

Experimental Design for the Synthesis and Characterization of Sp^3 -Bonded Diamond Nanothreads Derived from *Cannabis sativa* L. Furan Precursors

Abstract Global demand for high-strength, lightweight structural materials necessitates a transition away from petrochemical precursors. This paper proposes a testable hypothesis regarding the synthesis of Diamond Nanothreads (DNTs)—one-dimensional carbon allotropes with diamond-like sp^3 bonding—using furan derived exclusively from industrial hemp (*Cannabis sativa* L.) biomass. While theoretical models predict DNTs possess tensile strengths exceeding 100 GPa, empirical validation using bio-derived precursors remains absent from the literature. We outline a rigorous experimental protocol for the isolation of furan from hemp hurds, its polymerization under high pressure (10–15 GPa) in a Diamond Anvil Cell (DAC), and the subsequent characterization of the resulting material via *in-situ* Raman spectroscopy and Transmission Electron Microscopy (TEM). This study aims to validate the structural integrity and mechanical properties of bio-derived DNTs against petroleum-derived controls.

1. Introduction

1.1 Background and Motivation

The search for ultra-high-strength materials has largely focused on carbon nanotubes (CNTs) and graphene. However, Diamond Nanothreads (DNTs), first synthesized from benzene, offer a unique combination of extreme stiffness (theoretical Young's modulus >850 GPa) and chemically active surfaces suitable for functionalization. Current synthesis methods rely heavily on petrochemical precursors (benzene, pyridine).

Recent "Green Chemistry" frameworks suggest that furan ($\text{C}_4\text{H}_4\text{O}$), a heterocyclic organic compound, can undergo pressure-induced polymerization (PIP) to form similar sp^3 structures at lower pressures (10–13 GPa) compared to benzene (~20 GPa). Industrial hemp is a viable, scalable source of furan via the hemicellulose fraction of the hemp hurd.

1.2 Problem Statement & Hypothesis

Current literature describing "Hempoxies" or hemp-based vitrimers often relies on speculative assertions of "bulletproof" performance without empirical data. There is a critical gap in experimentally verifying whether furan sourced specifically from biological reduction of hemp pentosans can achieve the lattice purity and degree of polymerization required to match the mechanical properties of traditional DNTs.

Hypothesis: Furan synthesized from the acid hydrolysis and decarboxylation of hemp hurd hemicellulose will undergo solid-state polymerization at 12 GPa to form crystalline, sp^3 -bonded nanothreads with a Young's modulus statistically indistinguishable from DNTs synthesized from

petrochemical furan.

2. Materials and Methods

2.1 Precursor Synthesis: Hemp-Derived Furan

To ensure the validity of the bio-origin claim, the precursor must be isolated directly from biomass under controlled conditions.

1. **Biomass Preparation:** Industrial hemp hurds (shives) are milled to a particle size of <0.5 mm and dried to constant weight.
2. **Acid Hydrolysis (Pentose Extraction):**
 - Reagents: 10% H₂SO₄ (aq).
 - Procedure: Biomass is refluxed at 100°C for 4 hours. The solid residue (cellulose/lignin) is filtered out. The filtrate (rich in xylose) is retained.
3. **Dehydration to Furfural:**
 - The xylose-rich filtrate is heated to 250°C in a continuous flow reactor to facilitate dehydration to furfural (C₅H₄O₂).
 - *Purification:* Vacuum distillation to achieve >99% purity.
4. **Decarbonylation to Furan:**
 - Catalyst: Pd/C or Zn-Fe chromite.
 - Conditions: Vapor-phase reaction at 200°C.
 - *Validation:* The final furan product is analyzed via Gas Chromatography-Mass Spectrometry (GC-MS) to ensure it matches the purity profile of Sigma-Aldrich standard furan (>99.5%).

2.2 High-Pressure Synthesis (The Variable)

This step tests the core physics of the "bulletproof" claim.

