alarm; use Class D extinguisher for metal fires; evacuate if fire cannot be controlled
3. **Exposure:** Flush affected area with water for 15+ minutes; seek medical attention; report to safety officer
4. **Gas leak:** Evacuate immediately; do not operate electrical switches; alert emergency services

9.4 Waste Management

- Collect acid waste in designated acid waste container; do not mix with organic waste
- Neutralize dilute acid waste before drain disposal (check local regulations)
- Heavy metal waste (Mn, Zn) must be collected as hazardous waste
- Nanomaterial waste must be solidified or collected in sealed containers; do not dispose in regular trash
- Maintain waste log with chemical composition, quantity, and disposal date

10 Future Research Directions

The following areas represent high-priority research avenues for the Landry Hemp Carbon Materials Platform:

- FR-1: Green Oxidation Chemistry** – Develop oxidation protocols using benign oxidants (e.g., peracids, electrochemical oxidation) that eliminate concentrated acids and toxic byproducts.
- FR-2: Electrochemical Exfoliation** – Investigate electrochemical intercalation and exfoliation of hemp-derived graphitic carbon as a scalable, solvent-free alternative to chemical oxidation.
- FR-3: Plasma-Assisted Functionalization** – Explore atmospheric-pressure plasma treatment for controlled surface functionalization without wet chemistry.
- FR-4: Microwave-Assisted Processing** – Leverage microwave heating for rapid, energy-efficient carbonization, activation, and reduction.
- FR-5: Enzymatic Functionalization** – Use cellulases, laccases, and peroxidases for selective surface modification of HGO/HrGO.
- FR-6: Surface Modification for Matrix Compatibility** – Develop covalent and non-covalent functionalization strategies to improve dispersion and interfacial adhesion in bioepoxy and vitrimer matrices.
- FR-7: Composite Optimization** – Systematically study loading levels, dispersion methods, and curing protocols to maximize mechanical and electrical performance in hemp-based composites.
- FR-8: AI-Assisted Process Optimization** – Apply machine learning to correlate processing parameters with material properties and predict optimal synthesis conditions.
- FR-9: Scale-Up Studies** – Transition from laboratory-scale (~ 1 g) to pilot-scale (~ 1 kg) production while maintaining material quality and reproducibility.
- FR-10: Life-Cycle Assessment (LCA)** – Conduct cradle-to-gate LCA to quantify the environmental benefits of hemp-derived carbon materials versus petroleum-derived alternatives.
- FR-11: Closed-Loop Recycling** – Investigate the recovery and reuse of HGO/HrGO from end-of-life composites to enable circular material flows.

11 References

The following categories of literature should be consulted and cited in all research outputs:

- [1] Hemp Carbon Nanomaterials

- Mitlin, D., et al. “Interconnected Carbon Nanosheets Derived from Hemp for Ultrafast Supercapacitors with High Energy.” *ACS Nano*, 2013, 7(6), 5131–5141. DOI: 10.1021/nm400731g
- Hemp-derived graphene nanoplatelets from bast and inner bark fibers; carbonization at 800–1000°C under inert atmosphere; nanosheet thickness 10–30 nm; surface area > 2200 m²/g; electrical conductivity 211–226 S/m.

[2] Graphene Oxide Synthesis

- Hummers, W.S. & Offeman, R.E. “Preparation of Graphitic Oxide.” *J. Am. Chem. Soc.*, 1958, 80(6), 1339.
- Marcano, D.C., et al. “Improved Synthesis of Graphene Oxide.” *ACS Nano*, 2010, 4(8), 4806–4814. (Tour method)
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[3] Reduced Graphene Oxide

- Pei, S. & Cheng, H.-M. “The Reduction of Graphene Oxide.” *Carbon*, 2012, 50(9), 3210–3228.
- Fernandez-Merino, M.J., et al. “Investigating the Influence of Oxygen Functional Groups on the Thermal and Electrical Properties of Graphene Oxide.” *J. Phys. Chem. C*, 2012, 116(35), 18634–18640.

