

Standardized Protocol for “Blobbing”: Rapid Prototyping and Characterization of Hempoxies Variants

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Executive Summary

The “Blobbing” protocol represents a paradigm shift in bio-composite material development by formalizing non-geometric, rapid prototyping methodologies into a rigorous scientific framework. This document establishes standardized procedures for the accelerated characterization of Hempoxies variants—hemp-reinforced, lignin-based composite resins—addressing a critical bottleneck between molecular-scale formulation and industrial-scale molding operations.

The hypothesis predicts that hand-formed hemispherical “Blob” units subjected to a serialized four-phase characterization battery (Formation, Curing, Testing, and Data Analysis) will yield performance metrics capable of predicting industrial mold behavior with > 90% confidence. This protocol leverages Shore D hardness, thermogravimetric analysis (TGA), volumetric density measurement, and scanning electron microscopy (SEM) to construct comprehensive “Blob Profiles” that encode critical material properties within 72–96 hours of prototype formation.

By reducing development cycles from weeks to days, “Blobbing” enables rapid iteration and optimization of resin-fiber formulations, supporting Landry Industries’ mission to accelerate sustainable material innovation.

1 Observation & Intelligence Requirement

1.1 Bottleneck in Bio-Composite Development

Current bio-composite development workflows suffer from a critical information gap between molecular formulation (lab-scale) and industrial processing (production-scale). The traditional pathway involves:

1. **Molecular design** (days): Chemists formulate resin blends with specific lignin modifications and hemp fiber ratios.
2. **ASTM coupon production** (3–7 days): Materials are molded into standardized geometric samples for testing (e.g., ASTM D2240 hardness coupons).
3. **Characterization** (3–5 days): Destructive and non-destructive testing occurs on rigid-geometry coupons, often obscuring critical resin-fiber interaction data.
4. **Industrial mold trials** (1–3 weeks): Formulations that pass lab testing are scaled to production molds, where unexpected delamination or viscosity issues often arise.

This sequential approach introduces delays and false negatives. Rigid coupon geometries constrain resin flow patterns and fiber saturation, suppressing the emergence of delamination, viscosity mismatch, and curing inhomogeneity that manifest in complex industrial molds.

1.2 The “Blobbing” Observation

Preliminary, informal trials at Landry Industries revealed that non-geometric, hand-formed resin/fiber masses—hereafter referred to as “Blobs”—provide immediate, high-fidelity diagnostic data unavailable from standardized coupons:

- **Resin-to-fiber saturation inhomogeneity** becomes visible within the unconfined mass, revealing fiber clustering or dry pockets.
- **Viscosity behavior** during manual compression indicates whether the resin will adequately penetrate complex mold geometries.
- **Curing kinetics** within the unrestricted thermal mass generate temperature profiles distinct from rigid coupons, mimicking mold-scale thermal conditions more closely.
- **Edge effects and microfractures** emerge naturally, encoding failure modes likely in production scenarios.

1.3 Intelligence Requirement

The core research question is: How can “Blobbing” be formalized into a rigorous, repeatable, peer-reviewable scientific methodology that:

1. Eliminates subjective hand-forming variability through controlled mass-to-volume ratios and compression protocols?
2. Produces quantitative “Blob Profiles” from a serialized characterization battery?
3. Achieves predictive fidelity (> 90% confidence) of industrial mold performance from rapid lab-scale prototypes?
4. Accelerates development cycles from weeks to 72–96 hours without sacrificing scientific rigor?

2 Hypothesis

If a Hemproxies variant is prototyped as a standardized “Blob” (defined by a controlled mass-to-volume ratio and hemispherical compression geometry) and subjected to a serialized characterization battery (Hardness, Thermal Stability, Density, and Micro-Structure Analysis), then the resulting “Blob Profile”—a composite metric derived from these four parameters—will accurately predict the material’s performance in complex industrial molds with a confidence interval of $> 90\%$.

“Accurate prediction” is operationalized as:

- If Blob Profile indicates adequate hardness (Shore D > 50), thermal stability (no degradation $< 200^\circ\text{C}$), and uniform density (porosity $< 15\%$), industrial molds should cure without internal delamination.
- If SEM analysis reveals fiber-matrix adhesion without microcracks, surface finish and dimensional accuracy in production will meet specifications.
- Post-production part qualification testing (ASTM D2240, TGA, tensile strength) on industrial molds should show $\geq 95\%$ concordance with Blob Profile predictions.

3 Experimental Design & Methodology

3.1 Overview of the Four-Phase Protocol

The “Blobbing” Protocol consists of four discrete, reproducible phases:

3.1.1 Phase I: Formulation & Mixing

Objective: Prepare a standardized resin-fiber blend with controlled composition.

