

Article

Feed-Controlled Filament Extrusion of High-Loading Micronized Soy Hull Fiber/PLA Biocomposites for Fused Deposition Modeling

Muneeb Tahir , Tri Vu and Abdel-Fattah M. Seyam 

Wilson College of Textiles, North Carolina State University, 1020 Main Campus Dr., Raleigh, NC 27606, USA; tdvu@ncsu.edu

* Correspondence: mtahir2@ncsu.edu (M.T.); aseym@ncsu.edu (A.-F.M.S.)

Highlights

What are the main findings?

- Filament-making required a compaction-oriented route distinct from dispersion-oriented compounding.
- A 3.85 mm die and 160/170/180/195 °C profile enabled spool-fed filament at 30 wt.% SHF.

What are the implications of the main findings?

- Phase-resolved logging across 14 trials distinguished controlled and directional instability states.
- The best 10SHF run retained 95.45% within 1.70–1.80 mm, with 0.013% above 1.89 mm.
- The route supported 720 printed specimens over approximately 936 h; 35SHF exceeded the spool-fed handling limit.

Abstract

This study reports the filament-making stage of a sequential single-screw process-development pathway for compatibilizer- and plasticizer-free soy hull fiber (SHF)/PLA biocomposites used in fused deposition modeling. Thirty-three filament-making trials were interpreted through an event-linked process chain, and 14 trials were evaluated using phase-resolved in-line diameter records and capability-style Cp/Cpk metrics. Filament-making converged on a single-mixing-zone screw, a 3.85 mm orifice/5.75 mm land die, 10 rev/min, and a 160/170/180/195 °C barrel profile for 10–30 wt.% SHF, whereas neat PLA required 180/185/200/205 °C. The strongest sustained benchmark was a 10SHF filament produced under converged settings, with a mean diameter of 1.7411 mm, a standard deviation of 0.0236 mm, 95.45% of readings within 1.70–1.80 mm, and only 0.013% above 1.89 mm. Feed replenishment, depletion, irregular pellets, recycled material, and fines-rich feed shifted the same nominal configuration among controlled and unstable states. The highest reliably spool-fed formulation was 30 wt.% SHF. The 35SHF filament remained nozzle-depositable from loose coils but fractured repeatedly during take-up and direct spool unwinding in 3D printing. Operational validation of all four converged filament formulations comprised 720 printed mechanical-test specimens over approximately 936 h. The reported conditions define platform-specific operating windows, but the process insights hold global relevance for pellet-based extrusion systems.



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Keywords: soy hull fiber; PLA biocomposites; filament extrusion; feed continuity; in-line diameter monitoring; fused deposition modeling

1. Introduction

Fused deposition modeling (FDM), also referred to as fused filament fabrication (FFF), provides an accessible route for manufacturing geometrically complex polymer components with relatively low tooling needs and material wastage. Polylactic acid (PLA) is widely used in FDM because it is commercially available, bio-based, stiff, and processable using common material extrusion equipment. Incorporating lignocellulosic residues into PLA can further reduce polymer consumption, valorize agricultural byproducts, and modify stiffness, shrinkage, and thermal behavior. The present study examines micronized soy hull fiber (SHF), derived from soybean hulls, an abundant lignocellulosic processing byproduct, as a reinforcement that can partially displace PLA while valorizing an agricultural residue [1]. However, SHF and other natural fibers introduce moisture sensitivity, irregular morphology, fiber–fiber interactions, interfacial incompatibility, agglomeration, void formation, and reduced polymer-phase continuity [2–6]. Consequently, successful incorporation of SHF into PLA does not by itself establish downstream filament manufacturability.

Filament is a critical intermediate product because its dimensional, rheological, and mechanical characteristics govern printer feeding, melting, material delivery, and deposition consistency. Insufficient stiffness can prevent effective force transmission through the printer feed path, whereas excessive stiffness or brittleness can promote buckling, fracture during winding, or breakage during printer feeding. Feedability, therefore, depends on both the filament properties and the forces imposed by the particular printer architecture; predictive relationships established for one feeding system may not transfer directly to another [7–9]. These constraints are particularly consequential for high-loading SHF/PLA filaments, in which increasing reinforcement can increase stiffness while reducing flexibility and handling tolerance.

Filament diameter is likewise not only a final product specification. Variations in feed rate, screw filling, screw speed, pressure generation, die flow, cooling rate, drawdown, and winding can be transmitted as fluctuations in diameter and ovality. Such variations alter volumetric delivery during printing and can cause inconsistent deposition, surface defects, gaps, excessive overlap, feed resistance, jamming, or print failure [10–13]. Filament diameter can therefore function as an indirect process signal reflecting disturbances in solids feeding, melting, pressure stability, cooling, drawdown, and take-up. This insight is especially relevant to high-loading SHF/PLA feedstocks, for which changes in compounded-pellet condition or hopper discharge may destabilize extrusion even when the nominal machine settings remain unchanged.

Plant-derived reinforcements and fillers from date palm, cocoa husk, coffee silver skin, banana rachis, yucca, hemp shive, and forest industry residues have been incorporated into PLA-based FDM filaments, demonstrating the broad feasibility of agricultural residue valorization [14–20]. Higher-loading melt-processed systems have also been reported, including PLA filaments containing up to 30 wt.% hemp or harakeke fiber, 40 wt.% hemp hurd, 30 wt.% regenerated cellulose fiber, and 50 wt.% wood powder [21–25]. Collectively, these studies confirm that elevated plant-derived reinforcement contents can be converted into filament and used in FDM. However, the reported loading levels were achieved using different reinforcement morphologies and treatments, matrix formulations, compounding and filament-production routes, filament geometries, extrusion and take-up systems, and printer configurations. They therefore represent system-specific demonstra-

tions rather than a common experimental basis for defining transferable maximum loading. This non-transferability arises because fiber morphology, surface chemistry, lignin content, treatment, aspect ratio, dispersion, and interfacial behavior can produce substantially different flow, porosity, adhesion, and mechanical durability responses at equivalent nominal loadings [3,26]. The highest loading demonstrated in printing for flax-, hemp-, regenerated cellulose-, wood-, or other residue-filled PLA, therefore, does not establish an equivalent processability boundary for micronized SHF/PLA.

Prior studies involving soybean-hull-derived reinforcement in filament systems have examined non-PLA matrices, including flexible thermoplastic copolyester and a soy-protein-containing formulation, and have related fiber treatment and loading to rheology, surface quality, pore formation, moisture uptake, mechanical behavior, and printed-part performance [4–6,27]. Soybean hull/PLA filament has also been produced while varying hull-particle size, mixing ratio, and processing conditions [28]. The unresolved issue addressed in the present work is the detailed process pathway by which high-loading, compounded SHF/PLA pellets are converted into nominally 1.75 ± 0.05 mm, mechanically handleable, spool-fed filament, and how disturbances inherited from compounding or introduced during filament-making affect that conversion.

Two-stage composite-filament workflows commonly employ melt compounding followed by pelletization and extrusion of the resulting feedstock through a separate filament-making system [21–23,25]. This practice establishes that compounding and filament production may use different equipment and operating conditions. It does not, however, determine whether a screw or thermal architecture developed for compounding can be transferred directly to filament-making, particularly when both stages are performed on the same underlying extrusion platform. Direct experimental evidence remains limited in melt-processed plant fiber/PLA systems regarding the consequences of such transfer and how a processing mismatch propagates through filament production to final dimensional control.

The present study is interpreted through a coupled particulate-feeding and single-screw polymer-processing framework. Under conventional flood-fed operation, solids conveying depends on frictional drag, progressive channel filling, and compaction of a coherent pellet bed. Transient partial filling caused by restricted hopper discharge, bridging, or depletion can alter axial pressure development, residence time, melting behavior, melt temperature, and output [29,30]. Fluctuations in feed rate, screw operation, and melt delivery can subsequently propagate into filament diameter and mechanical quality [12,13]. Die geometry, screw speed, cooling distance, and downstream drawdown provide an additional control layer because they determine the relationship between extruder output and final filament dimensions [11].

The working process chain is therefore: inherited pellet condition → hopper discharge and feed continuity → screw-channel filling and solids-bed compaction → melting and melt continuity → die flow and pressure release → cooling and drawdown → in-line measurement and take-up → filament dimensional control, internal continuity, handling, and printability. The framework does not assume that the present extruder was deliberately operated in a controlled starve-fed mode. Partial-fill theory is used only to interpret transient underfilling caused by irregular or interrupted gravitational feeding.

Against this background, the present study addresses three specific process-development gaps. First, detailed evidence remains limited for reconstructing both successful and unsuccessful operating states during the conversion of high-loading, melt-compounded agricultural residue/PLA pellets into filament. Second, compounded pellets are commonly reported only as intermediate feedstock, while the downstream consequences of their physical condition, heterogeneity, and bulk-flow behavior are rarely traced systematically through feeding, extrusion, cooling, and take-up. Third, although in-line diameter monitor-

ing and process capability analysis have precedent in filament and additive-manufacturing research [11–13], their phase-resolved integration with documented feed-state events to distinguish directional oversize, undersize, and mixed filament-instability modes remains underdeveloped in melt-processed plant fiber/PLA systems.

The preceding stage of this research developed single-screw compounding routes for compatibilizer- and plasticizer-free PLA containing 0, 10, 20, and 30 wt.% micronized SHF and evaluated 35 wt.% SHF as a high-loading stress-test formulation [31]. That work established the compounded-pellet populations and material-processing lineage used in the present study.

Accordingly, the objectives of this study are to: (i) directly test the transferability of the compounding-stage screw and thermal architectures to filament-making on the same extrusion platform; (ii) determine how inherited pellet condition, hopper fill state, feed continuity, screw charging, air carry-forward, die geometry, cooling, drawdown, and take-up affect filament continuity and dimensional control; (iii) relate continuous in-line diameter records and capability-style metrics to documented process events and directional filament-instability modes; (iv) evaluate the effect of SHF loading on filament cross-sectional internal discontinuity; and (v) determine the highest SHF loading that can be converted into dimensionally usable, mechanically handleable, spool-fed filament on the present Brabender/Filabot platform and validate this through sustained FDM printing.

The contribution of this study is an event-linked, phase-resolved framework for diagnosing how feedstock and operating-state disturbances propagate through agricultural fiber/PLA filament production. By identifying the stages at which these disturbances compromise filament continuity, dimensional control, handling, and downstream feedability, the framework can support reductions in material waste, production interruptions, and unsuccessful scale-up. Although the reported loading boundary is specific to the present SHF/PLA material and Brabender/Filabot platform, the diagnostic framework may inform the development of other lignocellulosic/PLA filament systems.

2. Materials and Methods

2.1. Study Design and Trial Coding

In the trial coding, the prefix F denotes a filament-making run; the middle number denotes SHF loading; and the terminal number denotes the chronological sequence within each stage–formulation combination. Supplementary Tables S6.1A–S6.1D provide the complete 33-trial registry, including feedstock lineage, principal configuration changes, outcomes, pivotal observations, and linkage to the corresponding mechanism-based discussion in the main manuscript.

The filament-making trials were sequential process-development experiments rather than independent statistical replicates. A formulation or machine configuration was revisited when a preceding trial identified a failure mode, or when a specific intervention required evaluation. Because parent-pellet population, hopper history, material state, line adjustment, or the intervention under study could differ among nominally similar trials, repeated trial codes were not treated as true replicates.

Trial identifiers are retained for traceability but are not used as the primary organizational structure of the Results and Discussion. The main-text evidence is grouped by process function, feed state, failure mode, and formulation boundary, whereas the complete chronological trial history remains in Supplementary Section S6.

In this study, “converged” denotes an operationally converged configuration rather than a statistically optimized or production-qualified optimum. A configuration was classified as converged when it: (i) removed or substantially reduced the recurrent failure mode targeted by the preceding development sequence; (ii) supported continuous pellet

feeding under the specified hopper-agitation practice and continuous strand take-up without recurrent feed-throat blockage, melt backflow in the extruder, or strand fracture; (iii) produced dimensionally usable and mechanically handleable filament over a documented operating interval; and (iv) was retained in subsequent production or transfer trials and supplied filament for downstream printing. A favorable but short-lived segment within a disturbed run is described as a controlled operating window rather than as complete process convergence. No Cp or Cpk cutoff was used to declare convergence.

The principal process-chain variables used to interpret filament manufacturing are summarized in Table 1.

Table 1. Process-chain variables used to interpret filament manufacturing.

Process Stage	Principal Variables	Primary Response Examined
Inherited feedstock	Pellet size and shape, fines, texture, moisture condition, parent-batch history	Hopper discharge, interlocking, and compositional continuity
Hopper and feed throat	Fill level, replenishment, agitation, bridging, depletion	Screw replenishment and feed continuity
Screw and barrel	Screw architecture, speed, feed-throat cooling	Interpreted effects on channel filling, solids-bed consolidation, melting, and residence behavior, and melt continuity
Die and cooling	Orifice diameter, land length, die temperature, airpath relative position, and cooling intensity	Strand continuity, swelling, surface condition, and solidification
Measurement and take-up	Gauge alignment, vibration, draw distance, take-up speed, spool fill	Diameter trace, dimensional distortion, winding stability, handling

2.2. Materials

PLA (Ingeo biopolymer 4043D, NatureWorks LLC, Minneapolis, MN, USA) was used as the matrix polymer. Soy hull fibers (SHFs) (FI-1 grade, Unique Soy LLC, Cumberland, MD, USA) were used as the fibrous additive phase. The fibers have reported lengths of 40–70 μm and widths of 5–7 μm and are produced from ground soy hulls processed through alkaline treatment, washing, hydrogen peroxide bleaching, drying to 92–95% solids, and hammer milling [1]. All filament-making trials used compounded pellets produced during the compounding stage; the compounding conditions are reported in Part I.

