NatureWorks® PLA Spunbond Process Guide

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 will result in limited decomposition. Lactide, a non-hazardous gaseous irritant, can be produced during PLA melt processing. Appropriate air testing should be conducted 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 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

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

Boxed PLA resins should be kept in sealed containers until ready for use. 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

Corrosion resistant materials are recommended for vessels used to dry polylactide polymers.

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 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.

<table>
<thead>
<tr>
<th>Part</th>
<th>Steel Type</th>
</tr>
</thead>
<tbody>
<tr>
<td>Melt pumps and bearings</td>
<td>SUS440B</td>
</tr>
<tr>
<td>Pump blocks</td>
<td>SUS631</td>
</tr>
<tr>
<td>Transfer lines and spin beam</td>
<td>SUS440C</td>
</tr>
</tbody>
</table>

4.0 Line Preparation

Prior to introducing PLA into any melt spinning system, the system should be properly purged to prevent foreign polymer contamination and associated spinning problems.
4.1 Start-Up/Purging

**Note:** Be sure to clean feed hopper and all pellet storage/transport piping to be completely free of Polypropylene/Polyester before adding PLA.

Starting with Cleaned Extruder and Spin Pack
1. Set temperatures to those listed for PLA and begin running.

Starting with Polypropylene in Extruder
1. Use 10-15 MI polypropylene to purge extruder with spinneret out until all evidence of contamination is cleared from system.
2. Stop feeding polypropylene and run extruder until flow out drops off.
3. Start feeding PLA.
4. Change temperatures to those listed for PLA.
5. Install clean spin pack and continue to run PLA.

Starting with Polyester in Extruder
1. It is best to purge without the spin pack installed.
2. Keep extruder running at normal polyester running temperatures.
3. Transition to a low MI polypropylene (< 1 MI, if available).
4. Purge with polypropylene until all evidence of Polyester is gone.
5. Transition to a 5-15 MI polypropylene.
6. Change to polypropylene extrusion conditions.
7. Let extrusion system run empty.
8. Start purging PLA.
9. Change temperatures to those listed below for PLA.
10. Once system is purged install spin pack and continue running PLA.

**PLA Purging Procedure**

Purge should last for at least 3x average residence time without spinneret in place. Allow system to empty before introducing PLA.

After pack has been installed, allow PLA to purge through packs at reduced throughput for at least 15 minutes or until all capillary extrudate is uniform and free of bubbles, contaminants and flow anomalies. Begin spinning only after the capillary flow performance has been examined and found to be uniform and free of contamination.

Once spin pack extrudate appears clean and stable, increase pump speeds to desired rates, allow at least another 15+ minutes for thermal stability to reach acceptable levels, then introduce filaments to attenuation.

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.
2. Purge for at least 3x average residence time or until PP stream is clean PP without spinneret in place.

5.0 Equipment & Process

5.1 Dryer:

PLA resin can be successfully dried using most standard drying systems. 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. Air or nitrogen based desiccant drying systems can be used at the recommended temperatures. Typical desiccant drying system conditions follow:

<table>
<thead>
<tr>
<th>Drying Parameter</th>
<th>Typical Settings</th>
</tr>
</thead>
<tbody>
<tr>
<td>Residence Time (hours)</td>
<td>Minimum 4 hrs</td>
</tr>
<tr>
<td>Air Temperature (°C)/(°F)</td>
<td>80 C / 176 F</td>
</tr>
<tr>
<td>Air Dew Point (°C)</td>
<td>-40</td>
</tr>
<tr>
<td>Air Flow Rate (ft³/min/lb resin)</td>
<td>&gt; 0.25</td>
</tr>
</tbody>
</table>

Drying Curves for Amorphous and Crystalline PLA Pellets

Note: Starting point on drying curve is 2500 PPM and is only to be used as a reference. Actual equilibrium moisture level will vary.

Caution:

Note: 1 - Typical desiccant dryer regeneration temperatures exceed the melting point of PLA resins. To prevent 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 when 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 or PET Single-Screw extruder, 24 to 32:1 L/D with feed-throat cooling. Extrudate temperature uniformity is desirable if means for monitoring and adjusting are available. If not available, a mixing tip is recommended along with static mixers in the transfer line to promote temperature uniformity as well as to optimize additive dispersion and melt polymer homogeneity.

