Processing Guide
Compounding high heat PLA/PDLA

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PROCESSING GUIDE
COMPOUNDING HIGH HEAT PLA/PDLA

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INTRODUCTION

This processing guide describes the handling and compounding of high heat PLA. Compounding is a process of melt-mixing PLA with polymers, additives, fillers and/or reinforcing materials. Typically, this is done to improve certain properties of the PLA polymer. High heat PLA compounds can be produced by combining PLA and PDLA homopolymers. Compared to standard PLA, these compounds have higher melting points and an increased rate of crystallization. As a result, compounds containing PLA homopolymers are suitable for the production of semi-crystalline parts, which exhibit a higher temperature resistance. The compounding recommendations are also applicable to standard PLA, taking into account that it will not crystallize in further processing and results in amorphous materials with lower HDT/B values.

As compounding is a general processing technology with a lot of possibilities in terms of formulations, the information that is given in this processing guide only serves as a starting point. Optimization of the process is recommended to find the optimal process conditions for the formulation that is used. To validate the compound against customer requirements, testing of compounded PLA is recommended.

STORAGE CONDITIONS

It is recommended to store PLA polymers and compounds 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.

TYPICAL PLA RESIN PROPERTIES

Property	Unit	Typical value			
		Luminy® L105	Luminy® L130	Luminy® L175	Luminy® D070[1]
Density	g/cm^3	1.24	1.24	1.24	1.24
Optical purity	%L-isomer	>99%	>99%	>99%	<1%
MFI (210°C/2.16kg)	g/10 min	70	23	8	>100
Melting temperature	°C	175	175	175	175
Glass transition temperature	°C	60	60	60	60
Tensile modulus	MPa	3500	3500	3500	3500
Tensile strength	MPa	50	50	50	50
Strain at break	%	<5	<5	<5	<5
Impact (Charpy notched, 23°C)	kJ/m^2	<5	<5	<5	<5
HDT-B (amorphous)[2]	°C	60	60	60	N/A
HDT-B (semi-crystalline)[2]	°C	105	105	105	N/A

[1] D070 is only to be used as a nucleating agent.
[2] HDT B, 0.45MPa, flatwise using injection molded test bars. HDT depends on processing conditions. For crystalline resins, formulation included 3 - 7% nucleating agent (D070) and molded at 90 - 100°C tool temperature.

Table 1: Typical properties of TotalEnergies Corbion resin grades suitable for compounding

DRYING PLA

Luminy® PLA resins are supplied in sealed aluminum-lined 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 to 100 ppm. Moisture causes hydrolysis of the PLA homopolymer during melt processing, resulting in reduced mechanical performance in the final part.

Luminy® 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 necessary the 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).

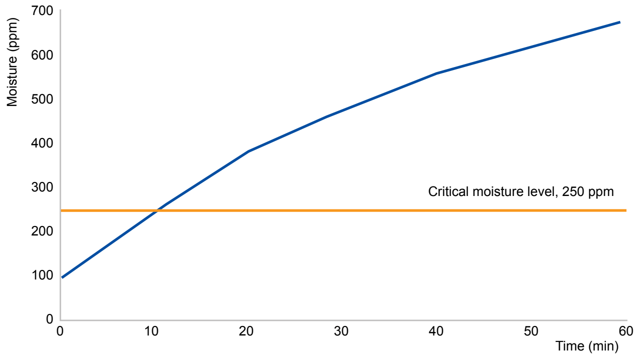

Figure 1: Moisture take-up curve PLA homopolymer

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 2.

Parameter	Amorphous PLA	Pre-crystallized standard PLA (Luminy® LX175)	Crystalline PLA homopolymers (Luminy® L105, L130, L175)
Drying time	24 hours	4-6 hours	4-6 hours
Air temperature	40°C	85°C	100°C
Air dew point	< -40°C	< -40°C	< -40°C

Table 2: Typical PLA drying conditions

START-UP AND SHUTDOWN

Before introducing Luminy® PLA, the 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 when processing PLA.