2.3. Equipment

The filament-making experiments were performed at the North Carolina State University Wilson College of Textiles' Textile Technology and Filament Laboratory. A single-screw extruder (C.W. Brabender Instruments Inc., South Hackensack, NJ, USA) with a length-to-diameter ratio of 25:1 was used during both the compounding and filament-making stages. The extruder featured four independently controlled heating zones, a die-section heating band with a separate temperature control knob, and a dedicated feed-throat cooling system to limit premature softening of PLA in the solids-conveying region. Die-section temperature was not a controlled variable in early development.

After early screw-assignment screening, the single-mixing-zone screw was adopted for all converged and subsequent filament-making trials. Early non-converged filament-making trials using the double-mixing-zone screw inherited from compounding and a no-mixing-zone screw are discussed in Section 3.2 because they established the stage-specific screw-role requirement. The converged single-mixing-zone screw selection was 32 in. (812.8 mm) in length and 1.25 in. (31.75 mm) in diameter. In the installed configuration, the screw extended from the unheated drive section into four heated barrel zones. Heating zones 1 and 2 corresponded to the heated solids-conveying/compression region, where compounded pellets were compacted, softened, and progressively converted from

a discrete pellet bed into a developing melt. Heating zone 3 corresponded to the shallow metering/conveying region, where the melt was transported and thermally stabilized before final mixing. Heating zone 4 corresponded to the only mixing element on the screw and the short, shallow-flight section immediately upstream of the die (Figure 1). A 3.85 mm orifice-diameter die with a 5.75 mm die land was used for filament-making. Die-section surface temperatures were monitored at four fixed locations using a handheld infrared thermal gun.

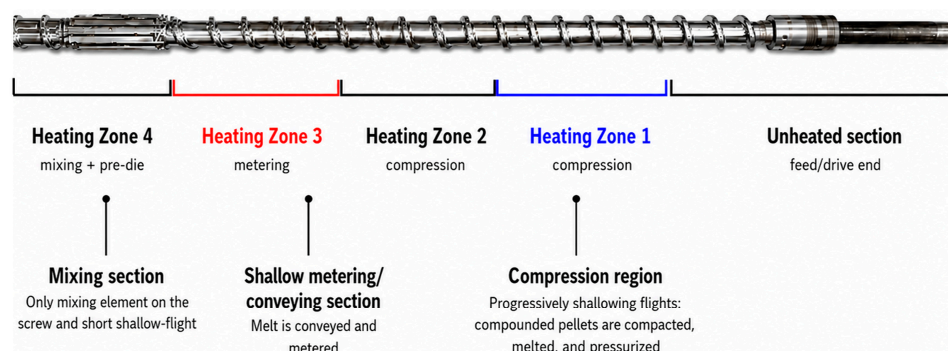


Figure 1. Single-mixing-zone screw configuration used for filament-making.

During filament-making trials, compounded pellets were loaded directly into the extruder hopper rather than through the volumetric feeder. The L-shaped hopper agitator described in Supplementary Section S1 was used for SHF-loaded pellet feedstocks. Extrudate cooling was provided by a custom-built air-cooling system consisting of six fans arranged in two modular assemblies, one commercial modular air-cooling path (Filabot Air Path, Barre, VT, USA), and an air conditioning unit (SRCOOL12K, Tripp Lite, Woodridge, IL, USA) for the provision of chilled air. The air conditioning unit was used after it was established that filament reaching the Mitutoyo diameter gauge (Mitutoyo, Aurora, IL, USA) in a still-soft state could be compressed by the gauge anvil, producing artificially low readings. In-line filament diameter was monitored using a Mitutoyo diameter gauge, with real-time diameter data transmitted to a computer running Filalogger software V1.5.5 (Barre, VT, USA). Filament winding and take-up were performed using a Filabot Spooler Precision Winder (Barre, VT, USA). The 500 g spools used for filament take-up were also sourced from Filabot (Barre, VT, USA).

A fused deposition modeling 3D printer (CR-10 Smart Pro, Creality, Shenzhen, China) was used for downstream print validation of the produced filaments. The printer featured a direct-drive geared filament-feed system, a 0.4 mm printing nozzle, and a heated build platform. The available build volume was 300 × 300 × 400 mm.

2.4. Filament-Making Route

For self-containment, the parent feedstocks were produced on the same Brabender platform during the preceding compounding stage using a dispersion-oriented double-mixing-zone screw and a 2.75 mm orifice-diameter, 5.75 mm land-length die. The production feedstocks contained 0, 10, 20, and 30 wt.% SHF in the PLA matrix, while the 35 wt.% SHF formulation was carried forward as a high-loading stress-test material. The complete compounding-stage process and trial history are reported in [31].

During filament-making, the compounded pellets were remelted and extruded as nominally 1.75 mm filament using the single-mixing-zone screw and the 3.85 mm orifice-diameter, 5.75 mm land-length die. The objectives of this stage were continuous pellet feeding and screw charging, sustained strand formation, dimensional control, sufficient solidification before in-line diameter measurement and take-up, and reliable filament

handling; additional fiber dispersion was not an explicit objective. For SHF-loaded formulations, the composite pellets were dried at 80 °C for 4.5–6 h before filament-making. The 0SHF pellets were dried at 70 °C for 4.5–6 h because pellet fusion had been observed at 80 °C. Pellets were introduced by direct hopper loading rather than volumetric feeding. Formulation-specific hopper agitation, a standardized airpath-to-die distance, and supplemental chilled-air cooling were applied as summarized in Table 2.

Table 2. Converged filament-making conditions.

Parameter	SHF-Loaded (10–30 wt.%)	0SHF
Feed form	Compounded SHF/PLA pellets	One-time extruded PLA pellets to mimic the compounding process
Pellet drying (°C, hours)	80, 4.5–6	70, 4.5–6
Screw	Single mixing zone	Single mixing zone
Feed system	Direct hopper loading	Direct hopper loading
Airpath-to-die distance (in./mm)	~1/25	~1/25
Air conditioning	On	On
Screw speed (rev/min)	10	10
Barrel profile (°C) heating zones Z1/Z2/Z3/Z4	160/170/180/195	180/185/200/205
Die orifice (mm)	3.85	3.85
Die land (mm)	5.75	5.75
Hopper agitation	L-shaped, automated	L-shaped, automated
Die temperature control setting and die section temperature ranges at the setting	50 (before extrusion): at barrel before heating band: ~160–172.5 °C; at die holder/bracket with heating band: ~144–153 °C; at heating band: ~125.7–131 °C; at die exterior: ~147–154 °C	50 (before extrusion): at barrel before heating band: 174.8 °C; at die holder/bracket with heating band: 133–136 °C; at heating band: 129.9 °C; at die exterior: ~152 °C
	50 (after extrusion): at barrel before heating band: ~164–179 °C; at die holder/bracket with heating band: ~132–160 °C; at heating band: ~126.3–141 °C; at die exterior: die exterior N/A due to close airpath-to-die position	50 (after extrusion): at barrel before heating band: ~175 °C; at die holder/bracket with heating band: ~125.3 °C; at heating band: ~139.5 °C; at die exterior: die exterior N/A due to close airpath-to-die position

After pelletization, compounded pellets intended for each experimental-design filament-production run were retained according to parent compounding batch and manually mixed within the collection container to reduce collection-order stratification. The resulting pellet population was divided into three sequential feed batches, transferred to separate permeable bags, and dried before filament-making. The relationship among these feed batches, filament-production phases, physical spools, and diameter-data files is defined in Section 2.5. In diagnostic trials involving pellets from different parent compounding runs or visibly different pellet populations, those populations were retained separately and introduced sequentially so that their downstream feeding behavior could be traced.

2.5. Production-Phase Designation

For the experimental-design filament-production runs, the three dried pellet feed batches were introduced into the filament-extrusion hopper in a predetermined beginning–middle–end sequence. Filament collected while each feed batch progressed through the filament-making run was assigned prospectively to the corresponding beginning, middle, or end production phase. Each physical spool and its associated diameter-data file were assigned in their entirety to the active phase at the time of collection. A phase could contain one physical spool or multiple consecutively collected spools; changing spools within the same phase did not create a new phase.

Beginning material represented the earliest retained filament associated with the first feed batch and could include startup, line-threading, stabilization, or initial hopper-state transients. Middle material represented filament associated with the second feed batch but was not assumed automatically to represent steady-state operation. End material represented the later retained filament associated with the final feed batch and could reflect hopper depletion, fines accumulation, later-feedstock characteristics, or other run-out effects.

The three phases were not necessarily equal in processing duration, retained filament length, pellet mass delivered during usable collection, spool mass, number of physical spools, or number of in-line diameter readings. Extrusion instability, discarded transition material, variation in production rate, spool capacity, and usable-filament yield affected the amount of material retained within each phase. Material hold-up within the hopper, screw, barrel, and die also prevented an instantaneous material transition when one feed batch followed another. The phases therefore represent sequential temporal production positions rather than compositionally isolated pellet populations or independent processing replicates.

For nonstandard diagnostic trials, including trials involving incomplete filament collection, recycled-material introduction, or intentionally segregated parent-pellet populations, phase designations were retained only when supported by explicit spool labels and contemporaneous trial records. Where only one or two phases could be assigned defensibly, the available phases were retained without reconstructing a missing phase. For the downstream experimental design, the B, M, and E phase categories were retained as spool-position factors even when a phase comprised more than one physical spool. The abbreviations B, M, and E are used in tables, specimen identifiers, and source-data filenames, whereas beginning, middle, and end are written in the main-text discussion.

2.6. Filament Cross-Section SEM and Apparent Dark-Area Analysis

Cross-sectional morphology of converged-setting 3DP filaments was examined by SEM. Representative 0SHF, 10SHF, 20SHF, and 30SHF filament samples were sectioned transversely using a blade and imaged without sputter coating or mounting using a TM4000 tabletop SEM (Hitachi, Tokyo, Japan) in backscattered-electron (BSE) mode at 15 kV. Images were acquired at 50 $\times$, 80 $\times$, and 180 $\times$ magnification. One full cross-sectional field was available per formulation at each magnification; the native 2560 $\times$ 1920-pixel 50 $\times$ image was the analysis unit used for the quantitative image audit.

Descriptive apparent internal dark-area fractions, rather than statistically representative bulk porosity values, were calculated from the native-resolution 50 $\times$ BSE images using a specimen-segmented fixed-threshold workflow. The lower 10% of each image was removed to exclude the scale bar. The filament body was separated from the external SEM background by Otsu thresholding, followed by 7-pixel morphological closing, interior hole filling, retention of the largest connected component, and inward erosion by 5% of the equivalent radius, $R_{eq} = \sqrt{A/\pi}$, where A is the segmented filament-body area [32,33]. Within this specimen-specific internal ROI, pixels with gray level <60 on the 0–255 BSE scale were classified as apparent dark areas. A single global threshold was applied unchanged to all formulations to avoid formulation-specific re-optimization as SHF loading altered image texture. Gray level 60 was retained as a conservative operational cutoff that captured the darkest cavity-like regions while limiting classification of the brighter matrix/fiber texture in the overlay audit. Sensitivity calculations at gray levels 50, 55, 60, 65, and 70 preserved the same formulation ranking, although the absolute percentages changed. Because $n = 1$ full cross-section per formulation, the values are used only as descriptive

comparisons of the imaged sections. Full calculations, overlays, sensitivity results, and the native-resolution script are provided in Supplementary Section S2.

Image processing and apparent-dark-area calculations were implemented using a custom Python script developed for this study (`sem_apparent_dark_area_analysis_v3.py`, version 3). The package-version metadata from the original execution environment was not retained. For final reproducibility verification, the unchanged script was re-executed using Python 3.13.5 (Python Software Foundation, Wilmington, DE, USA), with NumPy 2.3.5, pandas 2.2.3, Pillow 12.2.0, SciPy 1.17.0, scikit-image 0.26.0, OpenCV-Python 4.13.0.92, and Matplotlib 3.10.8. The documented environment reproduced the reported apparent-dark-area values. The complete script and reproducibility information are provided in Supplementary Section S2 and Supplementary S1.

The fixed threshold was applied without formulation-specific adjustment and is verified against the threshold-overlay and mask-audit files in Supplementary Section S2. No inferential statistics or claim of bulk-representative porosity is made.

2.7. In-Line Diameter Monitoring and Metrics

Continuous in-line diameter logging began with F-15-18-01 and was available for 14 filament-making trials. Each spool-associated file was assigned in its entirety to a prospectively designated beginning, middle, or end production phase when supported by explicit records. Multiple files within the same phase were combined in collection order, incomplete phase coverage was retained without reconstructing missing phases, and whole-trial metrics pooled all available files for the corresponding trial.

Raw Filalogger readings were retained in their recorded order and processed using a centered five-point moving average. For the first and final two readings, the average was calculated from the available neighboring points; no padding, reordering, interpolation across missing segments, or manual removal of process-relevant excursions was performed. The processed series was used for plotting and metric calculation. Full equations and calculation details are provided in Supplementary Section S3.

Mean diameter, sample standard deviation, fractions within and outside 1.70–1.80 mm, the fraction above 1.89 mm, signed worst excursion, Cp, and Cpk were calculated from the processed series. Cp and Cpk used LSL = 1.70 mm and USL = 1.80 mm and were interpreted as capability-style diagnostics rather than formal production-capability measures because the developmental records were serial, overlapping, and potentially nonstationary.

3. Results and Discussion

3.1. Process-Chain Framework

The essential outcome inherited from the compounding stage was that compounded pellets entered filament-making as feed-quality carriers rather than neutral intermediates. Their size distribution, surface texture, fines content, abnormally shaped fragments, and parent-batch history could affect hopper discharge and screw replenishment before melting began. Filament manufacture then determined whether those pellets formed a sufficiently continuous and compacted material stream for controlled die flow, cooling, drawdown, measurement, winding, and printing.