Extrusion Conditions:

| Feed throat | 25 |
| Zone 1      | 200 |
| Zone 2      | 220 |
| Zone 3      | 230 |
| Zone 4      | 235 |
| Melt pump   | 235 |
| Spin head   | 235 |

Note 1: Temperatures are only starting points and may need to be optimized based on individual system instrumentation and throughputs.

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

5.3 Pack Configuration & Filtration:

PLA resin will typically be provided pre-filtered to a level of 20 microns. The pack should be designed for minimum volume and maximum flow uniformity. The following pack makeup is recommended:

Screens – cascade configuration with appropriate support screens is recommended with finest filtration level of 325 mesh.

Spinninget – Recommended capillary dimensions range from 0.2-0.35 mm diameter, typically with a 2 to 4:1 L/D ratio. The following guide can be used to estimate spinninget requirements based on spun product dpf:

![Spinninget Capillary Sizing](image)

5.4 Quench, Fume Exhaust & Filament Attenuation

As with most melt spinning processes, uniform quenching is critical to providing a stable, uniform filament 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 spinninget for most spunbond systems. 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. For systems with independently
controlled vertical zones, quench air velocity in zones more distant from the spinneret can reach values of 1 meter per second. Depending on filament density, layout and machine design, these conditions (quench delay, velocity and temperature) will vary and should be adjusted within the recommended ranges to obtain acceptable filament curtain stability, draw tension uniformity and denier uniformity values.

A monomer exhaust system, or fume removal vent should be positioned near the spinneret. In the case of single-sided quench, the vent should be position opposed to the quench. Depending on the distance from the first row of filaments, the exhaust should be operated to provide an intake velocity at the port of ~0.50 – 1.0 meters per second. Exhaust airflow velocity should be made uniform across the width of the spin beam.

Filament drawing can be achieved by any number of known processes. Most processes involve some form of air-driven suction device. Mechanical, airgun and slot attenuation systems must provide sufficient spinline stress to induce crystallization. Filament structure and properties change dramatically with filament velocity. A balance between the enhancing effect of molecular orientation and the stabilizing effect of crystalline development controls shrinkage. Generally, filament speeds between 3000 to 5000 meters per minute are required to achieve sufficient crystallization to impart thermal stability to the as-spun filaments.

5.4.1 Air Attenuation Device

![High Speed Air Attenuation (Airgun)](image)

- Spin pack
- Exhaust
- Quench Air
- Filament Attenuation & Distribution

Version 1.0 3/15/05
5.4.2 Mechanical-Draw Spunbond Process

5.4.3 High Speed Slot-Attenuation Spunbond Process
5.5 Web Forming, Static Management & Bonding

Web forming or filament distribution can be achieved by any number of known or proprietary systems used for polypropylene, polyester or nylon spunbond. An experimental approach to determine optimums for each system is recommended.

Special precautions must be taken to manage static electricity. In addition to static bars and humidity control, thorough grounding of attenuation, webforming, press rolls, conveying belt and bonding components is recommended.

Bonding can be achieved thermally using calender, air knife or through-air bonding systems. Optimum bond temperatures will depend on the level of crystallization, filament diameter, line speeds and fabric weights. Again, an experimental approach is recommended to determine optimums for each system.

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.

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Melt Density vs Melt Temperature

1.065 1.070 1.075 1.080 1.085 1.090
215 220 225 230 235 240 245 250 255

Temperature Degrees C

gms/cc
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5.7 Additives

Delusterants such as TiO2 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. Particle size and size distribution must be suitable for high speed, low denier spinning. Quality control and specification should be based on dispersed particle size and particle-size distribution; and should include a pressure drop specification suitable to the denier of the product to be produced.

5.8 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® J / Therminol® LT 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 (230-240°C or 446-464°F).

Operation of the HTM system at a temperature as close as possible to the actual melt temperature (235±5°C or 455±9°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.
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 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 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.

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