NatureWorks

# 3D Printing Monofilament Guide for Ingeo Biopolymer

This information is intended for use only as a guide for making 3D printing monofilament using Ingeo™ biopolymer. It will consist of generalized concerns for safety, equipment, and process conditions.

Since making monofilament can be complex and may cover a broad array of 3D printing applications, polymers, additives, and blend systems, an experimental approach using Ingeo may be required in order to achieve optimal results.

3D print testing of the Ingeo monofilament is also recommended in order to make sure it meets customer requirements.

## 1.0 Safety and Handling Precautions

All safety precautions normally followed in the handling and processing of melted thermoplastics should be followed for Ingeo biopolymer resins.

As with most thermoplastics, melt processing and the variability of those conditions may result in minor decomposition. Lactide, a non-hazardous gaseous irritant, is a minor by-product of Ingeo melt processing. Appropriate air testing should be completed to ensure an acceptable Threshold Limit Value (TLV) of less than 5 mg/m$^3$ is maintained. The use of process area point source remediation measures such as monomer fume hoods or exhausts near melt processing equipment are typically recommended.

Molten Ingeo is lower viscosity and sticks more readily to cloth, metal, brass and wood compared to other molten thermoplastics. Be prepared for this when cleaning die faces, purging equipment, collecting molten patties, and emptying purge containers. Unlike polyolefins, molten Ingeo will not release as cleanly from a gloved hand so use caution when grabbing any stream or patty.

Ingeo is considered non-hazardous according to DOT (US Department of Transportation) shipping regulations. When handling Ingeo resin at room temperature, avoid direct skin and eye contact along with conditions that promote dust formation. For further information, consult the appropriate MSDS for the Ingeo grade being processed.

As with any melted thermoplastic waste, melted Ingeo waste should be allowed to cool before being placed into any waste container to minimize fire risks.

## 2.0 Pellet Storage and Blending Recommendation

Ingeo resins should be stored in an environment designed to minimize moisture uptake. Product should also be stored in a cool place at temperatures below 122°F (50°C).

Product that is delivered in cartons or super sacks should be kept sealed until ready for loading into the blending and/or drying system. Bulk resin that is stored in closed silos and hoppers for extended periods (more than 6 hrs) should be kept purged with dry air or nitrogen to minimize moisture gain. In the case of outside storage, if the product is supplied in boxes or other non-bulk containers, the unopened container should be brought into the extrusion production area and allowed to equilibrate for a minimum of 24 hours before opening to prevent excessive condensation.

## 3.0 Resin Properties

Ingeo 3D printing monofilament grades are specifically made for 3D printing applications. Typical properties are shown in the table below.

| Resin Property | Nominal Value |
|---|---|
| Specific Gravity | 1.24 |
| Peak Melt Temperature, °F (°C) | 302-347 (150-175) |
| Glass Transition Temperature, °F (°C) | 131-140 (55 – 60) |
| Crystallization Temperature, °F (°C) | 203– 248 (95 – 120) |
| Typical Part Shrinkage[(1)], in/in | 0.004 +/- 0.001 |

[(1)] Based on injection molded part measurements.

## 4.0 Materials of Construction

All metal parts in the extrusion process that are subjected to stagnant flow areas with molten polymer should be constructed of stainless steel to minimize corrosion. This includes melt pump and filter assemblies and some transfer lines. Furthermore, Ingeo biopolymer should not be left in the extruder, polymer filter, polymer transfer lines, dies or any other part of the extrusion system at recommended or higher melt temperatures for extended periods. Below is a guideline for the recommended types of steel that should be used in the extrusion system.

| Part | Steel Type |
|---|---|
| Melt pumps and bearings | SUS440B |
| Pump blocks | SUS631 |
| Transfer lines | SUS440C |
| Die | Hard Chrome plated tool steel |

If twin-screw extrusion with vacuum vent drying is used, SUS440C or similar corrosion-resistant material should be used for screw elements, vacuum vent lines, and trap components. Contact your NatureWorks technical representative for additional recommendations on proper handling of lactide gasses and trapped lactide.