The Results and Discussion are therefore organized according to the process chain in Figure 2. Direct evidence includes feed-throat blockage, backflow, strand fracture, hopper bridging, surface defects, diameter traces, cross-sectional morphology, and downstream printing. Intervention-linked evidence comprises responses to changes in screw architecture, screw speed, thermal profile, hopper state, material population, cooling path, or take-up geometry. Interpretations concerning screw filling, pressure development, air carry-forward, residence behavior, and melt continuity are identified as supported mecha-

nisms. Table 3 complements Figure 2 by grouping the 33 filament-making trials according to the process questions they address and summarizing the principal evidence, observed response, process conclusion, and evidential basis. Supplementary Tables S6.1A–S6.1D preserve the complete trial-level configuration and outcome record, while Supplementary Section S7 preserves the complete in-line-diameter evidence.

Feed-controlled extrusion framework for SHF/PLA filament-making

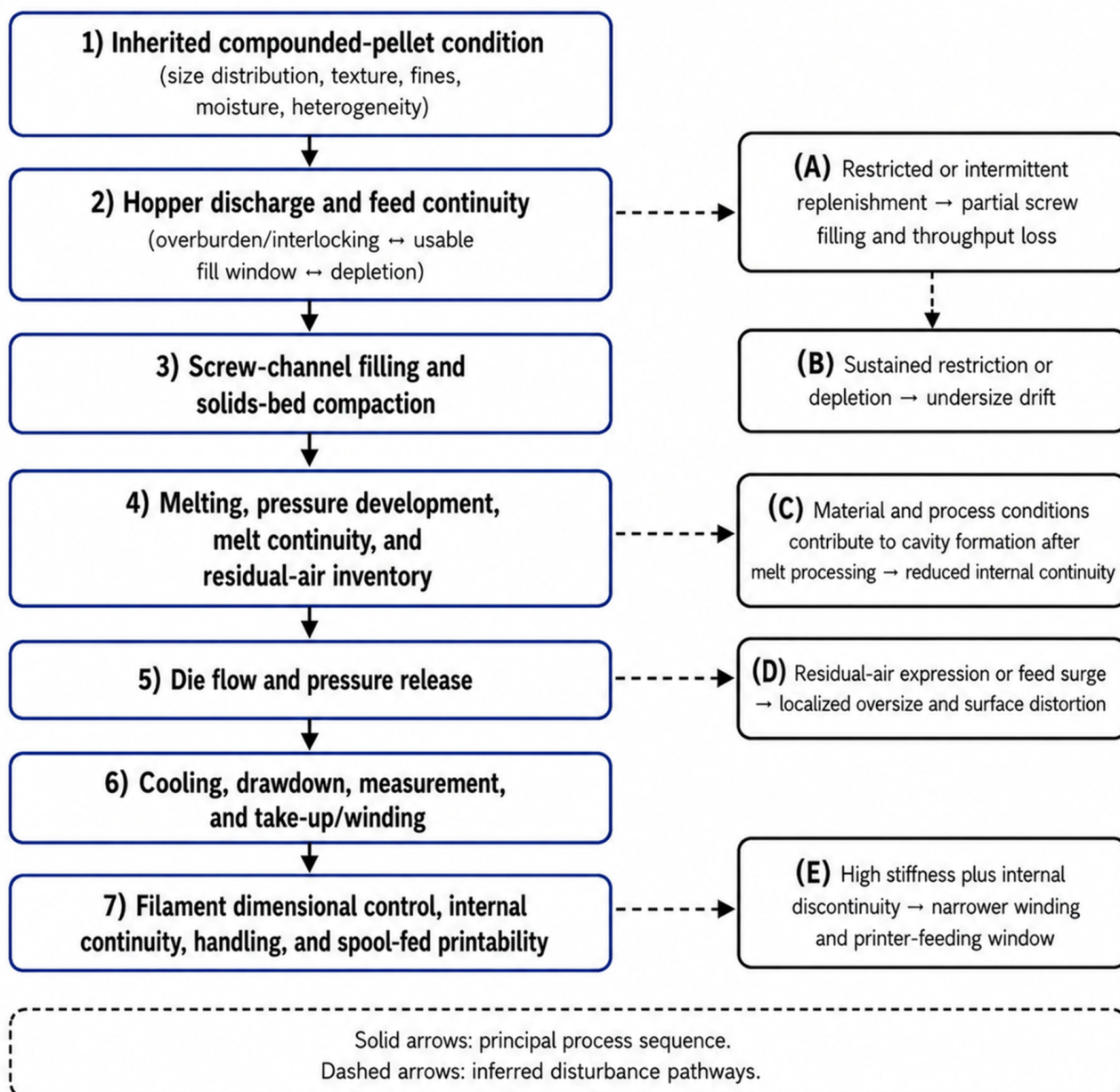


Figure 2. Study-specific process-chain model for converting compounded SHF/PLA pellets into spool-fed filament. Solid arrows show the principal process sequence; dashed arrows show causal disturbance pathways inferred from trial observations, in-line diameter records, cross-sectional morphology, and downstream printing.

Table 3. Mechanism-based summary of the filament-making trial groups and principal process conclusions.

Process Question	Principal Evidence	Observed Response	Process Conclusion	Evidential Basis
Could the compounding-stage screw architecture be transferred directly to filament-making?	Early 10SHF screw-comparison trials (F-10-01 to F-10-05) and the 25SHF screen trial (F-25-01).	Double-mixing-zone extrusion produced surging, backflow, feed-throat blockage, strand disruption, and diameter excursions; the no-mixing-zone screw also produced backflow and clumping, while the screen accumulated SHF-rich material and collapsed throughput.	Filament-making required the single-mixing-zone screw: a longer uninterrupted conveying path for solids-bed consolidation followed by limited final mixing. Melt filtration was unsuitable for the SHF-loaded filament route.	Direct intervention evidence supported by single-screw conveying and melt-flow theory.
Where was the practical SHF-loading boundary?	Converged 30SHF production trials compared with the 35SHF stress-test sequence.	The 30SHF entered dimensionally usable, spool-fed operating windows. The 35SHF route showed intermittent feeding, tearing, internal discontinuity, take-up fracture, and repeated breakage during spool unwinding, although loose-coil nozzle deposition remained possible.	The 30–35 wt.% transition was a platform-specific spool-fed handling boundary rather than an intrinsic SHF/PLA deposition limit.	Direct operational evidence and downstream print validation.
Was temperature adjustment alone sufficient to stabilize filament-making?	Early-to-converged 30SHF thermal sequence, the pivotal F-30-08 intervention, and the 0SHF transfer comparison (F-0-01/F-0-02).	Late-zone heating sometimes improved surface appearance temporarily but did not correct disturbed feeding. Restoring feed continuity before adopting 160/170/180/195 °C stabilized SHF-loaded filament, whereas neat PLA required 180/185/200/205 °C.	Thermal profile and feed state acted jointly, and the required filament-making profile was formulation-dependent.	Repeated intervention evidence interpreted through solids conveying, melting, viscosity, and pressure-development theory.
How did hopper inventory and replenishment affect filament dimensions?	Phase-resolved 0SHF, 20SHF, and 30SHF replenishment and depletion events.	Large additions and low-inventory depletion destabilized screw replenishment and generated oversize, undersize, or mixed-direction diameter responses depending on pellet morphology and formulation.	A usable hopper operating interval was required. The platform created the opportunity for discharge disturbance, while feedstock morphology governed its downstream expression.	Event-linked diameter analysis supported by the biomass-flow and partial-fill extrusion literature.

Table 3. *Cont.*

Process Question	Principal Evidence	Observed Response	Process Conclusion	Evidential Basis
Could feedstock history override converged machine settings?	Reprocessed, fines-rich, large/flattened-pellet, and later-batch 30SHF phases.	Controlled periods deteriorated after feed-material transitions even when the nominal screw, die, speed, and barrel settings were unchanged.	Inherited pellet condition and feed-material state could override nominal settings by altering hopper discharge, screw charging, air carry-forward, and melt delivery.	Within-trial contrasts linked to documented feedstock lineage and operating events.
Did the converged route produce operationally useful filament?	Converged 0SHF, 10SHF, 20SHF, and 30SHF runs; 14 logged trials; cross-sectional morphology; and production of 720 printed mechanical-test specimens over approximately 936 h.	Sustained downstream printing was achieved across all four retained formulations. The strongest 10SHF benchmark was tightly controlled, while 30SHF remained printable within a narrower structural and handling window.	Operational feasibility was demonstrated through 30 wt.% SHF without claiming statistical process control or industrial production capability.	Quantitative diameter evidence, morphology, and sustained downstream validation.

Note. Complete chronological trial settings, feedstock lineage, outcomes, and manuscript linkages are provided in Supplementary Tables S6.1A–S6.1D.

3.2. Screw Architecture and Line Stability

The principal outcome of the initial filament-making experiments was that screw architecture had to be matched to the function of the processing stage rather than transferred directly from compounding. The double-mixing-zone screw had been selected upstream to promote distributive and dispersive incorporation of SHF into PLA. During filament-making, however, the material entering the extruder was already compounded, and the dominant requirements shifted to repeatable pellet intake, progressive screw-channel filling, solids-bed consolidation, melting, and sufficiently uniform melt delivery for dimensional control. These functions are coupled in single-screw extrusion: solids throughput depends on the particle stream supplied through the hopper and feed opening, while downstream melt delivery reflects the interaction of drag flow, pressure flow, material rheology, and flow resistance [34,35].

Within the present Brabender extruder configuration, the double-mixing-zone screw produced unstable output when used for 10SHF filament-making. The two mixing sections reduced the length of uninterrupted helical conveying relative to the converged single-mixing-zone configuration, and the observed surging, twisting, and diameter excursions above 2 mm in the extruded strands were consistent with insufficiently stable forward conveying and irregular pressure generation. Lowering the first extruder heating zone temperature to apply greater push on the melt downstream, thereby overcoming pressure loss due to two dispersive mixing elements, did not suppress this behavior. Instead, the associated increase in local melt-flow resistance coincided with upstream melt reflux, encapsulation of incoming pellets, formation of partially molten pellet-rich agglomerates that caused feed-throat blockage, and, in one case, shear-pin failure.

The linked upstream and downstream defects further support this interpretation. Feed-throat blockage disrupted continuous solids replenishment, which was followed by air-discharge events and a local strand fracture at the die exit (Figure 3). Partial screw filling is known to alter melting, residence behavior, pressure development, and output in single-screw extrusion, including extrusion of wood–polymer composites [36]. In the present system, interrupted replenishment was therefore consistent with incomplete consolidation

of the incoming pellet bed and retention of air-bearing regions that were subsequently transported downstream. Expansion of that air during pressure release at the die provides a plausible explanation for the observed void growth and strand fracture.

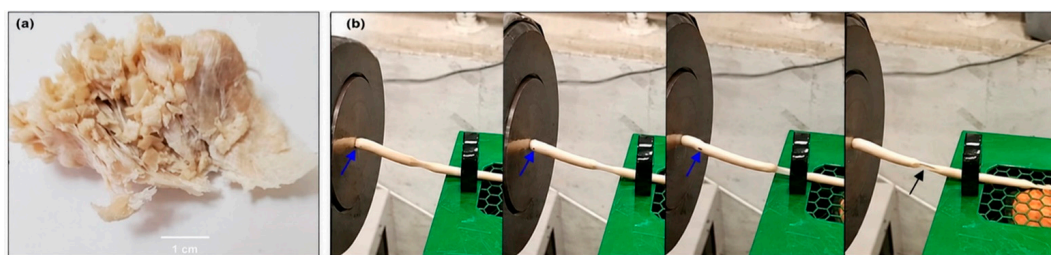


Figure 3. Feed-throat blockage and downstream extrusion instability during an early filament-making trial with the non-converged double-mixing-zone screw: (a) partially molten pellet-rich feed-throat blockage and (b) extrudate air-void evolution into fracture after die exit. Blue arrows indicate the evolving air void, meanwhile the **Black arrow indicates a fully developed split in the extrudate as the voids get drawn and keep increasing in size.**

The screw comparison showed that the solution was neither maximum mixing intensity nor complete removal of mixing. The same 10SHF feedstock became filament-processable when the single-mixing-zone screw was introduced, whereas a no-mixing-zone screw again produced melt backflow and pellet clumping. The converged architecture, therefore, provided a more suitable balance for this platform, i.e., sufficient uninterrupted conveying and solids-bed consolidation followed by limited final mixing near the die. The double-mixing-zone failures, single-mixing-zone success, and no-mixing-zone failure are represented by trials F-10-01–F-10-05, while the subsequent uneventful transfer to 15SHF provided an additional confirmation of the converged architecture. Detailed trial conditions and outcomes remain available in Supplementary Tables S6.1A–S6.1D rather than being reconstructed chronologically here.

A third baseline constraint concerned melt filtration. A 150 μm filter screen used with a twice-compounded, 25SHF pellet population accumulated SHF-rich material, after which throughput declined, melt reflux advanced toward the feed throat, and incoming pellets became encapsulated by the refluxed material. Woven screens inherently introduce an additional pressure drop into polymer-melt flow, and retained material can progressively increase the resistance of the filtration system [37,38]. The observed throughput collapse was therefore consistent with the combined effect of screen resistance and progressive SHF-rich deposition exceeding the available pressure-delivery margin of the screw–die configuration.

These baseline trials established the controls carried forward, i.e., stage-specific screw selection, line-aware interpretation of diameter, and filter screen-free filament extrusion.

3.3. The 35SHF Spool-Fed Processability Ceiling

The 35SHF trials tested whether the highest compounding-stage stress-test formulation could be converted into reliably spool-fed filament. Rough, interlocking pellets caused intermittent screw charging, unstable output, diameter variation, and material-deficient strand regions. Even when take-up was achieved, the filament contained hollow, porous, or torn sections and lacked sufficient handling toughness. No combination of temperature, screw speed, die geometry, or drawdown simultaneously restored feed continuity, dimensional control, and spool-fed reliability; 30SHF was therefore adopted as the highest reliably spool-fed formulation on the present platform.