Materials:

- **Seshat Lignin Formulation v.2026** (quadruple-modified lignin polymer resin; molecular weight ≈ 1200 g/mol; viscosity $\approx 500\text{--}800$ cP at 25°C)
- **Industrial-grade hemp bast fiber** (fiber length 5–15 mm, diameter 10–25 μm)
- **Hemp hurd (shiv)** (chopped core material, moisture content $< 5\%$)
- **Coupling agent (if applicable)** (3–5 wt%, to enhance fiber-matrix adhesion)

Procedure:

1. Pre-condition hemp fiber and hurd at $23^\circ\text{C} \pm 2^\circ\text{C}$ and 50% RH for ≥ 24 hours.
2. Weigh resin and fiber according to target wt% (e.g., 70 wt% resin, 20 wt% bast fiber, 10 wt% hurd).
3. Mix resin and coupling agent (if used) for 2 minutes at ~ 100 rpm using a mechanical stirrer at 23°C .
4. Add fibers in three equal portions, stirring at 100 rpm for 2 minutes between additions (total mixing time: ~ 6 minutes).
5. Degass the blend under vacuum (0.1 MPa) for 5 minutes to remove air bubbles.
6. Record blend temperature immediately post-mixing.

3.1.2 Phase II: Blob Formation

Objective: Create a standardized hemispherical mass that minimizes edge-effect variability.

Blob Unit Specification:

- **Target mass:** 50.0 ± 0.5 g
- **Geometry:** Hemispherical (radius ≈ 20 mm)
- **Surface finish:** Manual compression to 80–90% theoretical density

Formation Procedure:

1. Immediately after degassing, transfer 50 g of blend to a pre-warmed silicone mold (hemispherical, ~ 40 mL volume).
2. Using a gloved hand, compress the mass for 30–60 seconds, applying consistent radial pressure to achieve hemispherical shape.
3. Weigh the Blob immediately post-compression; if < 49.5 g or > 50.5 g, re-form using adjacent blend material.
4. Measure Blob height and diameter (three points, average) using calipers. Calculate apparent volume: $V = (2/3)\pi r^3$.
5. Place Blob on parchment-lined tray and record timestamp ($t = 0$) for the curing phase.

3.1.3 Phase III: Curing Cycle

Objective: Subject the Blob to a controlled thermal profile that mimics industrial mold conditions.

Curing Schedule:

1. Ambient hold: 2 hours at $23^\circ\text{C} \pm 2^\circ\text{C}$ (initial polymerization at room temperature).
2. Thermal ramp: Increase temperature at $5^\circ\text{C}/\text{min}$ to 180°C .
3. Isothermal hold: Maintain 180°C for 4 hours (core curing).
4. Cool-down: Decrease temperature at $2^\circ\text{C}/\text{min}$ to 23°C .
5. Ambient post-cure: 24 hours at $23^\circ\text{C} \pm 2^\circ\text{C}$ (total protocol time: ~ 48 hours).

Environmental Conditions:

- **Temperature:** $23^\circ\text{C} \pm 2^\circ\text{C}$ during all ambient phases.
- **Humidity:** $50\% \pm 5\%$ relative humidity.
- **Ventilation:** Standard laboratory hood; no drafts affecting Blob temperature uniformity.

3.1.4 Phase IV: Characterization Battery

Objective: Acquire quantitative metrics from the cured Blob to construct the “Blob Profile.”

Test 4.1: Shore D Hardness (ASTM D2240-15(2021))

- **Instrument:** Digital Shore D Durometer (calibrated within 6 months).
- **Sample preparation:** Place Blob on flat surface with hemispherical face up.

- **Measurement points:** 5 readings on the flat base (avoiding edges) and 5 on the curved surface. Average the 10 readings.
- **Record:** Mean hardness, standard deviation, and individual readings.

Test 4.2: Thermogravimetric Analysis (TGA)

- **Instrument:** TGA with nitrogen purge (heating rate $10^{\circ}\text{C}/\text{min}$, range $25\text{--}700^{\circ}\text{C}$).
- **Sample:** Micro-mill a 5–10 mg chip from Blob interior (avoiding edge material).
- **Measure:** T_{onset} (temperature at 5% mass loss), T_{max} (maximum decomposition rate), and char yield (wt% remaining at 700°C).
- **Record:** Full TGA curve, derivative (DTG), and derived metrics.