## 5.0 Drying

Ingeo resins can be successfully dried using most standard drying systems. Recommended conditions are provided for standard desiccant based column dryers. For other drying system designs, additional information can be provided upon request.

To prevent equipment corrosion, it is not recommended to dry or store hot Ingeo resin in carbon steel vessels (see Section 2.0).

**In-line drying is essential for Ingeo resins**. It is recommended that Ingeo should be dried to a maximum of 250 ppm of moisture as measured by a Karl Fischer method. A moisture level lower than 250 ppm will help keep the melt viscosity stable over time at elevated temperatures. Processes that have unusually long residence times or result in melt temperatures greater than 240°C should only extrude Ingeo at moisture levels less than 50 ppm for maximum retention of molecular weight and physical properties. Material is supplied in foil-lined containers dried to less than 400 ppm as measured by NatureWorks' internal method. The resin should not be exposed to atmospheric conditions after drying. Keep the package sealed until ready to use and promptly dry and reseal any unused material. The drying table below can be used to estimate the drying time needed for Ingeo. Air or nitrogen based desiccant drying systems can be used at the recommended temperatures. Typical Ingeo drying conditions are shown in the table below.

**Typical Ingeo Raw Material Drying Conditions**

| | Typical Settings | |
|---|---|---|
| **Drying Parameter** | **Amorphous** | **Crystalline** |
| Residence Time (hours) | 4 | 2 |
| Air Temperature °F (°C) | 104-113 (40-45) | 176-194 (80-90) |
| Air Dew Point °F (°C) | - 40 (-40) | - 40 (-40) |
| Air Flow Rate, CFM/lb resin ($m^3$ / hr - kg resin) | > 0.5 (1.85) | > 0.5 (1.85) |

Typical desiccant dryer regeneration temperatures exceed the melt point of Ingeo resins. To prevent issues with pellet bridging, sticking, or melting, the drying system should be verified to ensure temperature control is adequate during operation as well as during regeneration cycles since valve leakage is common in many systems. Installation of a water-cooled after-cooler may be necessary to prevent the drying air temperature from exceeding the recommended set point when drying amorphous materials. Ingeo is a semi-crystalline product that can come in either amorphous or crystalline form and can be dried accordingly as per the table above.

## 6.0 Melt Processing & Extrusion

Prior to introducing Ingeo into any melt processing system, the system should be properly cleaned and purged to prevent any polymer cross contamination. Ensure that the feeding & blending equipment is thoroughly cleaned & free from dust and contamination and all metal magnets have been wiped clean. Ensure that all hang-up areas such as elbows, transitions and slide gates have all dust and granules from previous runs completely removed. The purging procedures below are recommended for optimal removal of other polymers.