This boundary is platform-specific rather than an intrinsic SHF/PLA loading limit. Material contributions included interlocking pellet morphology, fines, reduced PLA-phase

continuity, and increased filament stiffness and brittleness, while platform contributions included gravity feeding, the hopper and feed-throat geometry, the absence of metered feeding and active venting, spool curvature, and printer-feed architecture. The 35SHF filament could still be deposited through the printer nozzle when loose coils were supplied without spool-unwinding resistance, and no nozzle clogging or printhead-internal filament fracture was observed. It nevertheless fractured repeatedly during direct spool unwinding. Thus, 35SHF exceeded the reliable spool-fed handling boundary rather than the basic deposition boundary; 30SHF could be wound, unwound, and supplied directly from the spool. Metered feeding, vented extrusion, positively driven spool assistance, or a larger winding diameter could shift this boundary.

3.4. Feed–Thermal Coupling

The principal thermal result was that the appropriate filament-making temperature profile depended on the feed state, material rheology, and functional requirement of the extrusion stage. The filament-making stage required continuous pellet intake, consolidation of the pellets into a coherent solids bed, progressive melting, formation of a continuous melt column, and stable delivery to the die. These requirements differ from those of compounding, where lower melt viscosity through the mixing regions supports fiber wetting and distribution. The filament-making temperature profile must consequently be interpreted as part of a coupled feed–screw–die system rather than as an independently optimized set of barrel temperatures.

Classical single-screw extrusion theory treats solids conveying, melting, and melt conveying as coupled stages whose performance depends on material properties, screw geometry, pressure development, and downstream resistance [29]. Experimental solid-conveying studies further show that throughput can become limited by the granule stream supplied through the hopper opening and can depend on pellet size and shape [34]. Within the melt-conveying region, output reflects the combined effects of drag flow, pressure flow, channel geometry, melt rheology, and thermal conditions [35]. Experimental and modeling studies of partially filled extrusion show that feed rate and screw fill can alter residence behavior, pressure development, melt temperature, melting progression, and output [30,36]. This framework explains why surface roughness, post-die extrusion swelling, hollow filament cross-sections, surface tearing, and diameter excursions observed during the present study could not be interpreted as direct temperature responses without first considering pellet replenishment and screw charging.

3.4.1. Limits of Late-Zone Heating

The early and intermediate SHF-loaded filament-making experiments tested final-zone temperatures between approximately 185 and 205 °C. Raising the temperatures of heating zones 3 or 4 occasionally produced a temporarily smoother strand, but the improvement was neither persistent nor transferable between pellet populations. At the higher temperature settings, smooth extrusion was repeatedly followed by extrudate swelling post-extrusion, hollow cross-sections, surface tearing, or strand breakage during the drawdown of material-deficient regions during the filament take-up. In other cases, surface quality improved only after screw-speed adjustment, restoration of hindered pellet feeding, or replacement of the material population in the hopper. These observations show that apparent responses to temperature were frequently responses to simultaneous changes in screw filling and feed continuity.

This observation is important because increasing temperature lowers PLA-rich melt viscosity and can facilitate flow through the metering and die regions. That response can temporarily conceal an irregularly charged screw by allowing partially consolidated

material to pass more readily through the die. It does not, however, correct the upstream cause of the disturbance. If intermittent feeding has left air-bearing or compositionally discontinuous regions in the screw channel, lower viscosity may permit those regions to remain mobile and reach the die rather than being compressed into a more continuous melt. Pressure release at the die can then expand the retained air, producing surface roughness, swelling, internal porosities, and mechanically weak regions susceptible to rupture during drawdown in the extrudate.

Filament-manufacturing studies show that diameter and roundness are jointly influenced by die or nozzle dimensions, screw or extrusion speed, barrel and cooling conditions, material rheology, and the distance available for cooling and downstream deformation, rather than by temperature alone [11–13]. More generally, extrusion-deposition research confirms that temperature, extrusion force, rheology, and die swell act together in determining extrudate diameter [39]. The temporary improvements observed after late-zone heating can therefore not be considered as evidence that insufficient melt temperature was the dominant cause of the SHF-loaded filament defects. Across the present experiments, pellet-batch transitions, abnormal fragments, hopper replenishment, agitation effectiveness, overburden, and low-fill conditions repeatedly modified or overrode the apparent temperature response. Detailed trial-level evidence remains available in Supplementary Section S6, but the general conclusion is that late-zone temperature escalation could improve local flow without establishing a stable feed and compaction state.

Because these experiments were sequential process-development trials rather than factorial experiments with independently controlled feed state, temperature, and pellet population, the individual contribution of each temperature change cannot be estimated statistically. The evidence instead supports an intervention-linked mechanistic conclusion that temperature adjustment became effective only after sufficiently continuous pellet feeding and screw charging had been restored. This limitation prevents temperature from being presented as an isolated causal variable but strengthens the broader conclusion that thermal and feeding conditions acted jointly.

3.4.2. Basis of the Converged SHF Profile

The decisive process transition occurred when feed continuity was restored before the temperature profile was reduced. During a pivotal 30SHF filament-making experimental intervention, a temporary increase in screw speed from 5 to 15 rev/min re-established autonomous pellet intake, improved screw charging, and displaced air-laden and thermally stressed material generated during the preceding disturbed-feed period. The barrel profile was then reduced from the earlier high-temperature condition to 160 °C/170 °C/180 °C/195 °C, after which the extrudate surface became smoother and lighter in color and entered the narrowest 30SHF diameter-control window achieved to that point. Screw speed was subsequently returned to 10 rev/min for take-up control, and this coupled condition became the converged condition for SHF-loaded filament production (F-30-08; Table 2).

The converged profile can be interpreted through the mapped function of the single-mixing-zone screw. Heating zones 1 and 2 covered the solids-conveying and progressively compressing region, where premature softening could interfere with the frictional transport and consolidation of the pellet bed. Heating zone 3 supported progressive melting and metered transport, whereas heating zone 4 covered the final mixing element and short die-entry region. Maintaining comparatively low temperatures through the first three zones limited premature loss of the pellet-bed structure and preserved the flow resistance needed for progressive consolidation. The 195 °C final-zone setting then provided sufficient melting and die-entry conditioning without the excessive softening and air-related instability repeatedly observed when the final zone was operated at 205 °C.

This interpretation does not mean that a more viscous melt directly removes gas or that a lower final-zone temperature eliminates pressure release at the die. A cooler and more viscous material may, in isolation, increase die resistance and intensify the expansion of any gas that reaches the die exit. The converged profile acted earlier in the process chain by supporting more continuous screw filling, consolidation, and melt-column formation, which reduced the amount of free or poorly constrained air-bearing material delivered to the die. The smaller upstream air inventory then outweighed the potentially greater expansion of any residual air during pressure release on extrusion from the die. The governing outcome was therefore determined by the balance between upstream air carry-forward and downstream die expansion, not by die-exit viscosity alone.

The low-temperature profile was consequently necessary but not sufficient. Once established, it did not prevent rough, hollow, or dimensionally unstable filament when hopper bridging, excessive hopper loading, oversized or irregular pellets, fines, or low-fill conditions interrupted screw replenishment. This finding is consistent with partial-fill extrusion theory and with the present hopper-state results that the nominal barrel settings cannot produce stable pressure and output when the mass flow entering the screw is itself unstable [30,34–36].

3.4.3. The OSHF-Specific Thermal Profile

The neat-PLA control provides an important counterexample to interpret the converged SHF-loaded profile as universally superior. When the 160 °C/170 °C/180 °C/195 °C profile was transferred to OSHF, it did not provide sufficiently stable melt delivery and diameter control. The OSHF filament required the hotter 180 °C/185 °C/200 °C/205 °C profile reported in Table 2. This difference is consistent with the specific feed and rheological conditions of neat PLA. The comparatively regular and dense PLA pellets did not present the same fiber-associated interlocking, heterogeneous packing, reduced polymer-phase continuity, or air-carry-forward burden as the SHF-loaded pellets. The dominant requirement, therefore, shifted from suppressing air-bearing feed discontinuities to achieving complete melting and stable melt throughput for drawdown.

The OSHF comparison also separates the function of the two extrusion stages. The compounding profile remained transferable across formulations because its principal output was a pelletizable strand where a high degree of dimensional control was not required. Alternatively, filament-making was more formulation-sensitive because it produced the final continuously drawn feedstock, and fluctuations in melt delivery were expressed directly as filament-diameter variation. The contrast between the initial and converged OSHF conditions (F-0-01 and F-0-02), therefore, reinforces the central process conclusion: the appropriate thermal profile depended on the material-specific balance among feeding, compaction, melting, and melt transport.

3.5. Die Geometry for Filament-Making

The 3.85 mm/5.75 mm die was selected for filament-making because the downstream objective was continuous strand formation and controlled drawdown rather than pelletizable-strand sizing. Relative to the smaller compounding die, the larger orifice was consistent with lower flow resistance and less severe downstream expression of residual discontinuities. Independent natural-fiber filament research confirms that die diameter, extrusion speed, and cooling distance influence final filament dimensions [11].

The trial record establishes more directly that die geometry could moderate the extruded strand behavior but could not correct deficient screw replenishment. Post-extrusion swelling, structural discontinuity, and diameter excursions persisted when screw assignment or feed continuity remained unstable, whereas the same die supported controlled

filament extrusion after conveying and feed continuity improved. Die geometry therefore functioned as a downstream stabilizer within the process chain, not as an independent remedy for upstream feeding instability. Detailed die and line-development observations are provided in Supplementary Section S6.

This observation ultimately led to hopper fill level and replenishment behavior as the next process-control variable. The following section, therefore, treats hopper state not as an operator convenience, but as a governing condition that determines whether the selected die receives a compact, continuous melt stream or an air-interrupted feed stream.

3.6. Hopper-State Effects

Stable single-screw extrusion requires the pellet stream entering through the feed opening to remain compatible with the solids-transport capacity of the rotating screw. If hopper discharge becomes intermittent or insufficient, the screw can depart transiently from the intended fully filled condition, altering solids-bed formation, melting progression, residence behavior, pressure development, melt temperature, and output [29,30,36].

The experimental records indicated a usable hopper operating interval bounded by two distinct disturbance regimes: an upper-inventory/replenishment regime and a low-inventory/depletion regime. These regimes originated from different hopper states but converged on a common downstream condition, i.e., irregular or insufficient screw replenishment. The resulting filament response was nevertheless formulation-dependent because the SHF-loaded pellets differed from the neat-PLA pellets in surface texture, geometric regularity, fines burden, bulk packing, and tendency to interlock. This operating interval was inferred from repeated event-linked changes in extrusion behavior and in-line diameter.

3.6.1. High-Inventory/Replenishment Disturbance

Large pellet additions repeatedly disturbed otherwise controlled extrusion, but these events should not be attributed to overburden alone. Replenishment simultaneously changed inventory, packing arrangement, particle contacts, agitator engagement, and, when a new pellet population was added, the proportions of fines and abnormal fragments approaching the feed opening.

Biomass-flow studies similarly show that hopper discharge depends jointly on particle dimensions and size distribution, density, packing, friction, hopper and outlet geometry, inclination, and surcharge [40–42]. The observed response is therefore interpreted as a coupled change in packing and discharge conditions rather than as a simple hydrostatic-pressure effect.

For the SHF-loaded feedstocks, the upper-inventory disturbance was expressed through mixed directional filament instability. Intermittent restriction and release at the hopper outlet could generate nonuniform screw replenishment, producing short-lived feed pulses interspersed with partially filled intervals. The downstream air-carry-forward and pressure-release mechanisms established in Sections 3.4 and 3.5 could then appear as localized extrudate surface distortion, sustained swelling on die exit, or oversize-diameter events. If restricted replenishment persisted, reduced average material delivery became dominant, and filament diameter shifted toward the undersized side. Thus, oversize excursions and subsequent undersize drift were not necessarily competing explanations; they could represent different temporal stages of the same irregular-feed event.

The neat-PLA control provided an important material comparison. Large pellet additions also disturbed extrusion on the same Brabender hopper and feed-throat platform, demonstrating that the upper-inventory effect was not unique to SHF. However, the comparatively regular, dense, and smooth 0SHF pellets were less susceptible to the fiber-associated interlocking, broad morphological heterogeneity, and fines-mediated packing effects of the

SHF-loaded pellets. The dominant 0SHF response was consequently reduced material delivery and undersize-biased filament rather than the pronounced mixed oversize/undersize instability observed in several SHF-loaded records. The hopper platform created the opportunity for discharge disturbance, whereas pellet morphology influenced the form and severity of its downstream expression.

3.6.2. Low-Inventory/Depletion Disturbance

Instability also developed before the hopper became empty. At low inventory, reduced coverage above the feed opening and altered interaction with the fixed agitator allowed individual oversized fragments, bridges, fines-rich regions, or local voids to exert greater control over screw replenishment. Early depletion could retain mixed-direction excursions, but sustained mass-flow loss reduced die throughput and shifted the filament toward undersizing. This common progression is consistent with the effects of partial screw filling on residence behavior, melting, pressure development, melt temperature, and output [30,36]. The end-of-run deterioration observed across multiple formulation records can therefore be interpreted as a common screw-replenishment failure rather than as a separate temperature, die, or formulation mechanism in every trial.

3.6.3. Influence of Pellet Morphology on Solids Conveying

Studies comparing regular virgin pellets, powders, and irregular regrind show that particle shape, size distribution, bulk density, and compressibility affect screw filling, throughput, and processing-window width [43,44]. The SHF-loaded pellets introduced additional surface roughness, fines, elongated flattened fragments, structural heterogeneity, and mechanical interlocking. Increasing SHF loading therefore increased the material-side sensitivity of the process to the fixed hopper and feed-throat design.

