Processing guide

Extrusion foaming of Luminy® FOAM 50F

TotalEnergies Corbion

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PROCESSING GUIDE
EXTRUSION FOAMING OF LUMINY® FOAM 50F
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INTRODUCTION

Extrusion foaming of polymers with a physical blowing agent, followed by thermoforming, is a common technique to obtain light-weight products for many segments, including packaging and insulation. Foamed products made from common materials like Polystyrene are increasingly under pressure by consumer sustainability awareness and stricter government regulations. Biobased, recyclable, and industrial compostable PLA materials enable the use of existing XPS processing equipment to obtain a drop-in solution for foamed products with almost similar properties.

The melt strength of standard PLA materials is too low to produce low-density foamed products and therefore PLA is typically modified during the extrusion step to increase its melt strength. But this requires an increased extrusion temperature and residence time which influences the process and throughput.
TotalEnergies Corbion developed Luminy® FOAM 50F with an inherently higher melt strength, specifically designed for extrusion foaming of PLA (XPLA). It does not require modification during the extrusion step and therefore has the potential to increase productivity and produce lower density product.

This processing guide describes the properties of Luminy® FOAM 50F and how to successfully convert the material into a low density sheet using a tandem extrusion set-up and subsequently thermoforming into a product.

LUMINY® FOAM 50F PROPERTIES

The physical properties of neat Luminy® FOAM 50F are listed in Table 1.

Property	Unit	Typical value
Density	g/cm3	1.24
MFI (190°C/10kg)	g/10min	5
Glass transition temperature	°C	60
Melting temperature	°C	165

Table 1: Physical properties of Luminy® FOAM 50F

STORAGE CONDITIONS

It is recommended to store Luminy® FOAM 50F in its closed, original moisture-barrier packaging at temperatures below 50°C. Storage in direct sunlight should be avoided. The supplied Luminy® FOAM 50F pellets are typically semi-crystalline and stay free-flowing up to temperatures of at least 100°C.

ADDITIVES

For controlling the foam morphology of the final products 0.5-1% talc with a particle size of 1-5 microns needs to be added together with Luminy® FOAM 50F into the extruder. As the talc needs to exfoliate in the PLA melt, it is highly recommended to use a talc masterbatch dispersed in a PLA carrier, or a carrier that is miscible with Luminy® FOAM 50F.
The recommended talc masterbatch is listed in Table 2.

Supplier	Product name
Granic	BioGranic 3171

Table 2: Talc masterbatches for Luminy® FOAM 50F

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PROCESSING LUMINY® FOAM 50F INTO FOAMED END-PRODUCTS

The steps to successfully make thermoformed XPLA articles typically include:

1. Raw material drying.
2. Extruder purging.
3. Extrusion into foamed sheet.
4. Conditioning of the foamed sheet
5. Thermoforming of the foamed sheet.

1. RAW MATERIAL DRYING

Moisture causes hydrolysis of Luminy® FOAM 50F during melt processing, resulting in decreasing the melt strength in the foaming process and reducing mechanical performances of the final part.
Therefore, it is important to process the material with a moisture level as low as possible.

Luminy® FOAM 50F will be supplied in sealed aluminum-lined barrier packaging with a maximum moisture content of 400 ppm. The moisture barrier packaging should always be kept sealed before usage and any unused material should be resealed under dry air or nitrogen immediately.
It is highly recommended to reduce the moisture content further before melt processing to a level less than 250 ppm, and preferably to less than 100 ppm.

Luminy® FOAM 50F can be dried using most conventional drying systems. The preferred method to dry the PLA material is by using a desiccant hot air dryer system, for which recommendations are given in Table 3. Another option is to use a vacuum drying oven.
The actual moisture content after drying should be checked with moisture sensitive analytical equipment, for which Karl-Fischer or Brabender Aquatrac methods can be used.
For extrusion foaming, usually a talc masterbatch with a PLA carrier is added together with the Luminy® FOAM 50F material in the throat of the extruder. It is also necessary the check the moisture content of the masterbatch material and dry it if necessary. Please check if the talc masterbatch (preferably with PLA carrier) stays free flowing at temperatures until 100°C. If not, then the drying temperature should be ≤ 40°C to keep the material free-flowing, and to prevent sticking of the material in the dryer.

Parameter	Pre-crystallized Luminy® FOAM and talc masterbatch	Amorphous talc masterbatch
Drying time (hours)	4-6	24
Air temperature (°C)	100	<40
Air dew point (°C)	<-35	<-35

Table 3: Typical drying conditions for Luminy® FOAM 50F and talc masterbatches

The dried Luminy® FOAM 50F and the talc masterbatch should be processed as soon as possible after drying. It is recommended to have a closed transport system from the dryer into the material feeder, a dryer installed on top of the feeder for transport under gravity, and/or to apply a dry nitrogen blanket in the feeder and throat of the extruder to prevent moisture uptake
Starting at 100ppm moisture content, the critical level of 250ppm is already reached after 15 minutes of exposure of the Luminy® FOAM 50F to atmospheric conditions (Figure 1).

