Processing Guide
Extrusion thermoforming PLA for high heat applications

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INTRODUCTION

This processing guide outlines the process of extruding and thermoforming PLA for high-heat applications like drinking cups and coffee capsules.

Polylactic Acid (PLA) is a GMO-free, biobased thermoplastic polymer derived from annually renewable resources. It is certified as industrially compostable under the EN 13432 and ASTM D6400 standards. Luminy® PLA can be processed using conventional extrusion and thermoforming technologies to produce thermoformed products.

STORAGE CONDITIONS

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

PLA RESIN PROPERTIES

Thermoforming heat-resistant articles involves crystallization of the PLA. High-heat PLA compounds, based on Luminy® PLLA resin and nucleating additives like Luminy® PDLA[1] and talc, enhance this crystallization process. Additional mineral fillers can be used to enhance the forming process and product properties. High-heat PLA compounds are typically made on a twin-screw extruder. TotalEnergies Corbion has developed specific compounds for these applications, titled Compound D and Compound E. Table 1 shows the typical properties of these compounds.

[1]PDLA has no FDA food contact approval

PLA grade		Compound D	Compound E
Measure	Unit	value	value
Density	g/cm3	1.39	1.34
Clarity		no	no
MFI, 210°C/2,16kg	g/10min	5-10	5-10
Glass transition temperature	°C	60	60
Melting temperature	°C	175	175
Tensile modulus	(MPa)	5500	5400
Tensile strength	(MPa)	60	60
Elongating at break	(%)	<5	<5
HDT/B (crystalline)	(°C)	120	120
Charpy notched impact, 23°C	(kJ/m2)	2	2

Table 1: Typical resin properties of a high heat thermoforming compound.

The properties of the high-heat PLA compound can be further tailored by adding other additives like impact modifiers. Your local representative can provide more information regarding formulations of high-heat Luminy® PLA compounds.

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Additives

PLA-based additive masterbatches can be dry blended with PLA high-heat compounds to enhance the extrusion thermoforming process or modify the properties of the final products. Table 2 provides an overview of potential additives for use with PLA and their suppliers.

Type	Functionality	Recommended loading	Suppliers
slip / anti-block	de-nesting	1-5%	Sukano Plastikakritis Granula Avient
Impact modifier	toughness improvement	1-5%	Sukano Plastikakritis Granula Avient
color	color	1-5%	Plastikakritis Granula Avient

Table 2: Masterbatches for extrusion thermoforming of PLA

The general process and processing temperatures of extrusion thermoforming PLA for high-heat applications are shown in Figure 1.
The process can be carried out in two ways:
1. Inline, where the article is thermoformed immediately after sheet extrusion.
2. Offline, where roll stock is extruded, stored, and later thermoformed into the final article.

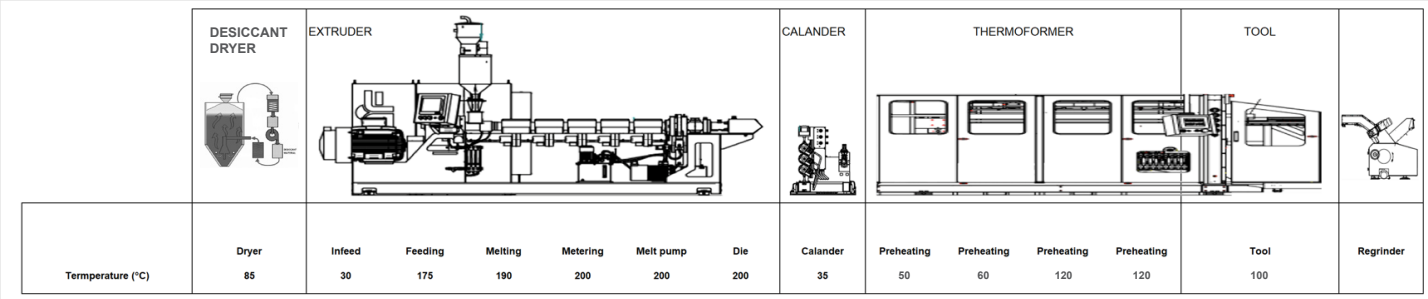

Figure 1: PLA extrusion thermoforming process for high-heat applications.