3.6.4. Material–Platform Contributions

Hopper sensitivity arose from a material–platform interaction. Material contributors included pellet geometry, surface texture, fines, abnormal fragments, bulk density, compressibility, and particle friction. Platform contributors included hopper and outlet geometry, feed-throat dimensions, agitator coverage, direct gravity feeding, and the absence of metered delivery or continuous level feedback. Without an independent feeder, disturbances at the hopper outlet were transmitted directly into screw replenishment [43,44].

The observed operating interval is therefore not an intrinsic SHF/PLA material constant. A mass-flow hopper, altered outlet-to-pellet size ratio, controlled agitation, continuous level sensing, or gravimetric or volumetric feeding could shift both the upper-inventory and depletion boundaries. Tighter pellet-size control and removal of fines or elongated fragments could likewise reduce instability without changing the extruder.

Stable extrusion on the present platform required avoiding both disruptive replenishment and low-inventory depletion while maintaining mass flow compatible with the screw's solids-conveying capacity. Section 3.8 presents the corresponding phase-resolved evidence.

3.7. Backward Transfer and Filament Cross-Sectional Morphology

The converged SHF-loaded filament-making conditions (160 °C/170 °C/180 °C/195 °C barrel profile, single-mixing-zone screw, 10 rpm screw speed, and 3.85 mm diameter-orifice die) were established through the 30SHF process-development sequence and then used for subsequent 30SHF filament production (F-30-09, F-30-10, F-30-11), while being transferred backward to 20SHF (F-20-01, F-20-02) and 10SHF (F-10-06, F-10-07). The 30SHF trials confirmed that the converged condition could produce dimensionally usable and spool-fed printable filament at the highest SHF loading, but with a narrower diameter-

control and feed-stability window than the lower-SHF formulations. The 20SHF and 10SHF filament-making trials produced acceptable filament and demonstrated broader transferable diameter control, although the in-line records in Section 3.8 show that individual phases still remained sensitive to hopper state, extruder screw-speed drift, pellet size, and drawdown effects. The 0SHF transfer was more staged, as described in Section 3.4.1, and required a different thermal processing profile while keeping the same general filament-making architecture used for the SHF-loaded formulations.

The backward-transfer sequence showed that the operating architecture established at 30SHF could be transferred to the less constrained 20SHF and 10SHF formulations, while neat PLA required a separate thermal profile. Cross-sectional SEM images of the converged-setting filaments are shown in Figure 4a–l: 0SHF in panels (a–c), 10SHF in (d–f), 20SHF in (g–i), and 30SHF in (j–l), each at 50×, 80×, and 180× magnification. Native-resolution 50× fixed-threshold overlays are shown in Figure 4m–p. At the common gray-level threshold of 60, the single-cross-section apparent dark-area fractions were 0.15% for 0SHF, 1.27% for 10SHF, 4.21% for 20SHF, and 12.51% for 30SHF (Table 4). Recalculation across thresholds of 50–70 preserved the order 0SHF < 10SHF < 20SHF < 30SHF, confirming that the principal comparison was not created by the selected cutoff, while also demonstrating that the absolute percentages were threshold-dependent.

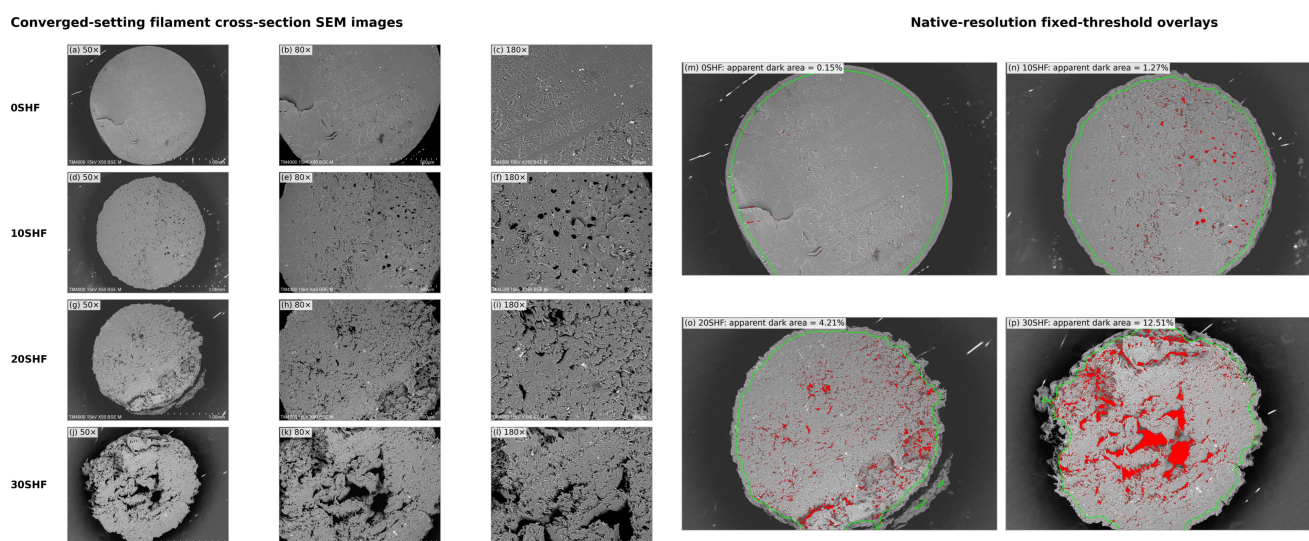


Figure 4. SEM cross-sectional morphology and specimen-segmented apparent dark-area overlays for converged-setting SHF/PLA filaments. Panels (a–c), (d–f), (g–i), and (j–l) show the 0SHF, 10SHF, 20SHF, and 30SHF cross-sections, respectively, at 50×, 80×, and 180× magnification. Panels (m–p) show the corresponding native-resolution 50× overlays. Red regions denote pixels with a gray level <60 inside the analysis ROI, and the green boundary denotes the specimen-segmented internal ROI generated by 5% inward erosion of the segmented filament body. The displayed percentages are descriptive single-cross-section apparent dark-area fractions, not statistically representative bulk porosity values.

The 0SHF and 10SHF sections were comparatively continuous, 20SHF showed more distributed dark features and internal heterogeneity, and 30SHF contained large, irregular, partly connected regions. The principal morphological result is therefore the pronounced 30SHF contrast rather than a statistically quantified population-level porosity progression. Increasing SHF reduced the continuous PLA phase and increased the likelihood of fiber agglomeration, incomplete wetting, and restricted matrix relaxation; the process record also identifies 30SHF as the formulation most sensitive to pellet interlocking, fines, interrupted charging, and air carry-forward [5,21,22,45].

Table 4. Descriptive single-cross-section apparent dark-area fractions from native-resolution 50× BSE filament cross-sections.

Formulation	Apparent Dark-Area Fraction at Gray <60	Conservative Interpretation
0SHF	0.15%	Very low apparent dark-area fraction in the analyzed cross-section.
10SHF	1.27%	Low apparent dark-area fraction; predominantly continuous cross-section.
20SHF	4.21%	Greater distributed internal dark features and cross-sectional heterogeneity.
30SHF	12.51%	Substantially larger and partly connected dark internal features; their origin is not uniquely attributable from the single section.

The BSE cross-sections cannot establish the origin of every classified dark region. The measured area may include true internal cavities, interfacial separation, fiber pull-out, blade-sectioning damage, topographic shadowing, or combinations of these effects. The values are therefore termed apparent dark-area fractions and are interpreted as descriptive indicators of internal discontinuity.

Large and connected internal discontinuities reduce the continuous effective cross-section and provide locations at which bending and feed stresses may concentrate. The 30SHF morphology therefore offers a plausible structural explanation for its narrower winding, unwinding, and printer-feeding window. However, one full cross-section per formulation is analyzed; no inferential statistical comparison is made, and the percentages apply only to the imaged sections. Statistically representative morphology would require multiple independently sampled cross-sections from separated filament locations.

The next question was whether this residual feed-state sensitivity was also reflected quantitatively in the filament in-line diameter records through changes in routine stability, tolerance-band retention, high-diameter excursion risk, and phase-resolved capability-style metrics.

3.8. In-Line Diameter Validation and Feed-State Effects

Continuous in-line logging was available for 14 trials. Files were assigned to beginning, middle, end, partial-phase, or whole-trial coverage according to Sections 2.5 and 2.7; incomplete records were retained as recorded rather than reconstructed. Phase-resolved metrics are reported in Table 5, and whole-trial metrics are provided in Supplementary Table S7.1. The F-0-01 entry appears in both views because only one recoverable beginning spool is available and is not treated as an independent duplicate.

Filament diameter was interpreted as the output of the complete extrusion line rather than as a direct measurement of one isolated variable. Published filament-manufacturing studies show that diameter and roundness respond jointly to screw speed, extruder output, barrel and die temperature, die geometry, cooling conditions, cooling distance, drawdown, and take-up [11–13]. Similarly, diameter excursions in the present work were interpreted as potential indicators of disturbances in hopper discharge, screw filling, melting, die flow, cooling, drawdown, measurement alignment, or winding. Documented process events and the directional form of the diameter response were therefore interpreted together rather than assigning each excursion automatically to a single mechanism.

Table 5. Phase-resolved filament-diameter metrics for trials with defensible beginning (B), middle (M), and/or end (E) assignment.

Trial Code	Phase	<i>n</i>	Mean (mm)	SD (mm)	% Within 1.70–1.80	% > 1.80 mm	% < 1.70 mm	% > 1.89 mm	Signed Value at $ \Delta $ max	Cp	Cpk
F-30-07	B	5784	1.7491	0.0326	89.5228	4.4779	5.9993	0.1729	+0.2900	0.51	0.50
F-30-07	M	12,401	1.7400	0.0392	80.4774	6.1527	13.3699	0.0403	+0.2160	0.43	0.34
F-30-07	E	16,273	1.7106	0.0407	57.7521	2.0095	40.2384	0.0307	+0.2500	0.41	0.09
F-30-08	M	17,953	1.7534	0.0173	98.1452	0.4902	1.3647	0.0000	−0.1120	0.96	0.90
F-30-08	E	4425	1.7607	0.1394	84.4520	7.9322	7.6158	6.5989	+1.8620	0.12	0.09
F-30-09	B	23,924	1.7305	0.0559	67.7144	6.8174	25.4681	0.6980	+0.4840	0.30	0.18
F-30-10	M	3234	1.7152	0.0216	76.9326	0.0000	23.0674	0.0000	−0.1060	0.77	0.23
F-30-10	E	11,046	1.7411	0.0725	69.2921	10.3838	20.3241	6.4820	+0.5275	0.23	0.19
F-30-11	B	13,120	1.7391	0.0293	89.6570	1.7530	8.5899	0.0000	+0.1240	0.57	0.44
F-30-11	M	4058	1.7746	0.0455	77.2794	22.0306	0.6900	2.3903	−0.2960	0.37	0.19
F-30-11	E	9563	1.7541	0.0491	72.0590	16.5848	11.3563	1.0771	+0.1920	0.34	0.31
F-20-01	B	9060	1.7570	0.0151	99.9448	0.0331	0.0221	0.0000	−0.0580	1.10	0.95
F-20-01	M	6335	1.7518	0.0806	88.9029	3.6622	7.4349	2.0837	+0.7740	0.21	0.20
F-20-01	E	10,897	1.7523	0.0198	96.6872	1.6427	1.6702	0.0000	−0.1300	0.84	0.80
F-10-06	B	8470	1.7406	0.0230	95.8560	0.9209	3.2231	0.0000	+0.1100	0.72	0.59
F-10-06	M	6852	1.7503	0.0196	99.1389	0.2335	0.6276	0.0000	−0.0960	0.85	0.85
F-10-06	E	14,537	1.7371	0.0246	93.4787	0.3921	6.1292	0.0275	+0.1800	0.68	0.50
F-20-02	B	15,747	1.7741	0.0391	73.2965	24.5507	2.1528	0.1270	+0.1600	0.43	0.22
F-20-02	M	11,561	1.7609	0.0241	95.2859	3.9270	0.7871	0.0000	+0.1100	0.69	0.54
F-20-02	E	6149	1.7525	0.0231	97.2353	1.3173	1.4474	0.0000	−0.0900	0.72	0.69
F-30-12	M	12,822	1.7204	0.0203	85.7433	0.0000	14.2567	0.0000	−0.1200	0.82	0.33
F-30-12	E	3533	1.8155	0.1484	26.7195	45.9949	27.2856	33.5409	+0.4360	0.11	−0.03
F-0-01	B	42,266	1.6938	0.1022	43.4557	10.7959	45.7484	1.3604	−0.4880	0.16	−0.02
F-0-02	B	24,526	1.7422	0.0362	88.0739	3.8041	8.1220	0.1998	+0.5620	0.46	0.39
F-0-02	M	30,162	1.7144	0.0731	64.1503	4.8936	30.9562	0.9416	−0.3200	0.23	0.07
F-0-02	E	19,143	1.7359	0.0458	77.1352	2.6537	20.2110	0.2560	+0.1740	0.36	0.26
F-30-13	B	11,960	1.7347	0.0199	96.8562	0.2926	2.8512	0.0000	+0.1000	0.84	0.58
F-30-13	M	13,462	1.7540	0.0363	92.7202	5.5490	1.7308	1.7159	+0.2880	0.46	0.42
F-30-13	E	19,183	1.7559	0.0423	81.7025	12.7196	5.5779	0.4900	+0.2280	0.39	0.35

Note. *n* = number of readings; SD = sample standard deviation; Cp = process capability index; Cpk = centered process capability index. Each reported phase could contain one or multiple spool-associated source files. Files were assigned in their entirety from explicit spool labels and contemporaneous trial records and were not retrospectively divided to create phases. Partial phase coverage was retained rather than forced into a complete B/M/E structure. Tolerance-band retention refers to 1.70–1.80 mm; % > 1.89 mm represents the high-risk printability threshold. Cp and Cpk were calculated using LSL = 1.70 mm and USL = 1.80 mm and are used only as descriptive summaries of spread and centering.