## 6.1 Ingeo Purging Procedure

### Following PET, PA, HDPE, or other higher melting polymers in your system

1. It is critical to clean the material handling system of PET, nylon and high molecular weight HDPE to ensure that these materials do not inadvertently feed into the extruder during or after the purging process.
2. Purge with low MFR (e.g., <1) transition resin at normal PET operating temperatures. PET and Ingeo are temperature incompatible, so the transition resin is one that can be processed at the high temperatures of PET and the low temperatures of Ingeo. Suggested transition resins include PP, crystal PS, and PETG. Purge for at least 7x average residence time, much of the time at the typical PET production rate (~30 minutes). Let system empty as much as possible. Clean out the hopper as much as possible.
3. Introduce a higher melt flow transition resin of the same type (5 - 8 MFR) and change to normal Ingeo operating temperatures. Ingeo is a relatively low viscosity resin, so the grade of transition resin that is viscous enough to push PET or nylon out of the extruder at the high extrusion temperatures will be too viscous for Ingeo to push it out at the lower extrusion settings. This is why a transition to a higher melt flow transition resin is suggested: to have a viscous version to push out the PET, PA or HDPE and a low viscosity version that the Ingeo will push out well.
4. Purge for at least 7x average residence time. Let system empty as much as possible.
5. Extrusion operations familiar with PETG processing may find success purging using a single PETG grade (similar to Eastman copolymer 6763) for a minimum of 40 minutes. Then reduce temperatures to Ingeo conditions.
6. Turn off extruder and completely clean all hoppers, elbow, slide gates, dryers, hopper loaders bins, hopper loader filters and material conveying lines of residual resin pellets, flake, dust, and floss. Load Ingeo into material handling system.
7. Transition to Ingeo and purge again for a minimum 7x the average residence time. Change the screen pack when it becomes obvious that primarily Ingeo is exiting the die. Be sure to flush screen pack completely during the change. Screen pack should be between 40- 125 mesh for optimal performance. The lower mesh screen will result in reduced melt temperatures exiting the die.
8. At the completion of a trial run, purge all Ingeo from the extrusion system, using a moderate to low melt index resin that processes well between 400 and 450°F (204-232°C), initially using the Ingeo temperature settings and extrusion rates.

**Notes:**

1. It is critical that all drying and conveying/receiving systems be free of all incumbent polymer and is vacuumed to ensure that there is no remaining polymer dust before adding Ingeo. Some polymers will not melt at Ingeo operating temperatures and will block screens if it is present in the system.
2. The brand of PP used for purging is unimportant, as long as it does not thermally cross-link.

## 6.2 Extrusion

A general-purpose single-screw extruder, 24 to 36:1 L/D with feed-throat cooling, is acceptable for processing Ingeo. A mixing section is generally recommended along with static mixers in the product line prior to the die to ensure temperature uniformity as well as optimum additive dispersion and melt polymer homogeneity. For single-screw extrusion systems, melt pumps are recommended for profile consistency. The following table shows a typical melt profile for Ingeo.

**Typical Ingeo Biopolymer Extrusion Conditions**

| Section | Standard Temperature Profile, °F (°C) | Reverse Temperature Profile, °F (°C) |
|---|---|---|
| Feed throat | 113 (45) | 113 (45) |
| Zone 1 | 355 (180) | 430 (220) |
| Zone 2 | 375 (190) | 410 (210) |
| Zone 3 | 390 (200) | 390 (200) |
| Melt pump | 390 (200) | 390 (200) |
| Die | 375 (190) | 375 (190) |

**Note 1:** In some instances where the extruder contains a screw not specifically designed for Ingeo biopolymer, a reverse temperature profile has proven to be beneficial to reduce melt temperature and reduce extruder motor load.

**Note 2:** Temperatures are only for reference and may need to be altered or optimized on each processing line. Ingeo target melt temperatures (after melt pump) should be in the range of 210±10°C (410±20°F). Ingeo resins should not be processed at temperatures above 240°C (464°F) due to excessive thermal degradation.

## 6.3 Additives

Color concentrates, slip and anti-block agents are best added as a masterbatch at 15-30 wt% in Ingeo resins and controlled dosing the required amount of dried masterbatch into the feed throat of the running extruder. The additions of colorants have been successfully done using liquid injection technology, as well. Since Ingeo is not compatible with most incumbent materials, it is important that all additive masterbatches use Ingeo as the carrier. Also, the additive dosing system should be clean of any incumbent masterbatches or additives, to prevent polymer cross-contamination. Some potential additives are inappropriate for extrusion with Ingeo because they are hygroscopic or hydrated salts (e.g. calcium carbonate, zinc stearate, or calcium stearate) and would lead to severe Ingeo molecular weight degradation and property loss. In some cases, slip or process aids (both internal and/or external topical coatings) may be used to help improve Ingeo’s frictional properties for such things as improved spool package build, filament unwinding, or easier printer head loading and take-off.

