

## NatureWorks® PLA Meltblown 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/m<sup>3</sup> are maintained. The use of process area point source remediation measures such as monomer fume hoods or exhausts near the die & forming areas 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 dry inert gas such as 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, manifold, or dies 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.

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

### ***4.0 Line Preparation***

Prior to introducing PLA into any meltblown 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 Die**

1. Set temperatures to those listed for PLA and begin running.

##### **Starting with Polypropylene in Extruder**

1. Use lower MFI polypropylene to purge extruder with die 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 die, start airflows before starting polymer flow.
6. After airflow has started, continue to run PLA

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

After die has been installed, air must be started prior to starting polymer flow. Allow PLA to purge through die at reduced throughput for at least 15 minutes or until all capillary extrudate is uniform and appears free of contaminants and flow anomalies. Increase polymer and airflows only after the capillary flow performance has been examined and found to be uniform and free of contamination.

Once die 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 increase airflows to desired levels.

Purge all PLA from your extrusion system immediately after completing a production trial or run.

#### **Important Notes:**

1. It is critical that all drying, conveying, and receiving systems be free of all foreign polymer (PP/PE/PET/Nylon) or other contaminants and are thoroughly vacuumed to ensure that dust or polymer contamination is completely removed prior to 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 crosslink.
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 Equipment & Process

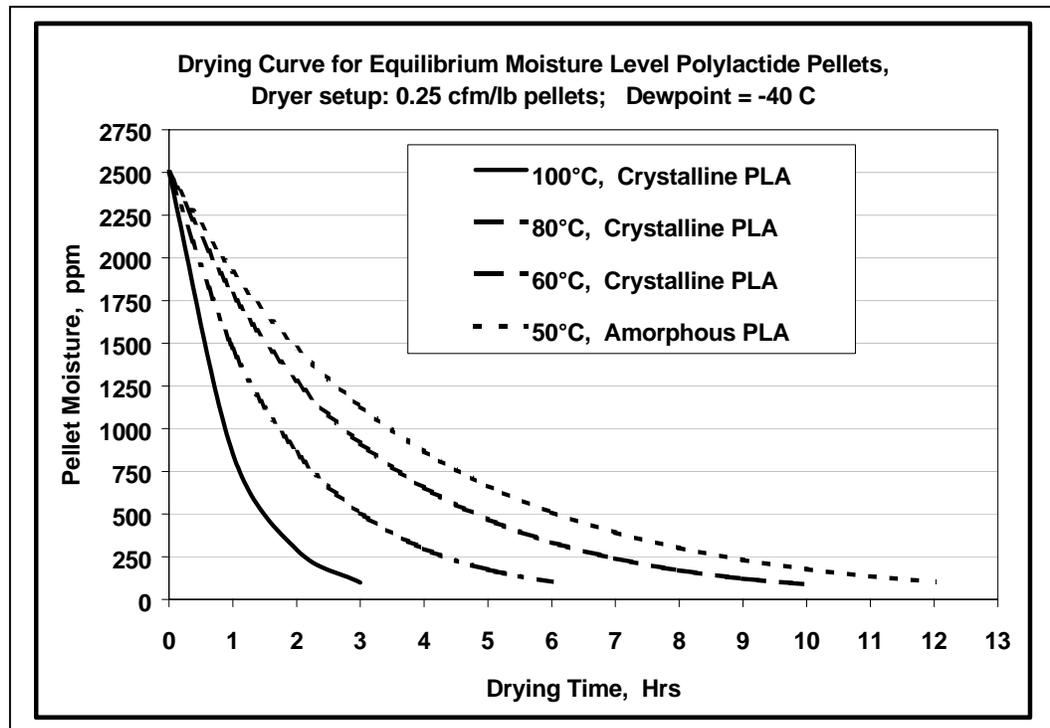
### 5.1 Dryer

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 50ppm is recommended to prevent viscosity degradation. Material is supplied in foil-lined boxes dried to less than 400ppm 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:

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 <sup>3</sup> /min/lb resin)	> 0.25	> 0.25

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 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:

<b>Feed throat</b>	25 °C
<b>Zone 1</b>	200 °C
<b>Zone 2</b>	230 °C
<b>Zone 3</b>	240 °C
<b>Zone 4</b>	245 °C
<b>Melt pump</b>	245 °C
<b>Spin head</b>	245 °C

**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 Filtration

PLA resin will typically be provided pre-filtered to a level of 20 microns. The die assembly should be designed for minimum volume and maximum flow uniformity.