Cp and Cpk were retained as capability-style summaries rather than formal evidence of process capability. Conventional capability interpretation presumes a statistically stable process and a representative estimate of process variation [46]. Those conditions were not established here because the records contained serially ordered, overlapping moving-average observations and could include startup, intervention, replenishment, material-transition, depletion, and run-out periods. Cp and Cpk were therefore interpreted only alongside mean diameter, standard deviation, directional tolerance loss, percentage of readings above 1.89 mm, signed worst excursion, trace shape, and the documented process state.

The categories below describe phase-level directional signatures; the same trial could transition among controlled, undersize-dominated, oversize-dominated, and mixed-direction states.

3.8.1. Controlled Diameter Windows Were Achievable but Phase-Dependent

The strongest sustained dimensional-control evidence was obtained from the complete beginning–middle–end record of the converged 10SHF process. Its whole-trial mean diameter was 1.7411 mm and its standard deviation was 0.0236 mm, and 95.45% of readings remained within the 1.70–1.80 mm analysis band. Only 0.013% of readings exceeded 1.89 mm. The beginning, middle, and end phases retained 95.86%, 99.14%, and 93.48% of their readings within the analysis band, respectively, with phase Cpk values ranging from 0.50 to 0.85. These results establish that the developed system could maintain a comparatively narrow and well-centered diameter distribution across an extended production sequence rather than only during a short favorable interval.

Controlled windows also occurred in 20SHF and 30SHF, but their durability depended on screw-speed correction, hopper state, and feed-material condition. The loss and recovery

of those windows are analyzed by directional response in Sections 3.8.2–3.8.4. Because the trials are sequential rather than replicated formulation comparisons, the results do not establish a monotonic loading–capability relationship.

3.8.2. Undersize-Dominated States

An undersize-dominated response was defined by lower-side tolerance loss clearly exceeding upper-side loss, generally accompanied by downward displacement of the mean or Cpk toward the lower analysis limit. Such responses were associated with an unsuitable thermal profile, restricted hopper discharge, bridging, irregular screw replenishment, or low-inventory depletion.

The initial transfer of the SHF-loaded filament-making thermal profile to 0SHF produced the clearest undersize-dominated negative comparator. The resulting record had a mean diameter of 1.6938 mm, a standard deviation of 0.1022 mm, and only 43.46% of readings within the analysis band and 45.75% below 1.70 mm, compared with 10.80% above 1.80 mm. Its Cpk was negative because the mean itself lay below the lower analysis limit. The record was therefore broad but strongly undersize-shifted, quantitatively supporting the formulation-specific thermal requirement established in Section 3.4.3.

Adoption of the hotter 0SHF-specific profile improved the beginning phase to 88.07% within the analysis band. Following a large pellet quantity addition and rapid increase in hopper inventory, however, the middle phase decreased to 64.15% within the band, with 30.96% of readings below 1.70 mm compared with 4.89% above 1.80 mm. The end phase recovered partially to 77.14% within the band but remained undersize-dominated, with 20.21% below 1.70 mm and 2.65% above 1.80 mm. Because the upper-inventory disturbance occurred in neat PLA as well as in SHF-loaded feedstocks, this response demonstrated that hopper sensitivity was partly a feature of the gravity-fed Brabender hopper and feed-throat arrangement rather than exclusively a consequence of SHF incorporation.

Several 30SHF records showed lower-side deterioration associated with restricted or depleted feeding. In one record, the middle and end phases retained only 80.48% and 57.75% diameter values within the tolerance band, while their below-band fractions increased to 13.37% and 40.24%, respectively. The end phase was therefore a strong undersize-dominated state associated with late-run loss of effective screw replenishment. A separate beginning-only 30SHF record retained 67.71% within the band, with 25.47% below 1.70 mm compared with 6.82% above 1.80 mm, consistent with disruption by flattened strand fragments at the feed throat.

A further 30SHF record showed an undersize-only middle phase, with 23.07% of readings below 1.70 mm and no readings above 1.80 mm. Its later end phase developed substantial excursions on both sides and is therefore classified separately as mixed-direction instability in Section 3.8.4. Similarly, the middle-phase preceding entry of a fines-rich final 30SHF pellet population retained 85.74% within the band but showed 14.26% below 1.70 mm and no upper-side loss. That phase was undersize-dominated, whereas the subsequent fines-rich end phase became severely mixed-direction-unstable.

The pooled 10SHF small-batch record also showed an undersize-dominated response near run-out. Although no readings exceeded 1.89 mm, 15.18% fell below 1.70 mm, and the worst excursion was -0.470 mm as bridging developed. This result demonstrates why absence of severe oversize events cannot be used independently as evidence of control. A low high-diameter exceedance rate may reflect a genuinely stable process, but it may also occur when restricted output or strong filament drawdown shifts the distribution downward to suppress oversize events.

3.8.3. Oversize-Dominated States

An oversize-dominated response was defined by upper-side tolerance loss clearly exceeding lower-side loss, even if isolated lower-side excursions remained present. This signature was associated with screw-speed drift, abrupt pellet-population or hopper-state changes, intermittent air-rich delivery, and process adjustments that improved visible surface condition without restoring dimensional centering.

The clearest 20SHF oversize-dominated phase occurred at the beginning of a production record affected by screw-speed drift. Only 73.30% of readings remained within the analysis band, while 24.55% exceeded 1.80 mm and only 2.15% fell below 1.70 mm. The mean was 1.7741 mm and Cpk was 0.22. After screw speed returned to the converged extruder screw speed setting, the middle and end phases recovered to 95.29% and 97.24% within the band. This progression directly links restoration of the converged operating condition with recovery from an upper-side displaced state.

A 30SHF record involving large pellets and a second-batch addition showed a stronger phase-localized oversize response. The middle phase retained only 77.28% of readings within the band, with 22.03% above 1.80 mm and only 0.69% below 1.70 mm. Although one isolated undersize excursion produced the signed worst absolute deviation, the distribution itself was unequivocally oversize-dominated. The corresponding end phase developed substantial losses on both sides and is therefore considered in Section 3.8.4 rather than treated as the continuation of a purely oversize state.

A later 30SHF record demonstrated progressive oversize-side deterioration despite thermal intervention to improve extrudate surface quality. Its beginning phase retained 96.86% of readings within the band. The middle and end phases then decreased to 92.72% and 81.70%, while the fractions above 1.80 mm increased from 0.29% to 5.55% and 12.72%, respectively. The corresponding below-band fractions were 2.85%, 1.73%, and 5.58%, confirming that upper-side loss remained dominant during the deterioration. Cpk decreased from 0.58 to 0.42 and then to 0.35. The final heating zone and die-temperature increases improved the 30SHF filament's visible surface sufficiently to enable take-up during this later record, but the diameter metrics continued to deteriorate. The intervention was therefore compensatory rather than curative, i.e., it improved local strand appearance without restoring the underlying dimensional state. Surface smoothness could not consequently be treated as evidence that extrusion had stabilized. This result separates visible surface improvement from quantitative diameter control and demonstrates the limitation of late heating zone temperature escalation when the feed population and hopper state continue to evolve.

3.8.4. Mixed-Direction Instability

Mixed-direction instability was defined by substantial losses on both sides of the analysis band, generally accompanied by elevated standard deviation, poor Cp/Cpk, or severe localized excursions. The losses did not need to be numerically equal. A mixed state could remain undersize- or oversize-predominant while still differing fundamentally from a nearly uniform one-sided displacement.

A large increase in the extruder hopper inventory within an otherwise controlled 20SHF record illustrates this response. The beginning and end phases retained 99.94% and 96.69% of filament diameter readings within the diameter tolerance band, whereas the middle phase decreased to 88.90%. The disturbed middle phase contained 3.66% of readings above 1.80 mm and 7.43% below 1.70 mm, together with 2.08% above 1.89 mm, a +0.774 mm worst excursion, a standard deviation of 0.0806 mm, and Cp/Cpk values of 0.21/0.20. The replenishment event therefore coincided with broadening in both directions rather than with a uniform displacement of the distribution. This contrasts with the primarily

undersize response produced by a similar large hopper inventory increase in the 0SHF record, demonstrating that the downstream signature depended on the interactions among formulation, pellet morphology, hopper discharge, screw replenishment, and line response.

A severe mixed-direction deterioration occurred when reprocessed 30SHF material was introduced after a highly controlled middle phase. The middle phase had retained 98.15% of readings within the band, but the corresponding end phase contained 7.93% above 1.80 mm and 7.62% below 1.70 mm. Standard deviation increased from 0.0173 to 0.1394 mm, the fraction above 1.89 mm increased from zero to 6.60%, and the worst excursion reached +1.862 mm. The nearly balanced routine losses and severe upper tail identify a broad mixed-direction state rather than a simple oversize shift.

Low hopper inventory produced another mixed-direction transition in a separate 30SHF record. The middle phase had been undersize-dominated, with no upper-side loss and 23.07% below 1.70 mm. The end phase then showed 10.38% above 1.80 mm and 20.32% below 1.70 mm, while 6.48% exceeded 1.89 mm and the standard deviation increased to 0.0725 mm. The end phase therefore remained undersize-predominant but developed a substantial and severe upper-side component, requiring classification as mixed-direction instability.

In the large-pellet/second-batch record introduced in Section 3.8.3, the end phase broadened to 16.58% above 1.80 mm and 11.36% below 1.70 mm, marking a transition from oversize-dominated to mixed-direction instability.

The most severe mixed-direction response occurred when a fines-rich final 30SHF pellet population entered the hopper. The preceding middle phase was undersize-dominated, with 14.26% below 1.70 mm and no upper-side loss. In the end phase, only 26.72% of readings remained within the band, while 45.99% exceeded 1.80 mm and 27.29% fell below 1.70 mm; 33.54% also exceeded 1.89 mm. Although the mean shifted above the upper analysis limit, the very large lower-side fraction demonstrates that this was not a simple oversize displacement. It was a severely broadened mixed-direction state with oversize predominance, consistent with highly irregular feed material delivery at the hopper and downstream filament response.

These mixed-direction records are consistent with fluctuations in screw filling and melt delivery propagating into filament dimensions, as reported for other hot-melt-extrusion and filament-manufacturing systems [12,13]. Diameter data alone cannot establish whether an individual excursion originated from air expansion, a feed pulse, pressure fluctuation, incomplete solidification, or take-up response. A specific process interpretation was therefore applied only when the directional diameter signature coincided with a documented change in feed material or operating state. Figures 5–7 summarize the phase-wise variability and severe oversize-event frequency, representative time-domain traces, and directional tolerance loss, respectively.

3.8.5. Sources of Diameter Variation

The diameter signatures reflected three coupled sources. Feedstock effects were evident when fines-rich, reprocessed, oversized, flattened, or irregular pellet populations destabilized otherwise unchanged settings. Platform effects were evident in 0SHF as well as SHF-loaded trials through hopper replenishment, depletion, screw-speed drift, and the absence of metered feeding or active venting. Downstream-line effects arose from cooling, draw distance, gauge alignment, and take-up while the strand was still solidifying. The in-line record therefore represents the coupled extrusion-line response, not material rheology or die output alone.

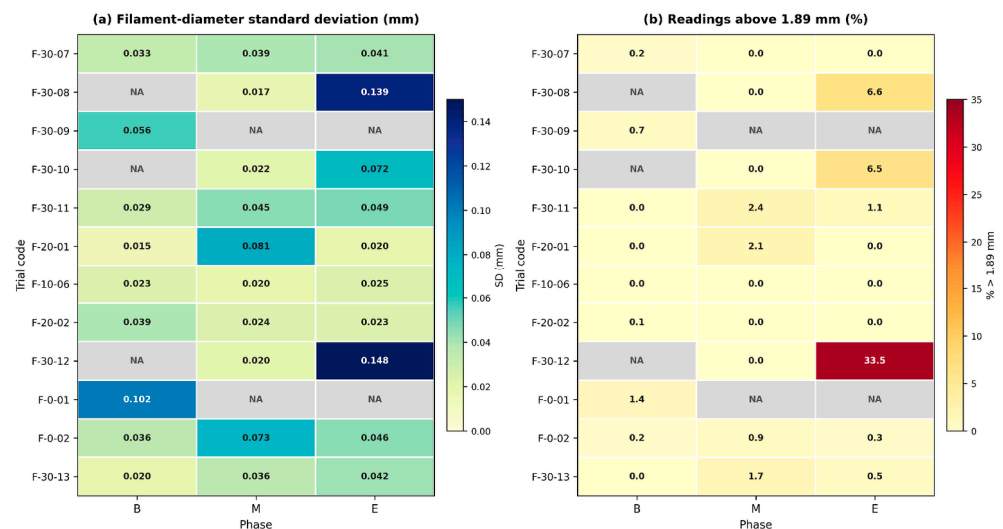


Figure 5. Phase-wise diameter variability and high-risk oversize events: (a) filament-diameter standard deviation and (b) percentage of readings above 1.89 mm. NA indicates unavailable phase data.