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Extrusion foaming of Luminy® FOAM 50F

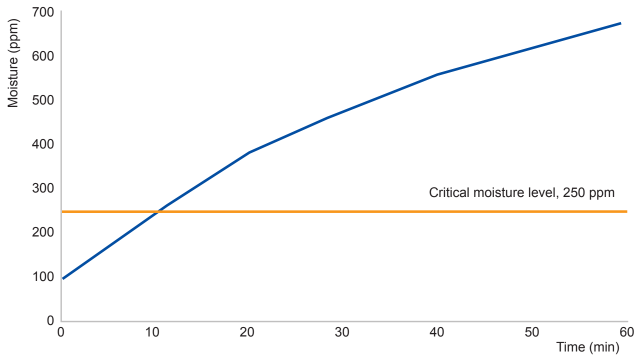

Figure 1: Moisture take-up curve PLA material.

2. EXTRUDER PURGING

Before introducing Luminy® FOAM 50F and the talc MB to the extrusion foaming machine, it needs to be well cleaned and purged to prevent cross contamination.
Ensure that the feeding and blending equipment in the material preparation steps (before the material enters the foaming extruder) is extensively cleaned and that it is free of dust and contamination of other previously used materials.
The purging procedures below are recommended for removing other polymers before processing PLA in the extrusion foaming setup:

1. Check if other polymers from previous runs can be 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. Remove as much as possible the previously used material from the barrel and screws.
3. Open the die as much as possible to clean out any non-molten or high viscous materials.
4. Preferably, purge the system with a purging compound (e.g. ASAclean, Dyna-Purge, etc.) followed by purging with a standard Luminy® PLA material with a low MFI or Luminy® FOAM 50F. Start with the material that has an MFI the closest to the MFI of the previously used material.
5. Change the temperature of the barrel to the standard processing temperature of PLA, which is 190-210°C.
6. Check that the processed material that exits the extruder-die is free of contamination before starting production.
7. Close the die to an opening that is needed for foam production.
8. At the completion of the run, it is recommended to purge the system again by using a purging compound to clean the machine from remaining PLA material. 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.

3. EXTRUSION LUMINY® FOAM INTO FOAMED SHEET

3.1. FOAM EXTRUSION SET-UP

Dried Luminy® FOAM 50F can be processed on most of the conventional XPS extrusion foaming machine setups. A typical tandem set-up is listed in Figure 2 and consists of:

a. A first extruder to melt and mix the Luminy® FOAM 50F with the talc masterbatch and the injection of a Physical Foaming Agent (PFA).
b. A second extruder to cool down the melt to a lower temperature to further increase the melt strength of the polymer mixture. Alternatively, a melt cooler that consists of internals with a cooling medium can also be used.
c. An annular die equipped on the second extruder to obtain a uniform tubular shaped, foamed sheet.

d. A cooling mandrel inside the tube and cold air blowing unit at the outside of the tube to cool down the sheet below 50°C, which is below the glass transition temperature of PLA.
e. Knives system to open the tube and wind the foamed sheet as roll stock.

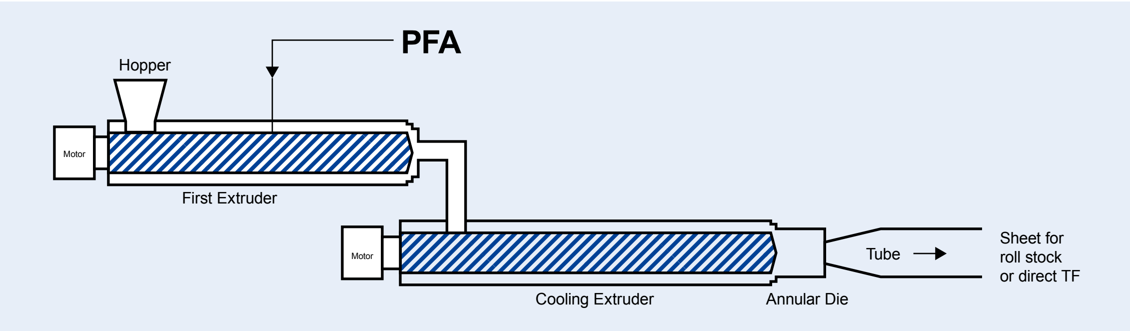

Figure 2: A typical tandem foam extruder setup. In some setups, the 2nd extruder is exchanged for a melt cooler.

Luminy® FOAM 50F has different flow characteristics compared to a standard PLA. If the torque of the first extruder exceeds its maximum limit, starve feeding is recommended to maintain or even increase the throughput.

