Prototyping Protocol: Seshat's Bones v1.1

A High-Performance Hemp-Derived Biocomposite

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Abstract

This document outlines the standard operating procedure for the synthesis and initial characterization of **Seshat's Bones**, a novel, high-performance biocomposite. The material is formulated from a matrix of epoxidized hemp oil and reinforced with modified hemp-derived fillers. The objective is to create a lightweight, structurally robust, and thermally stable material with potential for tailored conductivity, suitable for advanced applications. This protocol ensures reproducibility and systematic data collection.

1 Introduction and Objective

The goal of the Seshat's Bones project is to develop a sustainable alternative to petroleum-based composites without compromising performance. The central hypothesis is that a synergistic combination of an epoxidized hemp oil (EHO) resin matrix, functionalized hemp lignin, and micronized hemp biochar can produce a thermoset composite with superior mechanical and thermal properties. The optional inclusion of hemp nanosheets is explored to further enhance electrical conductivity and fracture toughness.

This protocol details the steps to create test coupons for validation against established ASTM standards.

2 Materials and Equipment

2.1 Required Materials

All materials must be logged with batch numbers and supplier certificates of analysis upon receipt.

Component	Specification / Grade	Example Source	Purpose
Epoxidized Hemp Oil	EEW: 250-300 g/eq (Must verify)	Bioresins Inc.	Resin Base
Modified Hemp Lignin	Kraft Lignin, esterified, ¡75 µm	Stora Enso	Structural/Thermal
Hemp Biochar	Micronized, ¡45 µm	Wakefield Biochar	Reinforcing Filler
Hemp Nanosheets	Liquid-phase exfoliated (Optional)	In-house / Graphene-Info	Conductive/Toughn
Bio-based Hardener	Diamine; AHEW: **145 g/eq**	**Priamine™ 1075**	Crosslinking Agent

Table 1: Component list for Seshat's Bones v1.1.

2.2 Required Laboratory Equipment

- Analytical Digital Scale (±0.001g) - Overhead Mechanical Stirrer with high-shear impeller
- Ultrasonic Homogenizer (Probe-type recommended)
- Ventilated Fume Hood
- Vacuum Oven with temperature control (up to 200°C)
- Vacuum Chamber with pump (¡1 torr)
- Silicone or polished Aluminum Molds (per ASTM spec for testing)
- Standard Personal Protective Equipment (PPE)
- Compression Press (Optional, for void reduction and fiber alignment)

3 Formulation and Stoichiometry

3.1 Target Formulation (by weight)

The following table provides the target compositional ranges. Initial prototypes should target the midpoint of each range.

Component	Percentage by Weight (%)
Epoxidized Hemp Oil (EHO)	40 – 50%
Modified Hemp Lignin	20 – 30%
Hemp Biochar	15 – 25%
Hemp Nanosheets (Optional)	2 – 5%
Hardener	Calculated Stoichiometric Ratio

Table 2: Prototype formulation ranges.

3.2 Hardener Stoichiometry Calculation

The precise amount of hardener is critical for optimal crosslinking. It is calculated as parts per hundred parts of resin (phr) based on the Epoxide Equivalent Weight (EEW) of the resin and the Amine Hydrogen Equivalent Weight (AHEW) of the hardener.

$$\mathrm{phr} = \frac{\mathrm{AHEW}}{\mathrm{EEW}} \times 100 \tag{1}$$

Example Calculation:

- Given EHO with an EEW of **280 g/eq**.
- Given Priamine™ 1075 hardener with an AHEW of **145 g/eq**.
- $\mathrm{phr} = \frac{145}{280} \times 100 \approx \mathbf{51.8\ phr}$
- This means for every 100g of EHO, 51.8g of hardener is required. Adjust mass based on the percentage of EHO in the total formulation.

4 Synthesis Procedure

Execute all steps within a ventilated fume hood.

1. **Filler Dispersion (if using nanosheets):**
   - Weigh the required amount of EHO into a primary mixing vessel.
   - Add the pre-weighed Hemp Nanosheets to the EHO.
   - Disperse using the Ultrasonic Homogenizer for 30-60 minutes, or until no visible agglomerates remain. Use pulsed cycles to avoid overheating.
2. **Matrix Preparation:**
   - Gently heat the EHO (or EHO/nanosheet mixture) to 60°C on a hot plate using an oil bath for uniform heating.
   - Set up the Overhead Mechanical Stirrer.
   - While stirring at moderate speed (300-500 RPM), slowly add the pre-weighed Modified Hemp Lignin and Hemp Biochar.
   - Continue stirring for 60 minutes at 60°C to ensure complete and uniform dispersion of fillers. The mixture will become viscous.
3. **Hardener Addition and Degassing:**
   - Turn off the heat and allow the mixture to cool to 40°C to prevent premature curing.
   - Add the stoichiometrically calculated amount of hardener.
   - Increase stirrer speed (800-1000 RPM) and mix vigorously for 3-5 minutes. **Note the time; the pot life has begun.**
   - Immediately transfer the vessel to the Vacuum Chamber and apply vacuum. Degas the mixture until bubbling subsides (approx. 15-20 minutes).
4. **Molding and Curing:**
   - Treat molds with a release agent prior to use.
   - Carefully pour the degassed mixture into the molds, avoiding air entrapment.
   - Place the molds in the Vacuum Oven.
   - **Initial Cure:** Cure at **80°C for 4 hours**.
   - **Post-Cure:** Increase temperature to **130°C and hold for 2 hours** to complete crosslinking and maximize thermal properties.
   - **Cool Down:** Turn off the oven and allow the samples to cool slowly to room temperature inside the oven over several hours to prevent thermal shock.
   - Once cooled, carefully demold the test coupons.

5 Characterization and Validation

The cured samples should be tested to validate their properties against theoretical targets.

Test Method	Standard	Property Measured
Tensile Properties	ASTM D638	Tensile Strength, Modulus, Elongation
Flexural Properties	ASTM D790	Flexural Strength, Modulus
Dynamic Mechanical Analysis (DMA)	ASTM D7028	Glass Transition Temp (Tg), Storage Modulus
Thermogravimetric Analysis (TGA)	ASTM E1131	Thermal Decomposition Profile, Stability
Dielectric Constant	ASTM D150	Electrical Insulation Properties
Density	ASTM D792	Material Density, Void Content
Scanning Electron Microscopy (SEM)	-	Fracture Surface Morphology, Dispersion Quality
Impact Strength (Izod/Charpy)	ASTM D256	Toughness, Impact Resistance

Table 3: Standard validation test matrix.

6 Safety and Documentation

- **PPE:** Always wear a lab coat, nitrile gloves, and chemical splash goggles. When handling powders or volatile chemicals, use a respirator with organic vapor cartridges.
- **Ventilation:** All mixing and curing steps involving uncured resin must be performed in a certified chemical fume hood.
- **Chemical Handling:** Review the Safety Data Sheet (SDS) for all chemical components before use.
- **Documentation:** Record every detail of the process in a dedicated lab notebook, including all weights, times, temperatures, and any visual observations. Label all samples clearly.