Sheet Extrusion Processing Guide

This information is intended for use only as a guide for the manufacture of PLA sheet ranging between 8 – 50 mils (0.2 – 1.3 mm). PLA is generally not recommended for producing sheet less than 8 mils (0.2 mm) and the production of heavy gauge sheet requires special consideration to heat transfer and sheet pinning systems. Because extrusion of PLA sheet is complex, an experimental approach may be required to achieve optimal 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 may 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 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 the die are typically recommended.

Molten PLA 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, collecting molten patties and emptying purge containers. A molten web of PLA will not release cleanly from a gloved hand so use caution when grabbing any stream or patty of PLA.

PLA is considered non-hazardous according to DOT (US Department of Transportation) shipping regulations. When handling PLA 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 PLA grade being processed.

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

2.0 Pellet Storage and Blending Recommendation

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 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

NatureWorks 2002D is the recommended PLA resin grade for sheet production intended for thermoforming applications. Typical properties of 2002D are shown in the following table.

Typical PLA Resin Properties

Resin Property	Nominal Value
RV	3.9 – 4.1
Melting Point, °F (°C)	300 – 320 (150 – 160)
Glass Transition Temperature, °F (°C)	136 (58)
Crystallization Temperature, °F (°C)	212 – 248 (100 – 120)

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, PLA should not be left in the extruder, polymer filter, polymer transfer lines, dies or any other part of the extrusion system at PLA melt temperatures or higher 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-resistance material should be used for screw elements, vacuum vent lines, and trap components. Contact your NatureWorks LLC Technical representative, for additional recommendations on proper handling of lactide gasses and trapped lactide.

5.0 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 2.0).

In-line drying is essential for PLA resins. It is recommended that PLA 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 positively affect the stability of PLA and will increase the viscosity stability over time at elevated temperatures. Processes that have unusually long residence times or result in melt temperatures greater than 465°F or 240°C should only extrude PLA 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 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 PLA drying conditions are shown in the following table.

Typical PLA Raw Material Drying Conditions

	Typical Settings	
Drying Parameter	**Dry pellets received in barrier liner**	**Pellets with 2000 ppm moisture**
Residence Time (hours)	2	4
Air Temperature °F (°C)	194 (90)	194 (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 PLA 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.

6.0 Melt Extrusion

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

6.1 PLA Purging Procedure

Following PP or PS in your system

1. Introduce a high melt flow PP (5 - 8 MFR) or PS (6 –10) first at normal PP or PS conditions, then reduce temperatures to recommended PLA temperatures. Purge for at least 7x average residence time. Let system empty as much as possible.
2. Turn off extruder and completely clean all hoppers, elbow, slide gates, dryers, hopper loaders bins, hopper loader filters and material conveying lines of residual PP. Load PLA into material handling system.
3. Transition to PLA and purge, again for a minimum 7x the average residence time. Change screen pack when it becomes obvious that primarily PLA is exiting the die.
4. At the completion of a trial run, purge all PLA from the extrusion system, using a moderate to low melt index PP or PS.

Following PET, PA, or HDPE in your system

1. It is critical to clean the material handling system of PET, nylon and high molecular weight HDPE to assure 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 PLA are temperature incompatible, so the transition resin is one that can be processed at the high temperatures of PET and the low temperatures of PLA. 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 PLA operating temperatures. PLA 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 PLA 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 PLA 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 PLA 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 PLA into material handling system.
7. Transition to PLA and purge, again for a minimum 7x the average residence time. Change screen pack when it becomes obvious that primarily PLA 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 PLA from the extrusion system, using a moderate to low melt index resin that processes well between 400 and 450°F (205-230°C), initially using the PLA temperature settings and extrusion rates.

Notes:

1. It is critical that all drying and conveying/receiving systems be free of all PET and vacuumed to ensure that there is no remaining polymer dust, before adding PLA. PET will not melt at PLA operating temperatures and will block screens, if it is present in the system.

6.2 Extrusion

A general-purpose single-screw extruder, 24 to 36:1 L/D with feed-throat cooling is acceptable for processing PLA. Generally, shorter extruders will result in a lower melt temperature and less sag at the exit of the die. 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 sheet profile consistency. The following table shows a typical melt profile for PLA.

Typical PLA 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 PLA, a reverse temperature profile has proven to be beneficial to reduce melt temperature and reduce extruder motor load.

Note 2: Temperatures are only starting points and may need to be altered. Target PLA melt temperatures (after melt pump) should be in the range of 210±10°C (410±20°F).PLA 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 PLA resins and controlled dosing the required amount of dried masterbatch into the feed throat of the running extruder. Some potential additives are inappropriate for extrusion with PLA because they are hygroscopic or hydrated salts (e.g. calcium carbonate) that would lead to severe PLA molecular weight degradation and property loss.

6.4 Filtration

Standard screen pack configurations are recommended to protect melt pumps and dies from incidental contamination that may occur during material handling. Screen mesh sizes of 40 - 125 mesh are generally sufficient. Finer filtration leads to excessive shear-heating with PLA and should be avoided.