DRYING

Luminy® PLA resins are supplied in aluminum-lined moisture-barrier packaging with a maximum moisture content of 400 ppm. Before melt processing, it is recommended to reduce the moisture content to below 250 ppm, ideally to 100 ppm. Excess moisture can cause hydrolysis of the PLA during melt processing, leading to reduced processing stability and diminished mechanical performance in the final product.

Luminy® PLA resins can be dried using most conventional drying systems. The preferred method is a desiccant hot air dryer, though a vacuum drying oven is also an option. It is highly recommended to verify the moisture content after drying using methods such as Karl-Fischer or Brabender Aquatrac. If additives are used, their moisture content should also be checked and dried if necessary.

Dried PLA should be processed immediately after drying, ideally in an inert (nitrogen) atmosphere to prevent moisture absorption. Starting at a moisture content of 100 ppm, the critical level of 250 ppm can be reached within just 15 minutes of exposure to atmospheric conditions. (Figure 2).

The packaging should remain sealed until use, and any unused material should be resealed immediately. To prevent moisture uptake, it is recommended to use a closed system from the dryer to the feeder, install the dryer on top of the feeder, or apply a dry nitrogen blanket in the feeder and extruder throat. Typical PLA drying conditions using a desiccant hot air dryer are shown in Table 3.

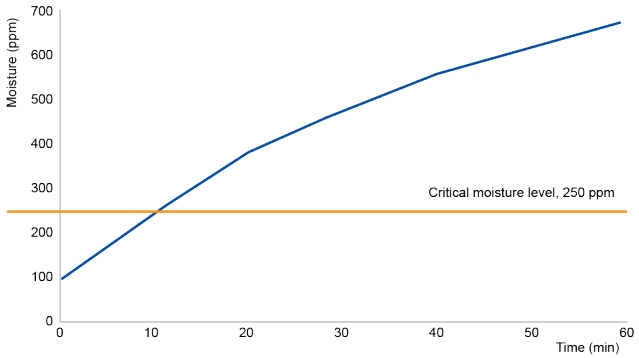

Figure 2: Moisture take-up curve PLA polymer

Parameter	Pre-crystallized PLA compound
Drying time	4-6 hours
Air temperature	85°C
Air dew point	< -40°C

Table 3: Drying conditions for Luminy PLA

EXTRUSION

This section provides a guideline for the successful extrusion of PLA-based sheets in the thickness range of 0.2-1.0mm. Extrusion can be performed on conventional single-screw and twin-screw extruders. Since PLA degradation products are corrosive, it is highly recommended that parts in contact with molten polymer be made of stainless steel. To prevent PLA degradation, avoid leaving PLA in the extruder and auxiliary equipment at high temperatures for extended periods.

Start-up and shutdown

Before introducing Luminy® PLA, ensure the extrusion equipment is thoroughly cleaned and purged to prevent cross-contamination. That includes cleaning the feeding and blending equipment to remove dust and contaminants before the materials and additives enter the extruder. The purging procedures below are recommended for removing other polymers when processing PLA.

1. Check if there are residual polymers from previous runs present in the barrel of the machine. Set the machine temperature to the processing temperature of either the previously used polymer or PLA, whichever is higher, to ensure the machine starts up without non-molten material.

2. Purge based on previous polymer:
   a. Polyolefins (e.g., PS, PP): Purge with a polyolefin of similar MFI to PLA or a purging compound (e.g., ASAclean, Dyna-Purge), then follow with PLA.
   b. PET: If PET's processing temperature is incompatible with PLA, use a low MFI resin (e.g., PETg, PP) at PET's operating temperature.
3. Adjust the barrel temperature to the required temperature for PLA.
4. Check that the processed material is free of contamination before starting production.
5. After completing the run, purge the system with a purging compound to remove residual PLA for five times the average residence time. Refer to the purging compound supplier's recommendations for specific conditions.