## 6.4 Filtration

Standard screen pack configurations are recommended to protect melt pumps and dies from incidental contamination that may occur during material handling. The appropriate screen mesh size should be considered, depending on your equipment set-up, monofilament die size, filament diameter, and also 3D printing nozzle diameter. Screen mesh sizes of 60 - 125 mesh are generally sufficient, or 80-125 mesh if needed for smaller filament diameters or nozzle designs. Finer filtration may lead to excessive shear-heating with Ingeo, and so an experimental approach may be necessary to achieve desired results.

## 7.0 Monofilament Processing

Extruding Ingeo monofilament for 3D printing can be accomplished using conventional quenching, guiding, and take-up/winding equipment. Stagnant flow or dead spots should be avoided in any melt transfer lines and die designs, since there is the possibility of resin degradation at these spots over time. A monomer exhaust system is preferred to prevent buildup of residual lactide around the die face and quench system.

The extrusion system should have good temperature and output control. Throughput control is critical for making consistent monofilament along the length of the filament within a spool, and from spool-to-spool. Some single screw extruders are capable of providing uniform throughput at a given screw speed. Uniform extrusion temperatures and pressures are key parameters to monitor over time to ensure consistent, steady-state conditions during monofilament manufacturing. Some extrusion systems may require the addition of a melt pump to achieve reliable output of the extrudate. An experimental approach may be needed to achieve desired results.

Because of PLA's inherently low melt strength, the die should be positioned relatively close to the water bath entry point for optimal quenching of the monofilament melt. Proper quenching is important to ensure uniform monofilament cross-sections (roundness or lack of ovality), take-up and roll transfer, and to prevent sticking of the monofilament to itself or the quench/take-up system. The quench system should have good temperature control and adequate circulation capabilities.

The Ingeo monofilament out of the die can be quenched and conditioned using typical bath temperatures and residence times, however an experimental approach may be needed to account for its lower glass transition temperature versus ABS.

The quench bath should be sufficiently long enough to allow for adequate cooling of the monofilament before take-up and winding. Two water baths are typically used to quench and control the quality of the monofilament. The first water bath is typically set above the $T_g$ of Ingeo (>55-60ºC) to control and set the diameter and ovality. Care should be taken to ensure the monofilament doesn't get pinched or damaged in this first water bath. Adjustable guide rolls in the water bath can be used to control dwell time and water exposure. An experimental approach may be needed to find the right balance of water bath temperature, dwell time, and water exposure to optimize the diameter and ovality and their variation. The second water bath is typically set below the Tg of Ingeo (<55-60ºC) to solidify the monofilament after the first water bath and before winding. Again, care should be taken to ensure the monofilament doesn't get damaged in this second water bath, due to pinching or guiding damage/abrasion. Guide rollers with low friction surfaces can be used to help with transferring the filaments from each process station.

Take-up and guide rolls should be designed and run to provide enough tension level on the monofilament during water quenching and take-up out of the bath, so as to promote smooth winding. If the tension is too excessive, the monofilament could begin drawing either when it is still hot or warm, or when it is cooled below $T_g$. This will cause either unnecessary draw orientation, which could result in filament diameter changes or stress in the monofilament. Cold drawing could also result in stress cracking, stress whitening, or damage to the monofilament.

Take-up winding speed may need to be adjusted depending on extrusion throughput, monofilament die design, and overall line design. Also, nip rolls or adjustable pre-tension stands may be used to control monofilament line tensions and to maintain cross-section and surface uniformity. Spool winding and package build should be adjusted so that the monofilament is not wound too tightly on the spool, but at the same time not so loose that it could unravel – either way affecting proper and controlled monofilament haul-off from the spool to the 3D printer. High tension or stress on the filament during winding should be avoided, to prevent introducing stress into the filament or cause damage.

## 7.1 Monofilament Diameter and Quality

Typical 3D monofilament diameters are either 1.75 mm or 2.85 mm. Accurate and precise monofilament diameter control is critical for good quality 3D printing. An on-line diameter and ovality measuring device and control system is recommended during production to ensure proper gauge and shape consistency and to catch any flaws. This enables uniform and consistent quality control of the monofilament's cross-section dimensions within a spool and from spool-to-spool. Maximum diameter deviation typically should not exceed +/- 3%.

