

Partially Oriented Yarn (POY) Process Guide

This information is intended to be used only as a guide for the manufacture of PLA fibers. Because melt spinning and downstream processing of PLA fibers is complex, an experimental approach may be required to achieve desired results.

1.0 Safety and Handling Precautions

All safety precautions normally followed in the handling and processing of melted thermoplastics should be followed for NatureWorks® PLA resins.

As with most thermoplastics, melt processing and the variability of those conditions will result in minor decomposition. Lactide, a non-hazardous gaseous irritant, is a minor by-product of PLA melt processing. Appropriate air testing should be completed to ensure acceptable Threshold Limit Values (TLV) of less than 5 mg/m³ are maintained. The use of process area point source remediation measures such as monomer fume hoods or exhausts near the spinneret are typically recommended.

PLA is considered non-hazardous according to DOT shipping regulations. Care should be taken to avoid direct skin/eye contact along with conditions that promote dust formation. Product may cause eye/skin irritation. Product dust may be irritating to eyes, skin and respiratory system. Caused mild to moderate conjunctival irritation in eye irritation studies using rabbits. Caused very mild redness in dermal irritation studies using rabbits (slightly irritating). Ingestion may cause gastrointestinal irritation, nausea, vomiting and diarrhea. For further information, consult the appropriate MSDS for the PLA grade being processed.

2.0 Pellet Storage Recommendations

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

Boxed material should be kept in sealed containers until ready for loading into the blending and/or drying system. Bulk resin stored in silos, hoppers etc 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 fiber production area and allowed to equilibrate for a minimum of 24 hours before opening.

3.0 Materials of Construction

Recommended materials of construction for vessels used for drying of polylactide polymers should be corrosion resistant.

All metal parts in the extrusion process should be constructed of stainless steel to minimize corrosion. Furthermore, PLA should not be left in the extruder, polymer filter, polymer transfer lines, spin beam, or the spinnerets at PLA melt temperatures or higher for extended periods. Below is a guideline for the types of stainless steel that should be used in the extrusion system.

Part	Steel Type
Melt pumps and bearings	SUS440B
Pump blocks	SUS631
Transfer lines and spin beam	SUS440C

4.0 Line Preparation

Prior to introducing PLA into any melt spinning system, the system should be properly purged to prevent any polymer contamination and spinning problems from occurring. The purging procedures below are recommended for optimal removal of other polymers.

PLA Purging Procedure Following PP in your system (not degraded by cooling and re-heating system)

1. At normal PLA operating temperatures, run a high melt index PP (15- 40 MI) without spin pack in place. Purge for at least 3x average residence time without spinneret in place. Let system empty as well as possible.
2. Transition to PLA and purge following the same guidelines as step 1.
3. Insert a pre-heated spin pack and allow temperature to equilibrate.
4. Purge with PLA and evaluate flow from capillaries. As long as flow is even from each capillary and there is no evidence of contamination, begin spinning.
5. Purge all PLA from your extrusion system immediately after completing a production trial or run.

Following PET, Nylon or HDPE in your system

1. Purge with low MI (<1) PP at normal PET operating temperatures. Purge for at least 3x average residence time or until PP stream is clean PP without spinneret in place. Let system empty as well as possible.
2. Change to normal PLA operating temperatures and run a high melt index PP (15-40 MI). Purge for at least 3x average residence time without spinneret in place. Let system empty as well as possible.
3. Transition to PLA and purge following the same guidelines as steps 1 and 2.
4. Insert a pre-heated spin pack and allow temperature to equilibrate.
5. Purge with PLA and evaluate flow from capillaries. As long as flow is even from each capillary and there is no evidence of contamination, begin spinning.
6. Purge all PLA from the extrusion system immediately after completion of the run

Important Notes:

1. It is critical that all drying and conveying/receiving systems be free of all PET/Nylon/PP or other contaminant and is vacuumed to ensure that there is no remaining polymer dust, before adding PLA. PET/Nylon will not melt at PLA operating temperatures and will block screens if they are present in the system
2. Brand of PP used for purging is unimportant, as long as it does not thermally cross-link.
3. When handling PLA pellets, the generation of small particles or fines is possible. Conveying pellets slower, such as at a velocity of 25 m/s, will generate fewer fines than at 30 m/s when conveying in dilute phase. Please note that with dilute phase conveying, enough velocity must be maintained to prevent the pellets from plugging the line. Internal and external testing did not show plugging problems at 25 m/s.

5.0 Spinning

5.1 Drying

PLA resin 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 PLA resin in carbon steel vessels (see Section 3.0).