1. Check if other polymers from previous runs are present in the barrel of the machine. 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 the PLA, whichever has the highest processing temperature.
2. Purge the system with a polyolefin with similar MFI to PLA, or a purging compound (e.g. ASAclean, Dyna-Purge, etc.) followed by purging with the PLA homopolymer.
3. Change the temperature of the extruder to the required temperature for PLA.
4. Purge with PLA homopolymer until it is free of contaminants.
5. Start adding other components (plasticizer, fillers, etc.) and purge for a minimum of 3 times the average residence time.
6. At completion of the run, stop feeding the other components and continue to run the PLA homopolymer for a minimum of 3 times the average residence time.
7. Reset the temperature settings to the recommended purging compound temperature profile.
8. Purge with a purging compound until all PLA and the other components have been removed from the extruder. Check the recommendations of the purging material supplier for the correct 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.

TYPICAL FORMULATION OF HIGH HEAT PLA COMPOUNDS

High heat PLA compounds can be made with a large variety of formulations. A typical formulation for a high heat PLA compound consists of:

- 70-80 % PLA homopolymer
- 3-7% PDLA homopolymer (i.e. Luminy® D070)
- Mineral fillers, like talc or calcium carbonate ($CaCO_3$)
- Crystallization enhancers / plasticizers
- Pigments
- Glass or natural fibers
- Stabilizers

Always check the local regulations and safety instructions before starting the compounding and make sure the formulation complies with local regulations.

EXTRUDER SETUP AND TEMPERATURE PROFILE

Luminy® PLA can be processed on conventional compounding equipment. A co-rotating intermeshing twin screw extruder with a L/D ratio of at least 28:1 is preferred.

A typical extruder set up consists of:

- a throat with the PLA feeders. PLA and PDLA (Luminy® D070) may be added by 2 separate feeders but it can also be added as a dry-blend mixture.
- a side feeder, where the mineral fillers and other non-melting additives should be added into the molten PLA stream. After the side feeder, there should be enough residence time and mixing capacity to ensure homogeneous mixing of these additives throughout the PLA.
- a liquid feeder, where liquid additives should be added. The liquid addition should preferably be installed after a degassing section to prevent evaporation of these additives.

It is recommended to have atmospheric and/or vacuum degassing sections along the barrel to remove volatiles caused by the introduction of additives and/or fillers.

Conventional pelletizing technologies can be used such as strand and underwater pelletizers. A typical extruder setup and temperature profile for compounding PLA compounds can be found in Figure 2 and Table 3. The use of a mild screw design with mixing sections after each addition port is recommended.

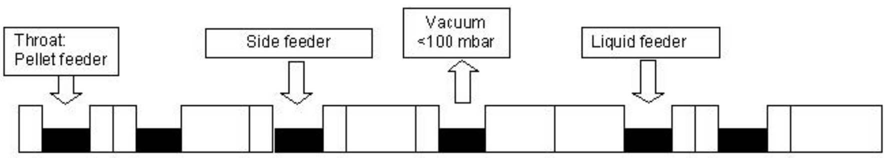

Figure 2: Typical extruder setup for compounding PLA compounds (from left to right)

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Parameter	Unit	Setting for high heat PLA
Feed zone	°C	20-40
Mixing & conveying	°C	190-210
Die head temperature	°C	190-210

Table 3: Typical extruder temperature conditions for compounding high heat PLA.

CRYSTALLIZATION, DRYING AND PACKAGING

The produced PLA compound should be crystallized and dried prior to packaging and/or further use. This prevents degradation and agglomeration of the produced compound during storage and further processing.

