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PROCESSING GUIDE PARTIALLY ORIENTED YARN EXTRUSION OF PLA Interested in solutions for bioplastics? Please contact us at www.totalenergies-corbion.com

#### INTRODUCTION

This processing guide describes the extrusion of Luminy<sup>®</sup> PLA into Partially Oriented Yarn (POY). For this application it is recommended to use Luminy<sup>®</sup> L130 or alternatively Luminy<sup>®</sup> LX530. Typically, POY, after resting for 8-48 hours, is drawn further into draw-textured yarns.

As melt spinning is a complicated process, the information that is given in this processing guide only serves as a starting point. Optimization of the process is advisable to find the optimal process conditions.

#### **STORAGE CONDITIONS**

It is recommended to store PLA polymers in its closed, original moisture-barrier packaging at temperatures below 50°C. Storage in direct sunlight should be avoided. The supplied PLA pellets are typically semi-crystalline.

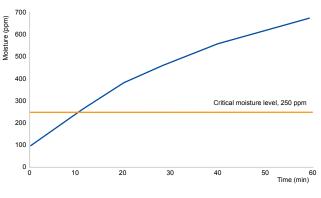
#### **DRYING PLA**

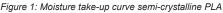
Luminy<sup>®</sup> PLA resins are supplied in sealed aluminum-lined moisture-barrier packaging with a maximum moisture content of 400 ppm. It is recommended to reduce the moisture content before melt processing to a level less than 250 ppm and preferably less than 100 ppm. Moisture causes hydrolysis of the PLA homopolymer during melt processing, resulting in reduced mechanical performance in the final part.

Luminy<sup>®</sup> PLA resins can be dried using most conventional drying systems. The preferred method to dry PLA is by using a desiccant hot air dryer system. Another option is to use a vacuum drying oven. It is highly recommended to check the actual moisture content after drying, for which the Karl-Fischer or Brabender Aquatrac methods can be used. In case additives are used, it is also recommended to check the moisture content of the additives and dry them if necessary.

The dried PLA should be processed as soon as possible after drying and preferably under an inert (Nitrogen) atmosphere to prevent moisture uptake. Starting at 100ppm moisture content, the critical level of 250ppm is already reached after 15 minutes of exposure to atmospheric conditions (Figure 1).

The packaging should be kept sealed before usage and any unused material should be resealed immediately. It is recommended to have a closed system from the dryer into the feeder, a dryer installed on top of the feeder, or to apply a dry nitrogen blanket in the feeder and throat of the extruder to prevent moisture uptake. Typical PLA drying conditions using a desiccant hot air dryer are shown in table 1.







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Parameter	Amorphous standard PLA	Pre-crystallized standard PLA (Luminy <sup>®</sup> LX175)	Semi-crystalline high heat PLA (Luminy <sup>®</sup> L105, L130, LX530
Drying time	24 hours	4-6 hours	and L175) 4-6 hours
Air temperature	40°C	85°C	100°C
Air dew point	< -40°C	< -40°C	< -40°C

Table 1: Typical PLA drying conditions

### TYPICAL PLA RESIN PROPERTIES

		Luminy <sup>®</sup> L130	Luminy® LX530
Density	g / cm³	1.24	1.24
Optical purity	% isomer	>99% L	98% L
MFI (Flow, 210°C / 2.16 kg)	g / 10 min	23	23
MFI (Flow, 190°C / 2.16 kg)	g / 10 min	10	10
Melting temperature (T <sub>m</sub> )	°C	175	165
Glass transition temperature (T <sub>g</sub> )	°C	60	60

Table 2: Typical properties of Luminy® PLA grades suitable for extrusion of POY

#### **START-UP AND SHUTDOWN**

Before introducing Luminy<sup>®</sup> PLA, the melt spinning equipment needs to be well cleaned and purged to prevent cross contamination. Also, make sure that the feeding and blending equipment in the material preparation steps (before the materials and additives enter the extruder) is extensively cleaned and that they are free of dust and contamination. The purging procedures below are recommended for removing other polymers before processing PLA. To prevent starting up the machine with non-molten material, the temperature range of the machine should be set to the processing temperature of the previously used polymer or that of PLA, whichever has the highest processing temperature, therefore a different guideline applies for PP as compared to PET or nylon.