In-line drying is essential for PLA resins. A moisture content of less than (50 PPM) is recommended to prevent viscosity degradation. Material is supplied in foil-lined boxes dried to less than 400 PPM as measured by NatureWorks LLC 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 PLA. Air or nitrogen based desiccant drying systems can be used at the recommended temperatures. Typical desiccant drying system conditions follow:

Drying Parameter	Typical Settings	
Residence Time (hours)	Minimum 4 hrs	Minimum 2 hrs
Air Temperature (°C)/ (F)	80 C / 176 F	100 C / 212 F
Air Dew Point (°C)	- 40	- 40
Air Flow Rate (ft ³ /min/lb resin)	> 0.25	> 0.25

Note: 1. Typical desiccant dryer regeneration temperatures exceed the melting point of PLA resins. To prevent issues with pellet bridging, sticking or melting, the drying system operation 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.

Note: 2. Above recommendations are based on using chip with taken from boxes at 400 ppm moisture or less. Actual drier performance may vary and chip moisture after drying should be measured.

5.2 Extruder

General Purpose Single-Screw extruder, 24 to 32:1 L/D with feed-throat cooling. A mixing tip is generally recommended along with static mixers in the product line to ensure temperature uniformity as well as optimum additive dispersion and melt polymer homogeneity.

Extrusion Conditions
Feed throat 25°C (77°F)
Zone 1 200°C (392°F)
Zone 2 220°C (428°F)
Zone 3 230°C (446°F)
Melt pump 230°C (446°F)
Spin head 230°C (446°F)

Note 1: Temperatures are only starting points and may need to be altered. Target PLA melt temperatures (after melt pump) should be in the range of $235\pm 5^{\circ}\text{C}$ ($455\pm 9^{\circ}\text{F}$).

Note 2: PLA resins should not be processed at temperatures above 250°C (482°F) due to excessive thermal degradation.

5.3 Additives

Delusterants such as TiO_2 are best added as a masterbatch at 15-30 wt% in PLA resins and controlled dosing the required amount of dried masterbatch into the feed throat of the running extruder. Sachtleben TiO_2 has been proven to work well with PLA. Proper grades can be recommended by CD and provided by Sachtleben upon request.

5.4 Heating Systems

To allow for the required temperatures to be obtained in spinning, typically vapor heat transfer system medium changes are required unless a vacuum assisted system is available. Dowtherm™ / Therminol™ or a comparable vapor HTM which has an atmospheric boiling point of 200°C (392°F) or less while remaining within specific system pressure design limits is generally recommended. For vacuum assisted systems, typically heat transfer medium changes are not required as long as the system vacuum can be operated at a level to provide vaporization and uniform heating at the suggested temperatures ($230\text{-}240^{\circ}\text{C}$ or $446\text{-}464^{\circ}\text{F}$).

Operation of the HTM system at a temperature as close as possible to the actual melt temperature ($235\pm 5^{\circ}\text{C}$ or $455\pm 9^{\circ}\text{F}$) is recommended to provide an adiabatic spinning system.

Similar reductions in spin pack preheating ovens should be made. Recommended settings are usually 250°C (482°F) to allow for some temperature loss during spin pack installation.

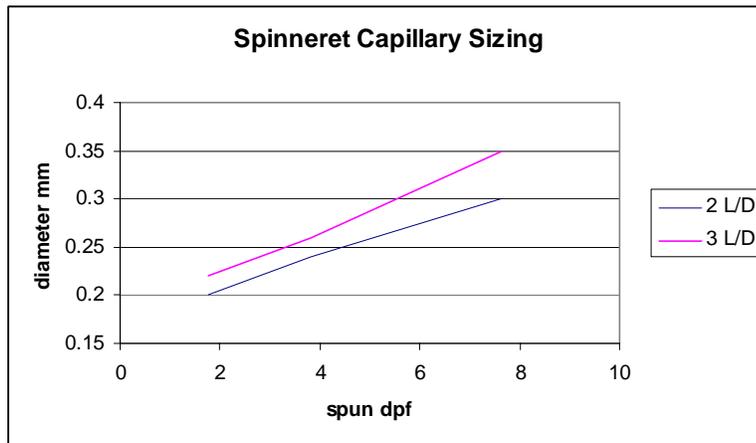
5.5 Pack Build/filtration

PLA resin will typically be provided pre-filtered to a level of 20 microns. The following pack makeup is recommended:

Loose media - 200-350 micron shattered metal is recommended for an uncompressed pack cavity fill. Loose media filtration is strongly recommended to capture residual dust contamination from other polymers that may remain in the spinning system and can adversely effect spinning performance.

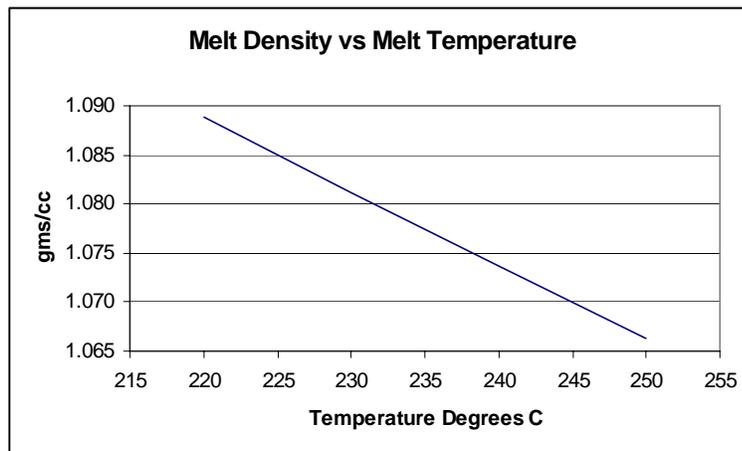
Screens – cascade configuration with appropriate support screens is recommended with finest filtration level of 20 microns.