Representative annotated diameter time-series traces at common vertical scale

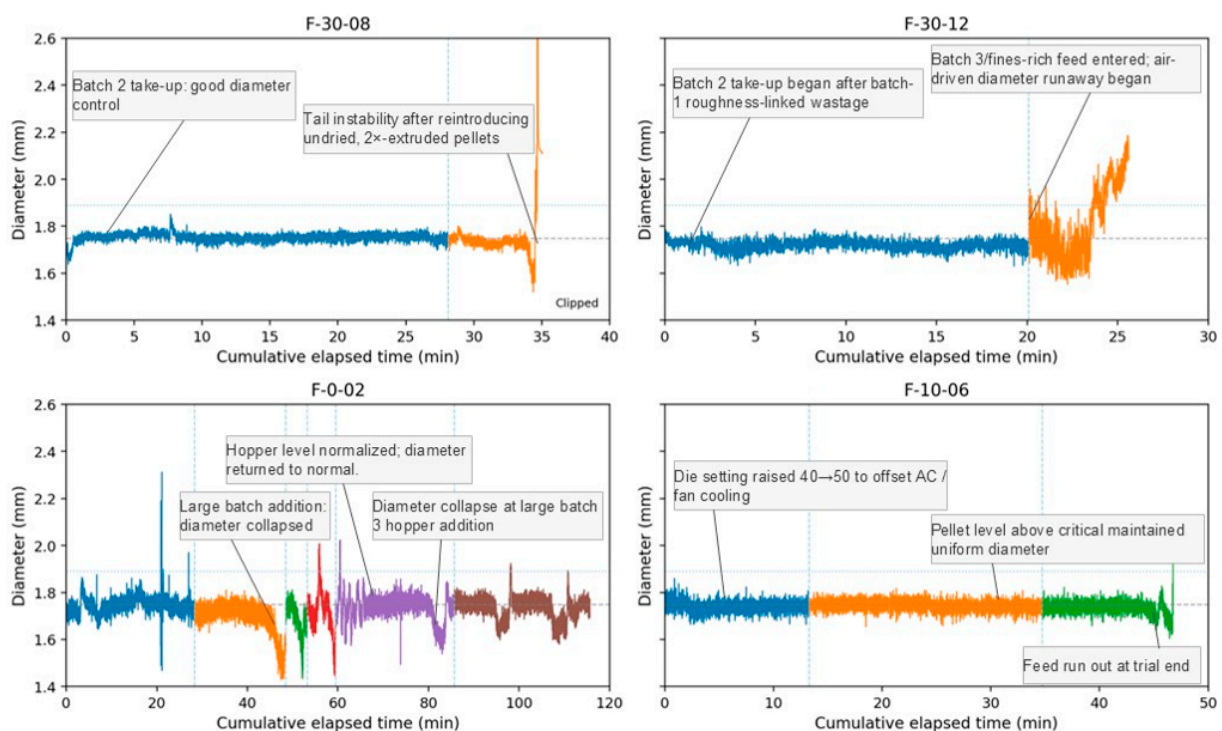


Figure 6. Annotated diameter traces for representative stable and destabilized filament-making cases: F-10-06, F-0-02, F-30-08, and F-30-12, where phase assignment is available, blue, orange, and green denote the beginning (B), middle (M), and end (E) production phases, respectively. In some traces, additional colors identify successive spool-associated or source-file segments within the plotted record and do not represent additional production phases. Vertical dashed lines indicate defensible phase boundaries where applicable.

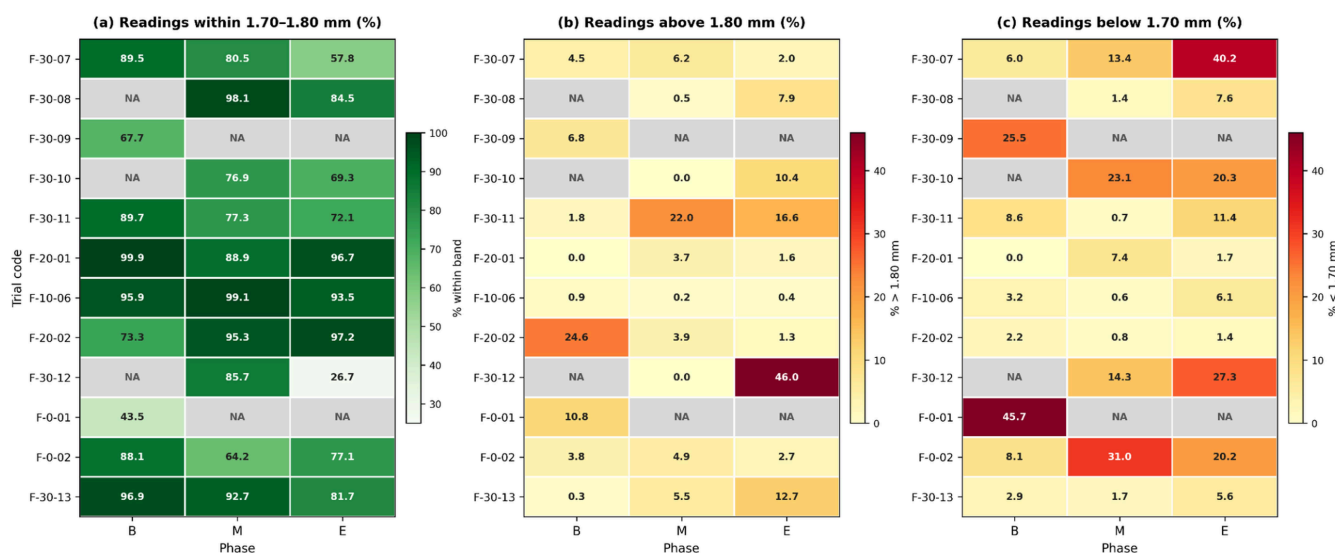


Figure 7. Phase-wise tolerance-band behavior: (a) % within 1.70–1.80 mm, (b) % > 1.80 mm, and (c) % < 1.70 mm. NA indicates unavailable phase data.

The earliest continuously logged record was the two-spool, approximately 15–18 wt.% SHF/PLA filament-making trial. It was treated as a methodological baseline rather than assigned to one of the directional process-signature categories because line geometry, cooling-air disturbance, and measurement alignment were corrected during the run. Improvement in the second spool was therefore interpreted as evidence of reduced measurement-path and downstream-line contamination, not necessarily as a change in the intrinsic extrusion state. This distinction prevents line-development effects from being misclassified as material instability.

The strongest evidence for a material–platform interaction was provided by the 30SHF records. Narrow and well-centered phases were achieved on the existing equipment, demonstrating that the formulation could be processed successfully under favorable feed and downstream-line states. Those phases subsequently transitioned to undersize, oversize, or mixed-direction instability after changes in hopper inventory, pellet morphology, recycled-material condition, or fines content. Material characteristics increased process sensitivity, while the platform architecture determined how strongly feed and line disturbances were transmitted into filament diameter.

3.8.6. Commercial Tolerance Benchmark

The 1.70–1.80 mm interval used in the primary diameter analysis was compared with publicly reported dimensional tolerances for selected commercially available plant- and wood-filled filaments (Table 6). The identified supplier specifications ranged from ± 0.02 to ± 0.10 mm around nominal filament diameter [47–52]. The ± 0.05 mm band adopted in the present study is an intermediate tolerance reported for several commercial plant-filled filament products, whereas ± 0.02 mm represents a more demanding supplier-reported specification and ± 0.10 mm represents a comparatively permissive one.

This comparison provides engineering context rather than evidence of direct product equivalence. Commercial suppliers generally report nominal diameter tolerances but do not disclose the complete measurement frequency, sampling plan, line speed, conditioning history, process-stability record, or Cp/Cpk calculation needed for a controlled capability comparison. The present in-line records, by contrast, include serial measurements collected throughout developmental extrusion runs, including startup, replenishment, intervention, material-transition, depletion, and run-out periods. Supplier tolerances and the present

capability-style metrics therefore answer different questions: the former state the advertised product requirements, whereas the latter describe how consistently an experimental process maintained a selected dimensional interval over time.

Table 6. Commercial plant-filled or wood-filled filament diameter-tolerance examples used to contextualize the 1.70–1.80 mm analysis band.

Commercial Filament	Plant Filler/System	Published Diameter Tolerance	Relevance to the Present Study
Fiberlogy FiberWood	Wood-filled filament	± 0.02 mm	Premium-tight wood-filled example with reported diameter and ovality/roundness control [47]
ZYLtech Wood+ PLA Composite Filament	20 wt.% wood-filled PLA composite	± 0.02 mm	Premium-tight plant-filled/wood-filled tolerance example [48]
colorFabb woodFill	PLA/PHA with wood fibers	± 0.05 mm for 1.75 mm filament	Directly supports the 1.70–1.80 mm tolerance band used in this study [49]
Filamentive Wood PLA	PLA-based composite with 40% recycled wood fibers	± 0.05 mm for 1.75 mm filament	Directly supports the 1.70–1.80 mm tolerance band used in this study [50]
Gembird Wood PLA	Wood-filled PLA	1.75 ± 0.05 mm	Additional commercial wood PLA example supporting a ± 0.05 mm band [51]
Fillamentum Timberfill	Biopolymer filament filled with natural wood fibers	± 0.10 mm	Shows that some natural-fiber/wood-filled commercial filaments use looser specifications [52]

The best complete 10SHF record and the strongest controlled 20SHF and 30SHF phases each retained more than 95% of readings within the ± 0.05 mm band, confirming dimensional feasibility at up to 30 wt.% SHF. These operating windows do not establish commercial equivalence or sustained production capability. Published high-loading natural-fiber/PLA filament systems have demonstrated approximately 30 wt.% hemp or harakeke fiber, 40 wt.% hemp hurd, 30 wt.% regenerated cellulose fiber, and 50 wt.% wood powder [21–25]. These loading levels establish feasibility in other material–equipment systems but are not direct dimensional or capability benchmarks because reinforcement morphology, formulation, compounding route, extrusion and take-up hardware, and printer configuration differ. The present contribution is therefore not a universal maximum-loading claim, but a quantified 30 wt.% spool-fed operating window and a platform-specific 35 wt.% handling boundary established on the present system.

The sensitivity analysis in Table 7 further distinguishes dimensional feasibility from sustained capability. Across the representative records, recalculated Cpk values ranged from 1.29 to 2.05 under a ± 0.10 mm band, from 0.58 to 0.95 under ± 0.05 mm, and from 0.08 to 0.32 under ± 0.02 mm. Because $Cpk \geq 1.33$ is commonly used as a reference criterion for process capability [46], three of the four representative records exceeded that value under the comparatively broad ± 0.10 mm band, while the remaining record approached it at $Cpk = 1.29$. None of the evaluated records reached $Cpk \geq 1.33$ under the ± 0.05 mm band used for the primary analysis or under the tighter ± 0.02 – 0.03 mm supplier-reported specifications. Because statistical stability was not established, even values above 1.33 remain capability-style comparisons rather than formal demonstrations of process capability.

The comparison quantifies the remaining scale-up gap rather than merely classifying the experimental filament as inside or outside a nominal tolerance. Selected 10–30SHF operating states achieved narrow diameter distributions within commercially recognizable

engineering intervals, while the phase-level analysis identified the disturbances that prevented those states from being maintained consistently. Industrial translation would require the favorable centering and spread achieved during the strongest phases to be sustained through automated feed delivery, controlled hopper inventory, more uniform pellet morphology, stabilized cooling and drawdown, and closed-loop take-up or diameter control.

Table 7. Effect of commercial tolerance band on recalculated Cpk for representative SHF/PLA filament diameter records.

Trial/Phase	Mean (mm)	SD (mm)	Cpk at ±0.10	Cpk at ±0.05	Cpk at ±0.03	Cpk at ±0.02
F-10-06 whole trial	1.7411	0.0236	1.29	0.58	0.30	0.16
F-20-01-B	1.7570	0.0151	2.05	0.95	0.51	0.29
F-30-08-M	1.7534	0.0173	1.86	0.90	0.51	0.32
F-30-13-B	1.7347	0.0199	1.42	0.58	0.25	0.08

Note. Cpk was recalculated from each representative record using centered specification limits around the 1.75 mm target. The selected bands correspond to the range of supplier-reported tolerances in Table 6 but do not imply equivalence between the developmental records and commercial production. The ±0.05 mm band remained the primary analysis interval. Trial identifiers are retained only to permit traceability to Table 5, Supplementary Table S7.1, and the underlying diameter records.

3.8.7. Developmental Capability

Whole-trial metrics provide useful broad summaries, but they can obscure localized disturbances because phases of different duration contribute unequally to pooled means and standard deviations. Phase-level results are therefore the primary basis for mechanistic interpretation.

Figure 8 provides the complete phase-evolution plot and shows that beginning, middle, and end phases within the same run could have differed substantially even when the nominal machine configuration remained unchanged. The full whole-trial trace atlas is provided in Supplementary Figure S4 and confirms that the logged records contained both sustained controlled windows and localized instability events that could be obscured by pooled summary statistics.

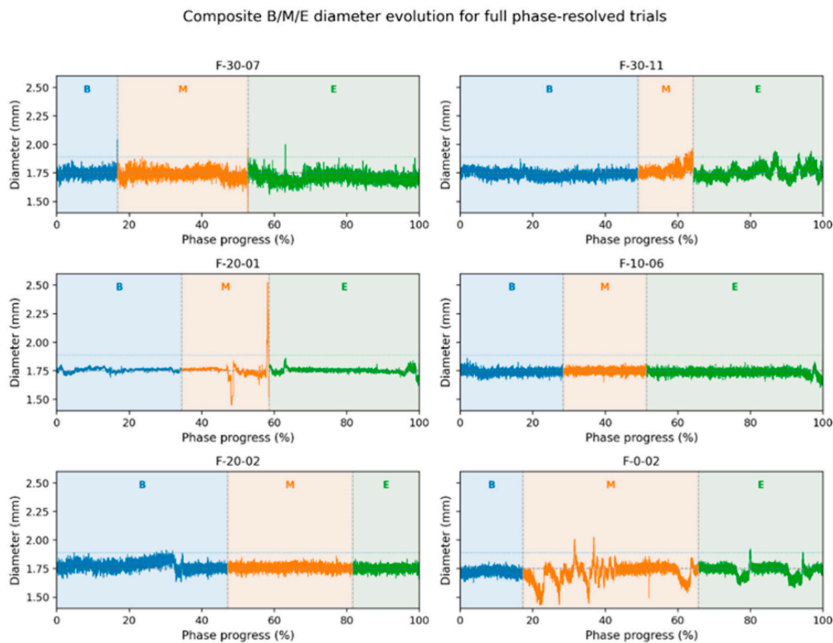


Figure 8. Composite beginning–middle–end (blue–orange–green) diameter evolution for all phase-assigned filament-making trials plotted at a common vertical scale. Trial identifiers are retained within the figure for traceability.