After completing the run, remove all PLA from the system, as it can degrade into lactic acid over time, which may cause corrosion of the equipment.

Sheet extrusion.

Sheet extrusion can be done on conventional extrusion equipment, equipped with a general purpose single screw extruder with L/D ratios between 24 and 32. To avoid cross-contamination, the extruder and auxiliary equipment must be purged and cleaned before and after PLA extrusion.

Horizontal, inclined, and vertical roll stacks with highly polished chrome plating have been successfully used. The roll temperatures should be individually controlled and set within the range of 25-50°C.

Air entrapment between the sheet and the chill roll can cause excessive residual lactide buildup on the chill rolls, resulting in poor sheet quality. To minimize this buildup, use techniques such as electrostatic pinning or vacuum, adjust the nip gap, ensure uniform sheet thickness across the width, and optimize chill roll temperatures. However, be cautious: excessively high roll temperatures can cause the film to stick to the chill roll, also leading to poor sheet quality.

Typical extruder conditions for PLA sheet/film extrusion are shown in Table 4.

Parameter	Setting [°C]
Resin intake	20-40
Melt zone	170-190
Mixing & Conveying	190-210
Melt pump	190-210
Die	190-210
Chill roll	25-50

Table 4: Recommended processing temperatures for sheet extrusion of Luminy® PLA.

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Edge trimming:

PLA is a stiff material, so preventive measures should be taken to minimize the risk of cuts from sharp edges for operators. Edge trim is preferably slit with rotary shear knives. The use of razor knives can only be considered if the sheet is locally heated to 40-60°C. Trimming cold sheets with razor knives likely will lead to many web breaks and is therefore not recommended.

Static eliminator:

To prevent dust and contamination, as well as to avoid static shocks to operators, eliminating static electricity is recommended. Effective measures include using a bar-type static eliminator to distribute ionized air over the operation or employing well-grounded anti-static copper tinsel.

ROLL STOCK STORAGE:

Roll stock made from Luminy® PLA resin should be stored at temperatures below 40°C. Temperatures above this can cause roll blocking and, depending on relative humidity, may lead to degradation and loss of physical properties. Pre-drying of roll stock to prevent moisture-related defects is not required.

THERMOFORMING:

This section provides a guideline for successful thermoforming of PLA based articles for high-heat applications like drinking cups and coffee capsules.

Managing crystallinity is crucial for successful thermoforming of high-heat articles. During extrusion and pre-heating, the sheet must crystallize sufficiently to allow for detailed forming. To achieve this, it is highly recommended to use ovens with flexible temperature control, featuring individually controlled longitudinal and transverse heating zones, to heat the sheet to 100-130°C.

The heated sheet must then be formed in a mold set at 80-130°C to complete the crystallization process and produce heat-resistant PLA articles. Key parameters controlling crystallization include the oven temperature profile, mold temperature, and thermoforming speed.

Sheet handling:

It is not necessary to preheat the sheet before entering the heating section.
Luminy® PLA is a relatively brittle material at ambient conditions. Therefore precaution measures should be considered when thermoforming PLA.

- Edge pre-heating of the sheet to a temperature of 60-80°C is recommended to prevent the sheet from cracking when pinned into the transportation chain.
- Good tension control throughout the whole thermoforming process is mandatory. Sudden tension changes can result in web breaks.
- Sharp path radii of the sheet throughout the thermoforming process should be avoided to minimize web breaks.

Sheet heating:

The purpose of preheating heat-resistant PLA differs from that of amorphous polymers. While amorphous polymers are preheated primarily to plasticize the sheet for better forming detail, preheating heat-resistant PLA also facilitates crystallization. This process begins at temperatures above 70°C in the pre-heater.

The temperature profile and time the sheet is exposed to the ovens determine the level of crystallinity developed before entering the mold, which is crucial for both forming and crystallization. If the sheet has too low a crystallinity when it enters the mold, achieving the desired crystallinity will take longer, extending cycle time. Conversely, if the sheet has too high a crystallinity, producing articles with good forming details may be impossible.