Spinneret – Recommended capillary dimensions range from 0.2-0.35 mm diameter, typically with a 2 to 3:1 L/D ratio. The following guide can be used to estimate spinneret requirements based on spun product dpf:



5.6 Spinning Parameters

The following guide can be used to determine PLA melt density as a function of melt temperature for determination of metering pump speeds as well as additional process calculations.



5.7 Quench Conditions

As with most POY processes, uniform quenching is critical to providing a consistent PLA POY fiber with acceptable draw tension variation and denier uniformity. Quench air should be provided to the filaments between 30 to 70 mm from the face of the spinneret for most yarn counts. An initial recommended setting of 0.55 ± 0.1 meters per second quench velocity with a stable quench air temperature in the range of 18-22°C (64-72°F) is desired. Depending on actual yarn count, filament spacing and machine design, these conditions (quench delay, velocity and temperature) will vary and should be adjusted within the recommended ranges to obtain acceptable draw tension uniformity and denier uniformity values.

If available, a monomer exhaust system, or fume hoods near the spinneret should be operated to provide exhaust velocities at intake port of ~0.50 meters per second.

5.8 Finish Application

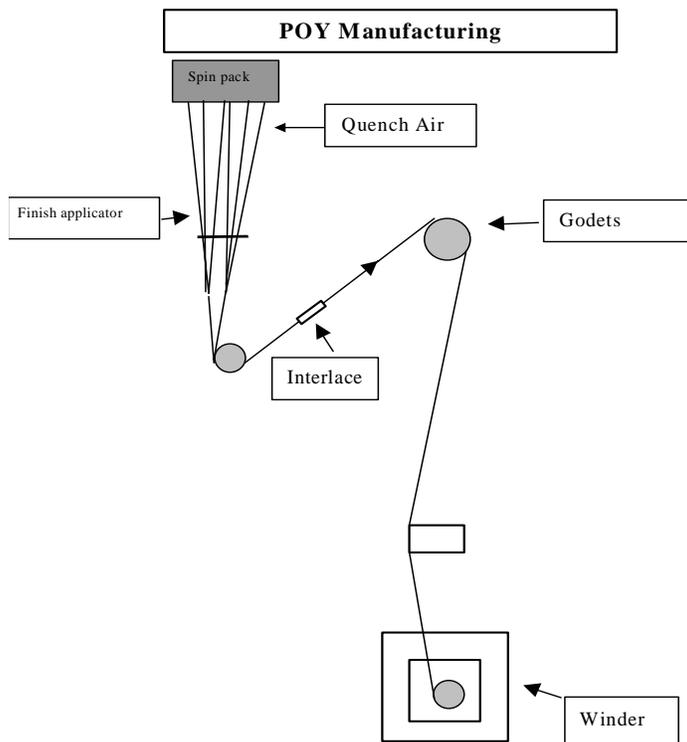
Spin finish should be selected based on planned downstream processing. Goulston Technologies, Inc or Takemoto Oil and Fat Company LTD can recommend and provide finishes for PLA that have been proven for a variety of applications. Depending on subsequent processing and finish types, application levels range from 0.4% up to 1% finish on fiber. In general, PLA requires higher finish levels (~1.5 to 2X) than that for PET fiber for false twist texturing applications.

Spin finish should be applied at a distance ranging from 1.5 (for higher dpf) up to 0.6 meters (lower dpf fibers) from the face of the spinneret. Levels should be adjusted with initial product run to maintain optimum fiber uniformity and desired threadline tensions.

5.9 Winding

Speeds: PLA is capable of being spun at a wide range of spinning speeds. The typical spinning speed for POY is in the range of 2600-3800 m/min. Orientation development (elongation reduction / tenacity increase) with increased spinning speed is similar to polyester .

Winder: PLA fiber can be wound on conventional PET/Nylon POY bobbins using standard winder configurations. Overfeed adjustments can be made depending on roll configuration and target tensions for optimum package build.



Safety and Handling Considerations

Material Safety Data (MSD) sheets for PLA polymers 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 PLA polymers; 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

PLA polymers 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 170°C), 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 recommended 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 PLA polymers. Use gloves with insulation for thermal protection when exposure to the melt is localized.

Combustibility

PLA polymers 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.

Product Stewardship

NatureWorks LLC has a fundamental duty to all those that make and use our products, and for the environment in which we live. This duty is the basis for our Product Stewardship philosophy, by which we assess the health and environmental information on our products and their intended use, then take appropriate steps to protect the environment and the health of our employees and the public.

Customer Notice

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