#### Transition to PLA, following polypropylene in the melt spinning system:

- 1. Without the spinneret being inserted, purge the system at regular PLA processing temperatures, with a high melt index PP for at least three times the residence time. Empty the system.
- 2. Change to PLA and purge, as in step 1, the system for at least three times the residence time.
- 3. Put the preheated spinneret in place. Wait until temperatures are stabilized.
- 4. Purge with PLA. Check that the processed material is flowing homogeneously out of the capillaries and that it is free of contamination before starting production.

#### Transition to PLA, following other polymers (such as PET or nylon) in the melt spinning system:

- 1. Without the spinneret being inserted, purge the system at regular PET/nylon processing temperatures, with a low melt index PP for at least three times the residence time. Empty the system.
- 2. Adjust temperatures to regular PLA processing temperatures and purge with a high melt index PP for at least three times the residence time. Empty the system.
- 3. Change to PLA and purge, as in step 1 and 2, the system for at least three times the residence time.
- 4. Put the preheated spinneret in place. Wait until temperatures are stabilized.
- 5. Purge with PLA. Check that the processed material is flowing homogeneously out of the capillaries and that it is free of contamination before starting production.

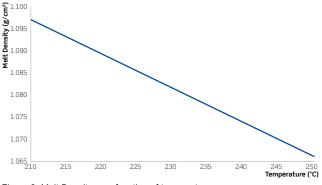


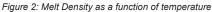
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At the completion of the run it is recommended to purge the system again by using a PP or purging compound to clean the machine from remaining PLA material for at least 5 times the average residence time. Check the recommendations of the supplier of the purging material for the right conditions.

After completion of the run, PLA must be removed from the whole system. PLA can degrade over time into lactic acid causing corrosion of the equipment.





#### MELT DENSITY FOR PROCESS CALCULATIONS

The melt density of PLA at the regular extrusion temperature of 235°C is approximately 1.077 g/cm<sup>3</sup> or can be approached for various extrusion temperatures by following the formula (also depicted in Figure 2) as stated by Witzke (1997):

$$\rho_{PLA,melt}\left(T[^{\circ}C]\right)\left[\frac{g}{cm^{3}}\right] = \frac{1.1452}{1+7.4\times10^{-4}\times(T[^{\circ}C]-150)}\left[\frac{g}{cm^{3}}\right]$$

#### **FILTRATION**

For POY yarns, the use of sintered metal powder filter packs (or comparable loose media filters), having particle size in the range of 200-360  $\mu$ m, is recommended. For example: a gradually changing loose media filter with particle size from 355 to 250  $\mu$ m for the production of a 6 dpf POY could be used.

Likewise, nonwoven metal fiber filter media can be used for which the aperture size of the finest layer is smaller than 20 µm, though very fine dpf might require even smaller aperture size.

#### **SPIN FINISH APPLICATION**

It is recommended to apply spin finish at ca. 1 m distance from the spinneret at a rate which yields 0.5-1% oil pick-up. The application distance is to be adjusted depending on spinline tension and linear density of the yarns, as a shorter distance is required for fine dpf, whilst a larger distance is required for coarser dpf.

#### MELT SPINNING OF PARTIALLY ORIENTED YARNS (POY)

Melt spinning of PLA POY can be done on conventional processing equipment with a (general purpose) single screw extruder having an L/D ratio of 24:1 – 30:1. The recommended processing temperatures are shown in Table 3. Note that (local) temperatures exceeding 250°C should always be avoided to prevent thermal degradation of PLA.

Depending on the targeted linear density, throughput is regularly set in the range of 0.5-4.0 g/min per spin hole. Recommended spin hole capillaries have a diameter of 0.2-0.4 mm and an L/D ratio of 2-4 for achieving linear densities of 2-10 dpf.

Quench air should be applied evenly. Common settings, which should be adapted to linear density and machine design, are a quench air temperature of 20-26°C, quench air velocity of 0.3-0.6 m/s and a quench air delay of 25-75 mm.



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Yarn take-up speed varies between 2500 and 3500 m/min for POY. Additionally, the velocity of the relaxation godet needs to be adjusted depending on the tension.

Parameter	Setting (°C)
Throat	20 - 40
Feed zone	195 - 210
Compression zone	210 - 225
Metering zone	220 - 230
Melt pump	230 - 235
Spin pack assembly	$235\pm5$

Table 3: Recommended processing temperatures for melt spinning POY using Luminy® PLA L130

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