- **Apparatus:** Symmetric Diamond Anvil Cell (DAC) equipped with Type IIa diamond anvils (300 μm culet size).
- **Sample Loading:**
 - A rhenium gasket is pre-indented to 50 μm thickness and drilled with a 150 μm hole.
 - **Group A (Experimental):** Hemp-derived Furan.
 - **Group B (Control):** Petrochemical Standard Furan (Sigma-Aldrich).
 - No pressure transmitting medium is used, as the sample acts as its own medium.
- **Compression Protocol:**
 1. Pressure is increased in 0.5 GPa increments.
 2. Target Pressure: **13 GPa**.
 3. Hold Time: 12 hours to ensure complete polymerization.
 4. Decompression: Slow release (0.2 GPa/min) to recover the sample to ambient pressure.

2.3 Characterization Plan

Evidence of sp³ bonding is the "go/no-go" metric for success.

1. **In-Situ Raman Spectroscopy:**

- Collected at every 2 GPa increment during compression and decompression.
 - *Target Signal*: Disappearance of the C=C stretching modes ($\sim 1500\text{-}1600\text{ cm}^{-1}$) and emergence of sp^3 C-C modes ($\sim 1000\text{-}1200\text{ cm}^{-1}$).
2. **Transmission Electron Microscopy (TEM)**:
 - Recovered samples are dispersed in ethanol, sonicated, and drop-cast onto TEM grids.
 - *Target Observation*: Diffraction contrast imaging to reveal 1D crystalline order (threads) vs. amorphous carbon.
 3. **Nanoindentation (Mechanical Proxy)**:
 - Since macroscopic tensile testing is impossible with DAC sample sizes, modulus is estimated via nanoindentation of the recovered pellet.
 - *Instrument*: Hysitron TI 980 TriboIndenter.

3. Statistical Analysis Plan

3.1 Power Analysis

To determine the necessary sample size for the Nanoindentation comparison (Young's Modulus), we assume an independent samples t-test (two-tailed).

- **Effect Size (Cohen's d)**: We define "equivalence" as a difference of less than 1.0 standard deviation between the hemp and control groups. However, to detect a meaningful defect-induced reduction in strength (e.g., a 20% drop), we assume a large effect size ($d = 1.2$).
- **Alpha (α)**: 0.05 (5% risk of Type I error).
- **Power ($1 - \beta$)**: 0.80 (80% probability of detecting an effect).

Calculation: Using G*Power 3.1, the required sample size is **N = 12 samples per group**.

3.2 Experimental Matrix

Total DAC runs required: 24 (12 Hemp-Derived, 12 Control). *Note: Given the low throughput of DAC, this represents a significant but necessary effort to establish statistical validity.*

3.3 Data Interpretation

- **Null Hypothesis (H_0)**: $\mu_{\text{hemp}} = \mu_{\text{control}}$ (There is no difference in modulus).
- **Alternative Hypothesis (H_1)**: $\mu_{\text{hemp}} \neq \mu_{\text{control}}$.
- If $p > 0.05$ and the mean modulus is within the theoretical range ($>800\text{ GPa}$), the material is validated as a high-performance bio-nanocomposite.

4. Discussion of Potential Failure Modes

This research acknowledges significant risks absent from previous monographs:

1. **Amorphization**: If the compression rate is too fast, the furan ring may collapse into amorphous carbon rather than ordered threads.
2. **Purity Sensitivity**: Trace impurities in the bio-derived furan (e.g., residual water or acids) could act as defect sites, drastically lowering the modulus. This would disprove the "bulletproof" capability of the raw hemp extract without further refinement.

3. **Metastability:** The recovered nanothreads may be unstable at ambient pressure, reverting to monomeric furan or degrading over time.

5. Funding and Ethics Statement

Funding: This research proposal is currently unfunded. Applications are being prepared for submission to the National Science Foundation (Materials Engineering and Processing) and the Department of Energy (Bioenergy Technologies Office).

Conflict of Interest: The authors declare no commercial interest in hemp processing facilities at this stage. This study is purely exploratory and aims to decouple scientific validation from commercial speculation.

Ethical Considerations: All biomass is sourced from certified industrial hemp producers in compliance with the 2018 Farm Bill (USA) or equivalent regional regulations (<0.3% THC). No human or animal subjects are involved in this materials science study.