First, the material needs to be crystallized. This can be done in batch or continuous crystallization process. It is important that the material is heated above its glass transition temperature for a certain time to allow the PLA molecules to rearrange from the amorphous phase into a semi-crystalline phase. Crystallization speed depends on the compound formulation and crystallization temperature. PLA crystallizes fastest at approximately 100°C and needs – in general and depending on the PLA grade - 0.5 to 4 hours to crystallize. It can be noticed that the pellets start to stick and form agglomerates during the crystallization process. A mechanical force is often needed to break up the agglomerates and make the PLA pellets free flowing again.

Typical crystallization equipment can be vertical silos with horizontal agitators (Figure 3) or horizontal paddle screw heat exchangers (Figure 4).

After crystallization, it is recommended to dry the produced PLA compound to prevent low temperature hydrolysis during storage. For drying instructions, see the prior section in this document on page 2. After drying, it is recommended to pack the crystallized and dried PLA compound using moisture-barrier packaging to avoid moisture take-up during transportation and storage.

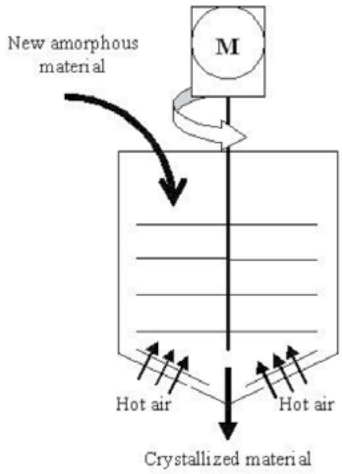

Figure 3: Vertical silo with horizontal agitators

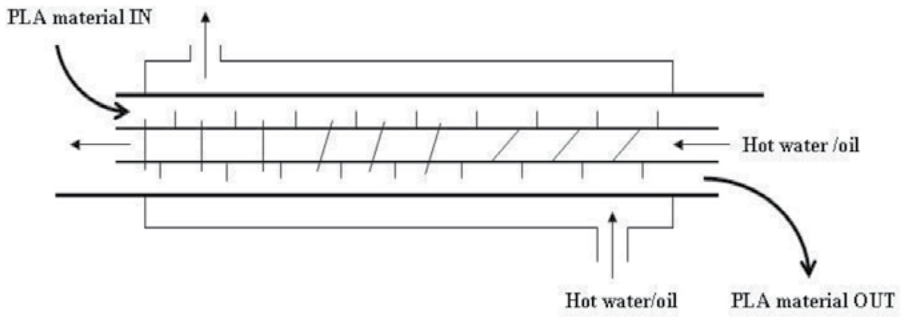

Figure 4: Horizontal paddles screw heat exchanger

FOOD CONTACT

In the European Union, Luminy® PLA polymers are compliant with EU commission regulation 10/2011 of 14 January 2011 (and amendments) on plastic materials and articles intended to come into contact with food. Lactic acid is considered a dual use substance, since lactic acid is approved as a food additive (additive number E270). There are no SMLs or SML(T)s for the ingredients used to produce Luminy® PLA. The regulation does include an migration limit of 10 mg/dm2 on the overall migration from finished plastic articles into food. It is the responsibility of the manufacturer of the final product, when intended as a food contact product, to determine that the use of the product is safe and also suitable for the intended application. While it is TotalEnergies Corbion's conclusion that the above mentioned polymers are permitted, it is the final product which must meet the given regulations and the manufacturer should take responsibility to check if the final product is in compliance with these regulations

In the United States of America, Luminy® PLA as supplied by TotalEnergies Corbion has been evaluated and was found to be suitable for use in food contact applications. On 30 November 2018, FCN 001926 as applied for by TotalEnergies Corbion to the FDA became effective. It is included in the list of effective notifications for FCNs on the website of the FDA. The evaluation performed was in line with the requirements of Section 201(s) and Section 409 of the Federal, Drug and Cosmetic Act, and Parts 182, 184 and 186 of the Food Additive Regulations. Luminy® PLA neat resin is approved for all food types and conditions of use B through H.

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