## 7.2 Packaging and Storage

To prevent moisture up-take over time and also unnecessary foreign material contamination, the monofilament should be packaged and sealed in an airtight container, barrier foil bag, or vacuumed packed film. Packaged spools should be stored in a cool, clean, and dry location. Storage < 40ºC with low %RH/moisture levels is recommended.

### Safety and Handling Considerations

Material Safety Data (MSD) sheets for Ingeo biopolymers are available from NatureWorks LLC. MSD sheets are provided to help customers satisfy their own handling, safety, and disposal needs, and those that may be required by locally applicable health and safety regulations, such as OSHA (U.S.A.), MAK (Germany), or WHMIS (Canada). MSD sheets are updated regularly; therefore, please request and review the most current MSD sheets before handling or using any product.

The following comments apply only to Ingeo biopolymers; additives and processing aids used in fabrication and other materials used in finishing steps have their own safe-use profile and must be investigated separately.

### Hazards and Handling Precautions

Ingeo biopolymers have a very low degree of toxicity and, under normal conditions of use, should pose no unusual problems from incidental ingestion, or eye and skin contact. However, caution is advised when handling, storing, using, or disposing of these resins, and good housekeeping and controlling of dusts are necessary for safe handling of product. Workers should be protected from the possibility of contact with molten resin during fabrication. Handling and fabrication of resins can result in the generation of vapors and dusts that may cause irritation to eyes and the upper respiratory tract. In dusty atmospheres, use an approved dust respirator. Pellets or beads may present a slipping hazard. Good general ventilation of the polymer processing area is recommended. At temperatures exceeding the polymer melt temperature (typically 170ºC), polymer can release fumes, which may contain fragments of the polymer, creating a potential to irritate eyes and mucous membranes. Good general ventilation should be sufficient for most conditions. Local exhaust ventilation is recom-mended for melt operations. Use safety glasses if there is a potential for exposure to particles which could cause mechanical injury to the eye. If vapor exposure causes eye discomfort, use a full-face respirator. No other precautions other than clean, body-covering clothing should be needed for handling Ingeo biopolymers. Use gloves with insulation for thermal protection when exposure to the melt is localized.

### Combustibility

Ingeo biopolymers will burn. Clear to white smoke is produced when product burns. Toxic fumes are released under conditions of incomplete combustion. Do not permit dust to accumulate. Dust layers can be ignited by spontaneous combustion or other ignition sources. When suspended in air, dust can pose an explosion hazard. Firefighters should wear positive-pressure, self-contained breathing apparatuses and full protective equipment. Water or water fog is the preferred extinguishing medium. Foam, alcohol-resistant foam, carbon dioxide or dry chemicals may also be used. Soak thoroughly with water to cool and prevent re-ignition.

### Disposal

DO NOT DUMP INTO ANY SEWERS, ON THE GROUND, OR INTO ANY BODY OF WATER. For unused or uncontaminated material, the preferred options include recycling into the process or sending to an industrial composting facility, if available; otherwise, send to an incinerator or other thermal destruction device. For used or contaminated material, the disposal options remain the same, although additional evaluation is required. (For example, in the U.S.A., see 40 CFR, Part 261, “Identification and Listing of Hazardous Waste.”) All disposal methods must be in compliance with Federal, State/Provincial, and local laws and regulations.

### Environmental Concerns

Generally speaking, lost pellets are not a problem in the environment except under unusual circumstances when they enter the marine environment. They are benign in terms of their physical environmental impact, but if ingested by waterfowl or aquatic life, they may mechanically cause adverse effects. Spills should be minimized, and they should be cleaned up when they happen. Plastics should not be discarded into the ocean or any other body of water.

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NatureWorks
15305 Minnetonka Blvd., Minnetonka, MN 55345