[4] Sustainable Carbon Materials

- Seitzhanova, M., et al. “Synthesis of Biomass-Derived Graphene Nanomaterials by Chemical Activation with KOH.” *Int. J. Mol. Sci.*, 2025, 26(23), 11255.
- Silviana, D.R., et al. “Structural Investigation of Biomass-Derived Graphene Oxide Prepared by a Single Step Pyrolysis Followed by a Modified Tour Method.” *Engineering Proceedings*, 2025, 11, 53.

[5] Bio-Based Composites

- Epoxidized hempseed oil (EHO) thermoset systems and vitrimer chemistries
- Hemp fiber-reinforced biocomposites and nanocomposites
- Dynamic covalent polymer networks with carbon nanomaterial fillers

12 Appendices

12.1 Appendix A: Batch Record Template

BATCH RECORD – HGO/HrGO Synthesis			
Batch ID:			
Date:			
Operator:			
Supervisor:			
Raw Material			
Source:		Moisture: %	Mass: g
Carbonization			
Temp: °C	Time: h	Atmosphere:	Yield: %
Oxidation			
Method:	Temp: °C	Time: h	Yield: %
Exfoliation			
Method:	Time:	Yield: %	
Reduction (if applicable)			
Method:	Temp: °C	Time: h	
QC Results			
C/O Ratio:	I_D/I_G :	Surface Area: m ² /g	
Deviations/Notes:			
Signatures:			
Operator:		Date:	
Supervisor:		Date:	

12.2 Appendix B: Equipment List

Equipment	Specification	Application
Tube furnace	Max 1200°C, programmable, inert gas	Carbonization, activation, thermal reduction
Muffle furnace	Max 1100°C, air atmosphere	Ash content, biochar preparation
Ultrasonic bath	40 kHz, heated	Exfoliation, dispersion
Probe sonicator	20 kHz, adjustable power	Exfoliation
Centrifuge	4000+ rpm, refrigerated	Purification, washing

Equipment	Specification	Application
Freeze dryer	−50°C, vacuum	Drying sensitive samples
Vacuum oven	200°C max, vacuum	Drying, degassing
Four-point probe	Keithley or equivalent	Electrical conductivity
BET analyzer	N ₂ adsorption, ASAP series	Surface area, porosity
Raman spectrometer	532 nm laser, confocal	Structural characterization
XRD diffractometer	Cu K α , 2 θ 5–80°	Crystallinity, layer spacing
XPS system	Al K α , high resolution	Elemental composition, bonding
TEM	200 kV, FEG	Morphology, layer count
SEM	5–30 kV, EDX	Surface morphology, elemental mapping
AFM	Tapping mode, ambient	Layer thickness, surface roughness
FTIR	ATR mode, 400–4000 cm ^{−1}	Functional group identification
TGA/DSC	N ₂ or air, RT–1000°C	Thermal stability, phase transitions
UV-Vis spectrophotometer	200–800 nm	Dispersion quality, concentration
Zeta potential analyzer	Laser Doppler electrophoresis	Colloidal stability
Mechanical testing frame	10–50 kN capacity	Tensile, flexural, fracture
DMA	3-point bending, tension	Viscoelastic properties

12.3 Appendix C: Glossary of Hemp Carbon Terminology

Term	Definition
Turbostratic carbon	Graphitic carbon with parallel but randomly rotated layers; intermediate between amorphous and crystalline graphite
Interlayer spacing	Distance between adjacent graphene layers; ~ 3.35 Å for graphite, ~ 6 – 10 Å for GO
sp^2 carbon	Trigonal planar carbon with delocalized π -electrons; responsible for electrical conductivity
sp^3 carbon	Tetrahedral carbon with single bonds; introduced by oxidation and defects
Defect density	Concentration of structural imperfections (vacancies, edges, Stone-Wales defects) per unit area
Functional group density	Number of oxygen-containing groups per carbon atom
D-band	Raman peak at ~ 1350 cm ^{−1} ; indicates defects and disorder

Term	Definition
G-band	Raman peak at $\sim 1580\text{ cm}^{-1}$; indicates sp^2 carbon network
2D-band	Raman peak at $\sim 2700\text{ cm}^{-1}$; indicates layer stacking and quality

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