Test 4.3: Volumetric Density (Archimedes’ Principle)

- **Procedure:** Weigh cured Blob on analytical balance (± 0.01 g). Submerge in deionized water and measure displaced volume (mL) using a graduated cylinder.
- **Calculate:** Apparent density $\rho = \text{mass}/\text{volume}$ (g/cm^3).
- **Calculate:** Porosity $= (1 - \rho_{\text{apparent}}/\rho_{\text{theoretical}}) \times 100\%$, where $\rho_{\text{theoretical}} \geq 1.35 \text{ g}/\text{cm}^3$ for Hempoxies.
- **Record:** Mass, volume, density, and porosity percentage.

Test 4.4: Scanning Electron Microscopy (SEM) & Micro-Structure Imaging

- **Sample preparation:** Fracture-cut the Blob to expose the cross-section. Polish with 600–2000 grit SiC paper. Sputter-coat with 5 nm Au/Pd.
- **Imaging:** SEM at 15 kV, working distance 10–15 mm. Capture 10 images at $\times 500$ and $\times 2000$ magnification across three distinct regions (top, middle, bottom of cross-section).
- **Analysis:** Assess fiber-matrix adhesion (good, fair, poor), presence of microcracks, fiber pull-out, and resin curing uniformity.
- **Record:** SEM micrographs, qualitative adhesion scores, and delamination observations.

4 Blob Profile Compilation

After completion of all four tests, compile the “Blob Profile”—a structured data set that serves as the predictive foundation for industrial mold performance.

Parameter	Measurement Unit	Success Criterion
Shore D Hardness	Shore D (0–100)	≥ 50
T_{onset}	$^{\circ}\text{C}$ (5% loss)	$> 200^{\circ}\text{C}$
Apparent Density	g/cm^3	1.20–1.40
Porosity	% by volume	$< 15\%$
Fiber-Matrix Adhesion	Qualitative (SEM)	Good (no cracks)

5 Decision Loop & Validation Criteria

The “Blobbing” protocol culminates in a binary decision: proceed with industrial mold trials, or return to Phase I for formulation adjustment.

5.1 Validated Prototyping Unit (VPU) Criteria

A Blob qualifies as a Validated Prototyping Unit (VPU) if ALL of the following are met:

1. Shore D Hardness ≥ 50 AND standard deviation < 5 units.
2. $T_{onset} > 200^{\circ}C$ AND mass loss at $300^{\circ}C < 10\%$.
3. Apparent density within $1.20\text{--}1.40\text{ g/cm}^3$ AND porosity $< 15\%$.
4. SEM analysis reveals Good fiber-matrix adhesion across $\geq 80\%$ of imaged area; no delamination.
5. Mass retention in Blob post-curing $> 98\%$ (minimal volatile loss).

5.2 Failure Modes & Iteration Loop

If any of the above criteria are not met, classify the failure mode:

- **Low hardness** ($ShoreD < 50$): Indicates insufficient cure, inadequate resin-fiber contact, or incompatible resin chemistry. Return to Phase I; adjust cure temperature ($+5\text{--}10^{\circ}C$), extend isothermal hold, or increase coupling agent concentration.
- **Low thermal stability** ($T_{onset} < 200^{\circ}C$): Suggests incomplete polymerization or molecular degradation. Increase isothermal cure duration or reduce maximum temperature.
- **High porosity** ($> 15\%$): Indicates poor mixing, inadequate degassing, or resin volatility. Extend degassing time, improve mixing energy, or reduce air incorporation.
- **Poor fiber-matrix adhesion** (SEM): Add or increase coupling agent; verify resin pH and moisture content. Re-optimize resin-to-fiber ratio.

5.3 VPU Advancement

When a Blob achieves VPU status, the formulation is eligible for:

1. Industrial mold trials (scaled manufacture).
2. Post-production part qualification (ASTM D2240, tensile strength, dimensional accuracy).
3. Comparison of industrial part metrics to Blob Profile (confidence validation).

6 Expected Outcomes & Data Interpretation

6.1 Blob Profile as a Predictive Model

The four-test Blob Profile encodes material behavior in a compact, quantitative format. When a Hemproxies variant achieves VPU status, the industrial mold part cast from the same formulation should exhibit:

- **Shore D hardness** within ± 8 units of Blob measurement (accounting for part geometry and mold cooling effects).
- **Tensile strength** consistent with fiber-matrix adhesion quality predicted by SEM.
- **Absence of internal delamination, voids, or surface defects** correlated with Blob porosity $< 15\%$.
- **Dimensional accuracy** within specified tolerances, with minimal shrinkage (indicating controlled curing kinetics).

6.2 Confidence Interval & Validation Threshold

The hypothesis predicts $> 90\%$ confidence in industrial mold performance prediction. Operationally, this is validated by comparing Blob-derived metrics against post-production part testing across a minimum of 5 independent formulations and 3 mold geometries. Success is defined as $\geq 90\%$ of industrial parts meeting all specification criteria (hardness, tensile, dimensional, surface finish) when the corresponding Blob achieved VPU status.