7.0 Sheet Casting

Most conventional sheet dies have successfully been used to extrude PLA sheet. External deckles should be avoided if possible since there is the possibility of resin degradation behind the deckles. This will lead to a constant supply of low Mw resin at the edge and may result in edge instability. The die gap should be set at approximately 10% greater thickness, or alternatively 1 to 2 mils more (25-50 microns more) than the desired sheet thickness. It is very important to establish a flat extrudate from a well adjusted die gap when pneumatic nips are used at the casting and polishing roll stand. A hydraulic nip will be better suited to flatten the web (resulting from an inconsistent die gap across the die width) than will a pneumatic nip because the pressure required to exert flatness becomes greater than a pneumatic nip is capable of especially with lighter gauges of sheet (i.e., 10-15 mils or 250 to 375 microns).

Sheet forming is generally accomplished on a 3-roll stack. Vertical, inclined and horizontal roll stacks have all been used. Since PLA has lower melt strength than conventional plastics, there is a slight advantage to inclined and horizontal roll stacks particularly at very high extrusion outputs. The initial roll gap should be approximately 1-inch (25-cm) lower than the die gap to accommodate the slight droop that is common with PLA. The molten PLA web should contact the lower roll of the initial roll gap as close as possible to the gap (nip point). This will reduce the possibility of air entrapment between the sheet and the roll which will result sheet defects. Hydraulic roll stands are preferred to pneumatic since high nip pressures are possible. PLA tends to solidify quickly. Approximately 800 – 900 pounds/linear inch of die (PLI) are required to prevent floating of the rolls which would lead to an uneven polish. Pneumatic nips can be used but uniform die flow is required. Also, the pneumatic gap needs to be set evenly at the desired final sheet thickness. In either case, PLA does not run well with a large bank at the gap.

Temperature profiles vary depending upon the output, melt temperature and roll diameters but in general, good starting points for the roll control set point are:

Top roll	80 F / 25 C
Middle roll	100 F / 38 C
Bottom roll	120 F / 50 C

Excessive buildup of residual lactide can occur on the top and middle roll if there is not intimate contact between the molten web and two rolls. Both gap management (adjust the die gap for uniformity across the die and adjust the nip gap) and low roll temperatures can contribute to this. Of course, a too high a temperature will result in the web sticking to the rolls and give poor quality sheet.

Because of PLA's inherent lower melt strength, the die should be positioned as close to the entrance nip as possible and preferably slightly higher than the nip. This will minimize any early contact of the web onto the middle chill roll that will result in air entrapment that results in small air bubbles and subsequent visual defects in the sheet. Ideally, the web should contact the middle roll just as the web is entering the nip of the middle and top roll. The best quality sheet is achieved when there are no or very minimal rolling banks at the top nip of the roll stand.

The thermal properties of PLA, sheet thickness, line speed, and the heat transfer characteristics of the rolls will dictate the maximum output rate of a particular roll stack. PLA pellets and sheet have the following thermal properties:

Glass Transition Temperature	136°F (58°C)	
Thermal Conductivity, BTU/ ft-hr-°F (cal / cm-sec-°C)	Amorphous Crystalline	0.075 (3.1×10^{-4}) 0.11 (4.5×10^{-4})
Specific Heat, BTU/ lb °F (cal / g °C)	Below Tg Above Tg	0.29 (0.29) 0.51 (0.51)

When making thinner gauge sheet (less than 15 mils or 0.38 mm), hydraulic nips are required to achieve a uniform polish on the sheet. Pressures in the vicinity of 900 pounds / linear inch (160 kg / linear cm) of die are necessary to overcome the viscosity of the rapidly cooling polymer.

8.0 Web Handling and Slitting

The stiffness of PLA is similar to PS and quite higher than either PP or even PET. Be careful of sharp edges particularly on thick cast sheet. Heavy leather gloves should be worn to minimize the risk of cuts. The material also has high yield strength and good tension control between unit operations is imperative. Edge trim is easily slit with rotary shear knives. In general, razor knives give unacceptable performance, yielding a rough edge and numerous web breaks. If razor knives are used, the cutting bed should be supported and the sheet should be slightly warm (about 125°F or 50°C).

9.0 Regrind

PLA regrind from edge trim and skeleton reclaim can be added back into the process provided that the regrind is dry. A maximum of 40% amorphous regrind is recommended to ensure smooth extrusion operation. In addition, screw cooling should be used through the first two flights of the transition zone (in single screw extrusion) when extruding amorphous regrind to eliminate the possibility of material sticking to the screw and causing a screw blockage. Amorphous regrind can be reincorporated as relatively dry material if the time period between exudation from the die as sheet and input of the ground sheet into the extruder throat is just a few minutes. Amorphous regrind that has absorbed 2000 ppm moisture from the atmosphere will require drying times of about 12 hours at 115°F (45°C) to approach 250 to 400 ppm moisture content. In warm humid climates, the dryer may not be capable of drying the flake to 250 ppm at these low temperature settings necessary to avoid agglomeration.

Twin-screw extrusion systems typically are less prone to sticking during extrusion and higher levels of amorphous recycle/regrind.

Crystallized regrind has been added back to the process up to 100% with no loss of either process control or sheet properties. Crystallizers should be operated at 100 C (212 F) and flake bed should be agitated sufficiently to prevent blocking. As with all crystallizers, the unit should be seeded with previously crystallized pellets or flake and allowed to come to steady state before amorphous material is fed into the unit.

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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 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 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.

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