To optimize the process, start by setting the heaters to a low temperature to ensure the sheet does not exceed 70°C initially. Quickly raise the temperature from 70°C to 125°C in the last 10 seconds before the sheet enters the mold. Adjusting the heating profile, mold temperature, and thermoforming speed for each product is essential. Additionally, web spreading can help compensate for sagging.

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Sheet forming

Conventional forming technologies, such as vacuum forming or pressure forming can be used to form the sheet into its desired shape. For deeper drawn articles, such as trays and cups, plug assist forming is recommended to achieve optimal material distribution.

The forming of the sheet is typically performed in a temperature-controlled aluminum mold set at a temperature between 80 and 130°C.

The shrinkage of PLA is comparable to other semi-crystalline polymers such as PP.

Trimming:

PLA is a hard and brittle material. It is therefore recommended to trim the part when the sheet is still above its softening temperature of 60°C.

PLA runs on machines equipped with cut-in-place (tilt) molds and on combined forming and cutting machines.
Steel-rule and matched-metal dies can be used for trimming PLA.
Typical thermoforming conditions for PLA sheets are shown in the Table 5.

Parameter	Setting [°C]
Pre-heating	Not required
Sheet temperature	100-130
Mold temperature	80-130

Table 5: Recommended processing temperatures for thermoforming Luminy® PLA.

REGRINDING:

The obtained edge trim and skeleton waste during the extrusion and thermoforming process can be regrinded for reprocessing. Ensure that the regrind system has been cleaned to prevent cross-contamination and that the temperature of the incoming sheet is less than 40°C.

RECYCLING:

PLA edge trim and skeleton waste can be regrinded and recycled into new sheet. Precaution should be taken in the handling of the recycle stream. When PLA is (re)processed at the recommended processing conditions, hydrolysis is the main mechanism for degradation, which will lead to molecular weight loss and consequently loss of mechanical properties. Proper drying of the regrind along with virgin resin is mandatory to minimize the effect of the hydrolytic degradation during melt processing. Sheets containing up to 50% properly handled recycle have been successfully processed into new sheets.

The obtained edge trim and skeleton waste during the extrusion and thermoforming process will be in the amorphous form. The amorphous regrind can either be recrystallized before reprocessing into sheets or used as such.

However, it is recommended to recrystallize the amorphous regrind to prevent agglomeration during mixing with virgin hot dried PLA resin or at the extruder intake section. Crystallization can be performed using conventional crystallization equipment such as infrared rotary drum systems or fixed bed crystallizers equipped with an agitator. The optimum crystallization temperature of PLA lies between 90-110°C and typically within 30 minutes the PLA has achieved a degree of crystallinity losing its stickiness.

Be aware that the Luminy® LX975 and Luminy® LX930 are amorphous PLAs and cannot be crystallized.

Methods for reprocessing either amorphous or re-crystallized regrind is described below.

Reprocessing amorphous PLA regrind:

Similar to virgin PLA resins, amorphous PLA regrind should be processed at a moisture level not higher than 250ppm, and needs to be pre-dried in a desiccant hot air dryer. The drying temperature should not exceed 40°C to prevent agglomeration in the drier. The required drying time will depend on the moisture content of the regrind. If the regrind is exposed for a longer period to ambient conditions, a drying time of at least 24 hours is recommended.

When mixing regrind with other materials, such as virgin resin, ensure that the temperature is less than 50°C to prevent agglomeration.

The extruder intake and 1-3 diameter screw length should be actively cooled to prevent sticking of the regrind to the screw. If sticking to the screw happens, it will lead to surging and ultimately a complete blocking of resin feed into the extruder.

Reprocessing recrystallized PLA regrind:

Similar to virgin PLA resins, recrystallized PLA regrind should be processed at a moisture level not higher than 250ppm, and needs to be pre-dried in a desiccant hot air dryer.

Drying can be performed in a desiccant hot air dryer at 85°C. The required drying time will depend on the moisture content of the regrind. Typical drying times between 4-6 hours are sufficient to reduce the moisture content to less than 250ppm.