No formal production-capability claim is made from the reported Cp or Cpk values. The significance of the analysis is diagnostic. It demonstrates that the process could enter narrow and well-centered filament-making windows; distinguishes deterioration through lower-side drift, upper-side displacement, or mixed-direction broadening; and relates those signatures to documented changes in feedstock state, hopper discharge, screw speed, cooling, drawdown, measurement, and take-up. Stable high-loading SHF/PLA filament production consequently requires control of both the average processing conditions and the transient feed and downstream-line disturbances that determine whether those conditions remain effective.

3.9. Operational Validation by Downstream 3DP

The converged filament route supplied a downstream three-factor design spanning four SHF levels, four raster orientations, and three production phases. It produced 192 tensile, 192 compression, 144 impact, and 192 flexural specimens over approximately 936 h of printing, including approximately 234 h for 30SHF. All specimens were printed using a 0.4 mm nozzle, 0.1 mm layer height, 0.4 mm raster width, 190 °C nozzle temperature, and 40 mm s^{−1} print speed. Successful printing of 30SHF under these fixed conditions is notable because filled PLA systems commonly use or recommend 0.6 or 0.8 mm nozzles to reduce clogging risk [53–55]. Given the sensitivity of the SHF/PLA system to pellet morphology, feed continuity, hopper state, and air carry-forward, this execution scale demonstrates that the methodology had moved beyond isolated successful spools and provided sufficient filament continuity, dimensional usability, and formulation/spool-position coverage for systematic downstream characterization. The 30SHF formulation was the upper boundary and most process-sensitive material in the design; accordingly, filament-making at this loading still produced off-specification filament during periods of extrudate surface roughness, diameter variation, start-up/transient instability, and feed-state disturbance. These off-specification segments defined the remaining process variables—feed control, cooling, drawdown, and take-up—that govern yield improvement and industrial scale-up, while the converged settings produced enough dimensionally usable, spool-fed printable filament to execute the full downstream experimental design. The resulting filaments and representative 30SHF printed test specimens are shown in Figure 9.

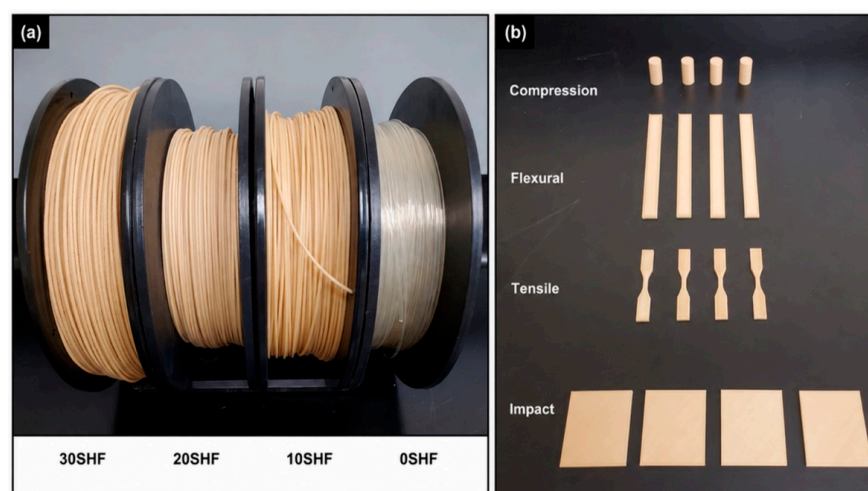


Figure 9. Operational validation of the converged SHF/PLA filament-making process. (a) Spooled filaments produced for the final experimental-design formulations: 0SHF, 10SHF, 20SHF, and 30SHF. (b) Representative 30SHF compression, flexural, tensile, and impact specimens produced for downstream mechanical characterization.

Apart from mechanical property characterization coupons, representative demonstration objects were also printed to confirm that the developed filaments could be fed, deposited, and built into non-test geometries beyond simple coupon forms (Figure 10).



Figure 10. Representative print operational validation objects produced using 30SHF/PLA bio-composite filament (only the left humanoid figure in the bottom left image was printed using 10SHF filament).

Although operationally printable, 30SHF required a narrower handling envelope than lower-SHF filaments because residual feed instability, high stiffness, and brittleness affected both take-up and printing. During filament-making, the take-up system had to be positioned closer to the die so that sufficient draw could be applied while the extrudate remained deformable; however, this reduced the cooling time available before winding. If wound too soft, adjacent coils partially fused and resisted unwinding during printing; if allowed to solidify too much before take-up, the filament snapped over the curved spool geometry. During printing, the filament could also snap if kinked during feeding to the printhead, making unsupervised printing less reliable initially. A practical workaround was to transfer filaments from the original take-up spools onto empty spools while preserving a larger individual coil circumference, effectively mimicking winding on a larger package. This reduced bending severity during unwinding and feeding. The observation supporting this workaround was that fully wound 30SHF spools did not show kink-related breakage at the outer layers, where winding circumference was larger, but became more susceptible to breakage deeper in the spool, where circumference decreased, and bending became tighter. Retraction to suppress stringing during non-printing travel moves was also unviable for 30SHF because repeated reverse motion from the printer's toothed feed gears added mechanical stress and caused breakage. These constraints define the downstream handling envelope for converged 30SHF filament rather than its material printability limit. Comparable handling problems were not encountered during printing with the produced 20SHF and 10SHF filaments.

Warping during 3D printing was observed predominantly in the 0SHF and 10SHF formulations, with the problem more pronounced in 0SHF. The behavior persisted across the practical nozzle-temperature range explored, from below 190 °C to 205 °C or higher, indicating that nozzle-temperature adjustment alone did not remove the warping tendency.

In contrast, warping was virtually absent in printed parts produced from the 20SHF and 30SHF formulations. Similar loading-dependent shrinkage or warpage suppression has been reported in other lignocellulosic-fiber/thermoplastic systems [23,56–58]. These precedents support the interpretation that increasing SHF content constrained thermally driven contraction and distortion of the PLA-rich phase during cooling. Taken together, the downstream printing results show that the converged process was sufficiently robust to support systematic specimen production and practical print demonstrations, while defining a clear development boundary; i.e., filament production, spool handling, and printer feeding remained as coupled constraints requiring further instrumentation and optimization for industrial transfer. The below limitations, therefore, distinguish what this process-development study established from what remains to be instrumented, generalized, and optimized.

4. Limitations and Scale-Up Needs

This study targeted printable high-loading SHF/PLA filament at R&D scale and used sequential, problem-driven development because the governing variables were initially unknown and physically coupled. The resulting conditions are experimentally justified operating outcomes rather than a statistically optimized design; formal factorial or response-surface optimization is now appropriate for the variables identified here.

Second, the available Brabender platform did not provide synchronized-torque or barrel-pressure acquisition across the full trial sequence. Mechanistic interpretation, therefore, rests on multiple evidence streams from repeated trial behavior, in-line diameter response, direct feed-state interventions, and observed extrusion outcomes. Future instrumentation with torque, die-pressure, hopper-fill, feed-rate, and synchronized diameter logging would allow these mechanisms to be quantified more directly and converted into predictive scale-up control variables.

Third, the process architecture was developed from extrusion behavior, in-line diameter response, and downstream printability rather than from an independent rheological model. Future rheological testing should quantify how SHF loading changes melt flow resistance, melt strength, and die-swell tendency during filament extrusion. These measurements would support the compaction-based interpretation of the filament-making window.

Fourth, the C_p/C_{pk} results function as translational capability diagnostics for an R&D filament-making platform. In an industrial production environment, capability metrics would normally be applied after machine design, feed delivery, cooling, winding, sensing, and control architecture have been fixed. Here, capability-style analysis was applied earlier in the development chain to identify when the process entered usable filament-making windows, when those windows degraded, and which feed-state, pellet-size, hopper-fill, drawdown, cooling-path, gauge-path, and take-up variables needed to be stabilized for industrial capability.

Fifth, the SEM analysis comprised one full native-resolution $50\times$ cross-section per formulation ($n = 1$). The fixed-threshold values therefore describe the imaged sections only and do not constitute statistically representative porosity distributions. Future work should analyze independently sampled cross-sections from multiple separated filament locations to quantify within-formulation variability.

The substantially larger apparent internal dark-area burden in the analyzed 30SHF cross-section is consistent with both composition-driven and process-induced contributions. Fiber agglomeration and reduced matrix continuity can increase brittleness, whereas feed interruption, fines-rich material, residual moisture, or retained air may contribute process-dependent discontinuities. Improved pellet uniformity, fines removal, controlled pellet-size,

and vented or vacuum-assisted de-aeration should therefore be evaluated using replicated cross-sectional sampling.

Downstream 3DP reliability also showed environmental sensitivity. When relative humidity in the printing area reached approximately 40% or higher, under-extrusion and print instability were observed not only for SHF-loaded filaments but also for the 0SHF control, even when the filament was fed from a heated drying box. Under these conditions, a filament that was nominally within the 1.70–1.80 mm diameter range could soften and expand prematurely above the melt zone, sometimes exceeding the empirically observed ~1.89 mm clogging threshold. This behavior indicates a heat-creep-type failure amplified by humidity and local printer-side thermal conditions. Because similar behavior occurred in 0SHF, $RH \approx 40\%$ is best interpreted as a setup-specific heat-creep threshold governed by filament moisture, room humidity, cold-side cooling, hot-end geometry, and filament conditioning history.

For brittle high-loading filaments, future investigations should also evaluate positively driven spool-assist systems synchronized with the printer feed rate. Actively driven unwinding could reduce the tensile and bending stresses imposed by passive spool rotation, improving feed reliability for 30SHF and other highly filled printable systems.

Industrialization should prioritize controlled pellet morphology and fines removal, regulated hopper mass flow, synchronized torque/pressure/diameter logging, controlled cooling, and closed-loop take-up. These controls provide the most direct route from the demonstrated R&D process to stable production.

5. Conclusions

A filament-making pathway was established for converting compatibilizer- and plasticizer-free SHF/PLA compounded pellets into 1.75 ± 0.05 mm FDM filament using the Brabender single-screw extruder and Filabot cooling and take-up platforms. The central process result was that filament-making required a compaction-oriented architecture that limited air carry-forward, capable of destabilizing filament diameter and surface smoothness. The converged SHF-loaded filament used a single-mixing-zone screw, a 3.85 mm orifice/5.75 mm land die, a screw speed of 10 rev/min, and a 160/170/180/195 °C barrel temperature profile. Neat PLA required a separate 180/185/200/205 °C profile, confirming that the filament-stage thermal requirement was SHF-loading-dependent.

Hopper discharge and screw replenishment were the principal operating-state controls. Large replenishment events, low-inventory depletion, bridging, irregular pellets, reprocessed/recycled material, fines-rich feed, and screw-speed drift could move the same nominal machine configuration among controlled, undersize-dominated, oversize-dominated, and mixed-direction states. Phase-resolved in-line diameter analysis was therefore more informative than whole-trial averaging alone. The strongest sustained benchmark was a 10SHF filament-making trial based on converged settings, which had a whole-trial mean diameter of 1.7411 mm, a standard deviation of 0.0236 mm, and 95.45% of readings within 1.70–1.80 mm and only 0.013% above 1.89 mm. Cp and Cpk were useful for summarizing spread and centering but remained capability-style diagnostics rather than evidence of a statistically controlled production process.

The highest formulation that could be wound sustainably during filament-making and unwound and supplied reliably from a spool during 3D printing was 30SHF. The 35SHF filament could still 3D print when loose coils were supplied without spool-unwinding resistance, but it fractured repeatedly during direct spool feeding. The observed boundary is therefore a platform-specific spool-fed handling limit rather than an intrinsic SHF/PLA deposition limit. Operational validation comprised 720 mechanical-test specimens printed across four formulations, four raster orientations, and three production phases over approxi-

mately 936 h, demonstrating that the converged route could support sustained downstream experimentation.

This study was intentionally structured as a sequential process-development investigation to identify coupled failure mechanisms and establish workable operating windows under high-loading conditions. Accordingly, the reported process settings and spool-fed loading boundary are specific to the SHF/PLA formulations and Brabender–Filabot platform evaluated, while the Cp/Cpk values are used as comparative diagnostics rather than formal production-capability certifications. Future research should build on the established process map through designed experiments that independently vary hopper inventory, pellet morphology, screw speed, and thermal profile; incorporate synchronized-torque, melt-pressure, pellet-feed-rate, and filament-diameter measurements; and extend morphology across multiple independently sampled cross-sections and processing states. These studies would quantify within-formulation variability, evaluate transferability to other extrusion, cooling, and take-up systems, and support closed-loop feed and diameter control for production-scale implementation.

Supplementary Materials: The following supporting information can be downloaded at <https://www.mdpi.com/article/10.3390/fib14070084/s1>: Supplementary Section S1: filament-stage equipment, hopper-agitation hardware, and the complete process-variable matrix; Supplementary Section S2: native-resolution SEM apparent dark-area methodology, fixed-threshold justification, threshold-sensitivity results, overlay verification, mask-audit verification, and reproducibility details; Supplementary Section S3: in-line diameter-processing and metric definitions; Supplementary Section S4: supplementary data-file map; Supplementary Section S5: main-manuscript crosswalk; Supplementary Section S6: complete 33-trial filament-making registry; Supplementary Section S7: whole-trial diameter metrics, trial-level interpretation, and full-trial trace atlas; and Supplementary S1: the native-resolution Python script used for the SEM apparent dark-area analysis [32,33,59–61].

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