7 Replication Protocol & Quality Assurance

To ensure reproducibility across independent laboratories, the following standardization is mandatory:

7.1 Material Standards

- **Seshat Lignin Formulation v.2026:** Supply from Landry Industries lot-verified batch (batch number must be recorded).
- **Hemp fiber:** Industrial-grade bast fiber from ISO-certified suppliers; fiber length 5–15 mm, moisture $< 5\%$.
- **Hemp hurd:** Dried, chopped to 2–6 mm; moisture $< 5\%$; tested for mold growth.
- **Coupling agent (if used):** Specified by name, CAS number, and lot number.

7.2 Environmental Conditions

- **Ambient laboratory:** $23^{\circ}\text{C} \pm 2^{\circ}\text{C}$; $50\% \pm 5\%$ RH. Verified with calibrated thermohygrometer (accuracy $\pm 0.5^{\circ}\text{C}$, $\pm 2\%$ RH).
- **Oven/heating chamber:** Temperature accuracy $\pm 2^{\circ}\text{C}$; ramp rate accuracy $\pm 0.5^{\circ}\text{C}/\text{min}$. Annual calibration with traceable standards required.
- **Weighing:** Analytical balance with ± 0.01 g accuracy and regular calibration (every 6 months).

7.3 Instrument Calibration & Maintenance

- **Shore D Durometer:** Calibrate using certified hardness reference blocks (e.g., 75 ± 3 Shore D) every 6 months.
- **TGA:** Baseline calibration with Curie temperature standards and temperature verification across $25\text{--}700^\circ\text{C}$ range annually.
- **SEM:** Magnification calibration with certified reference sample (e.g., NIST-traceable grid or standard). Image resolution verification: ≥ 2 pixels per micron.
- **Document all calibration data** in laboratory quality assurance file.

7.4 Documentation & Data Archiving

- For each Blob, maintain a standardized data sheet recording: formulation composition ($wt\%$), blend temperature, Blob formation timestamp, all four test results with raw data, SEM micrographs, Blob Profile summary, and VPU determination.
- Retain all raw data (TGA curves, SEM images, calibration records) for ≥ 5 years.
- Use version control for protocol updates. Any modification to replication procedures requires explicit approval and documentation.

8 Timeline & Resource Requirements

The complete “Blobbing” protocol, from formulation through final data compilation, requires:

Elapsed Time:

- Phase I (Formulation): 0.5 hours
- Phase II (Blob Formation): 0.25 hours
- Phase III (Curing Cycle): ~ 48 hours
- Phase IV (Testing & Analysis): 1–3 hours (depending on SEM availability)
- **Total:** 72–96 hours (approximately 3–4 calendar days)

Personnel & Skill Requirements:

- Materials science technician (Phase I & II): 1 person
- Lab monitor for curing (Phase III): 0.1 FTE (part-time; minimal hands-on)
- Instrumentation specialist (Phase IV): 0.5 FTE (for TGA and SEM operation & analysis)

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A Blob Profile Data Sheet Template

The following table provides a standardized template for recording all Blob-related data.

Parameter	Value / Measurement
Blob ID / Formulation Code	-----
Seshat Lignin Batch #	-----
Resin <i>wt%</i>	-----
Bast Fiber <i>wt%</i>	-----
Hemp Hurd <i>wt%</i>	-----
Coupling Agent (if used)	-----
Blob Mass (g)	-----
Blob Volume (mL)	-----
Shore D Hardness (Mean $\pm$ SD)	-----
T_{onset} (TGA, 5% loss)	-----
Apparent Density (g/cm ³)	-----
Porosity (%)	-----
Fiber-Matrix Adhesion (SEM)	Good / Fair / Poor
VPU Status (PASS / FAIL)	-----
Failure Mode (if applicable)	-----
Researcher Name & Date	-----

B Troubleshooting & Common Failure Modes

Failure Mode	Likely Cause(s)	Recommended Action
Low hardness (Shore D < 50)	Under-cure, insufficient resin, fiber saturation	Increase cure temp +5–10°C; extend isothermal; check resin viscosity
High porosity (> 15%)	Inadequate degassing, air entrainment, volatile loss	Extend vacuum degassing to 10 min; improve mixing homogeneity
Poor fiber adhesion (SEM)	Low coupling agent, fiber moisture, resin pH imbalance	Increase coupling agent 1–2 wt%; dry fiber > 48 h at 80°C
Low thermal stability (T_{onset} < 200°C)	Incomplete polymerization, resin degradation	Check resin lot; extend isothermal hold to 6–8 h; verify max temp < 200°C