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

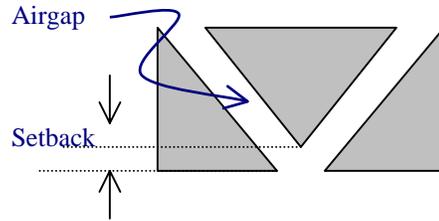
## 5.4 Die Configuration, Air Temperature and Flow, and DCD

Optimal capillary diameter, and capillary density (holes per inch) will depend upon intended applications and will require an empirical optimization approach. Start with existing polypropylene equipment and set-up and adjust as needed. Compared to polypropylene, PLA is relatively Newtonian and may require finer capillary diameters.

Optimal machine settings will depend upon polymer selection and intended applications. An empirical approach to optimization is recommended, starting with existing set-ups typically used for polypropylene. Compared to polypropylene, PLA attenuates differently and solidifies quicker. Therefore, all settings will need to be adjusted for optimal performance – e.g. DCD, Vacuum Airflow, Die Temperature, Die temperature uniformity, Forming Belt Porosity, Vacuum airflow, Belt Speed and Throughput.

The meltblowing/webforming region of the system should be enclosed and equipped with a monomer exhaust system, or fume removal vent.

Suggested die set-up:



**Die:**

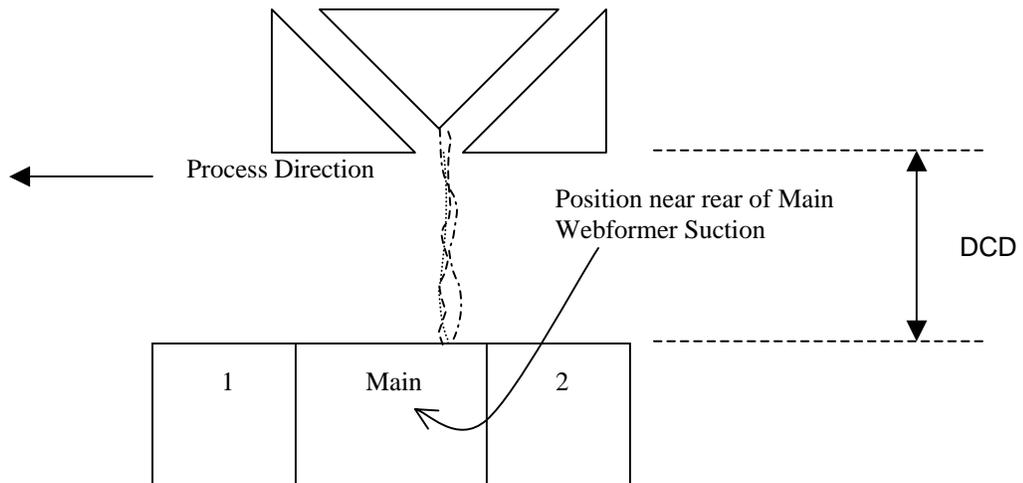
Airgap = 1.00 – 2.00 mm (+/-0.02mm)

Setback = 0.75 – 1.25mm (+/-0.02mm)

Holes/meter = 1000 to 1200 (for fine filtration); 2000+ for other

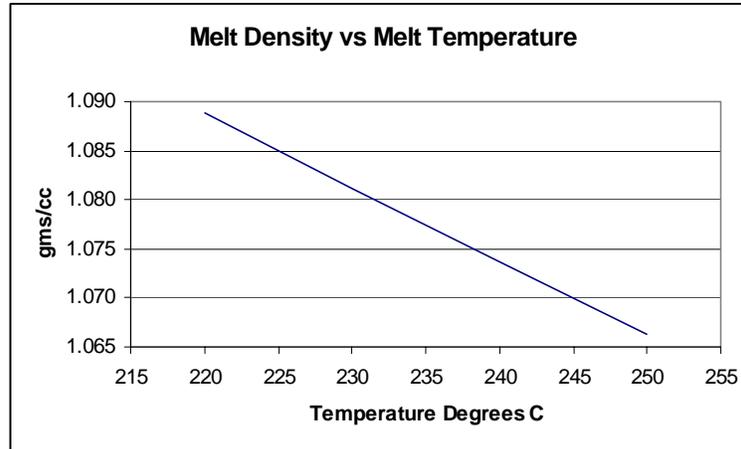
Capillary Diameter. = 0.25 to 35mm

Airgap tolerances need to be kept very tight to avoid quality problems, especially for higher quality filtration substrates.



## 5.5 Melt Density

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.



## 5.6 Additives

Delusterants such as  $\text{TiO}_2$  are best added as a masterbatch at 15-30 wt% in PLA resins. The preferred method for additive addition of dried masterbatch is into the feed throat of the running extruder. Particle size and size distribution must be suitable for meltblown applications. 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 grade of meltblown to be produced.

## 5.7 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®<sup>i</sup> J / Therminol®<sup>ii</sup> 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 (230-245°C or 446-473°F).

#### 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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