The pressure in both extruders needs to be maintained at a high level (typically 50-150 bar) to ensure the PFA remains dissolved in the polymer melt. Once the melt exits the annular die, foaming starts immediately

3.2. MATERIAL FORMULATIONS

Luminy® FOAM 50F is typically mixed with 0.5% to 1% of micronized talc with a particle size of 1-5 micron. As the talc needs to exfoliate in the PLA melt, it is highly recommended to use a talc masterbatch with a PLA carrier, or a carrier that is miscible with Luminy® FOAM 50F.

Typical blowing agents that are used for XPS extrusion foaming can also be used for extrusion foaming with Luminy® FOAM 50F. These are hydrocarbon materials like Iso-butane, butane and LPG mixtures.

3.3. EXTRUSION PROCESSING CONDITIONS

The following procedure is recommended to start-up the process.

a. After cleaning and purging the extrusion foaming machine, the temperature of both extruders should be set to a temperature in the range of 190-210°C.
b. Start the rotation of the screws, feed dry Luminy® FOAM 50F material together with the dried talc masterbatch into the throat of the first extruder.
c. Wait when the polymer melt exits the die and process is stabilized.
d. Inject the PFA through the injection nozzle in the first extruder. Adding the foaming gas reduces the melt viscosity
e. Ensure no gas escapes via the throat of the extruder.
f. Wait at least two times the residence time of the material in both extruders.
g. Start reducing the barrel temperature of the second extruder (or melt cooler) initially in steps of 10°C, subsequently lower the barrel temperature in smaller steps of 5°C or even 1-2°C until the desired temperature is reached.

ad. g) By decreasing the temperature of the 2nd extruder, the melt strength will increase which is beneficial for the foaming process of the sheet. However a decreasing temperature results in an increase of the viscosity leading to higher pressure and torque. Ensure that torque and pressures do not exceed 90% of the maximum allowable values.
A good balance between these two needs to be obtained.

Depending on the extruder setup, typical barrel and melt temperatures during the constant foaming phase can be found in Table 4.

Extruder 1		
Parameter	**Unit**	**Settings**
Throat	°C	20-40
Feed zone	°C	190-210
PFA injection zone	°C	180-200
After PFA injection zone towards end of extruder	°C	160-180
Connecting pipe between extruder 1 and extruder 2 or melt-cooler	°C	160-180

Extruder 2		
Parameter	**Unit**	**Settings**
Start	°C	150
Middle	°C	140
End	°C	120-130
Annular die	°C	120-130
Tmelt	°C	130-140
Pressure in die	Bar	50-100
Polymer residence time in extruder	min	5-15

Table 4: Typical extruder settings and actuals for Luminy® FOAM 50F during the foaming phase.

3.4. COOLING AND CUTTING THE FOAMED TUBE

When the polymer melt exits the annular die, the tube immediately starts to foam. The tube must be pulled away from the die at a speed that does not deform the sheet and keeps the foamed cells inside the sheet as round as possible. Due to the foaming speed, corrugation at the die is almost always visible. If the pulling speed is too slow, the corrugation at the die will too much influence the sheet flatness.
When the expansion of the tube diameter is finished, it can be pulled over a cooling mandrel to cool it down to a temperature below 50°C. Only when the sheet is below that temperature, it can be cut in machine direction to collect it as rolls of foamed sheet.
As the cutting temperature for the foamed Luminy® FOAM 50F sheet is lower than the temperature for XPS foamed sheet, increased cooling capacity might be needed. Sometimes, just a lower mandrel cooling temperature is required, but during high-speed processing it might be needed to use a longer cooling mandrel than for XPS production. The outside of the sheet is cooled with blown air. To improve the cooling on this side of the sheet, refrigerated air can be used.

4. CONDITIONING OF THE FOAMED SHEET.

Usually, flammable hydrocarbon PFA gasses are used to foam the sheet made from Luminy® FOAM 50F. It is necessary that these gases migrate (mostly) out of the sheet to prevent safety issues during the heating of the sheet in a next thermoforming setup. The storage of the sheet should be done in a safe warehouse with enough venting. Typical waiting times for the roll stock foamed sheet are 4-7 days. Longer waiting time does not significantly influence the thermoforming step.

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5. THERMOFORMING OF THE FOAMED SHEET

5.1. THERMOFORMING SETUP: STANDARD AND HIGH HEAT PRODUCTS

A standard XPS thermoforming setup can be used to process the Luminy® FOAM 50F sheet into standard foamed products. An additional feature of Luminy® FOAM 50F is that it can be crystallized during the thermoforming processing to get heat-stable products. When that feature is required, it is necessary to equally heat up the mold to a temperature of ~100°C. It is also necessary to use a mold with a bottom and a top part, both with the possibility to apply vacuum to get the sheet pulled towards the mold surfaces in the molding step.

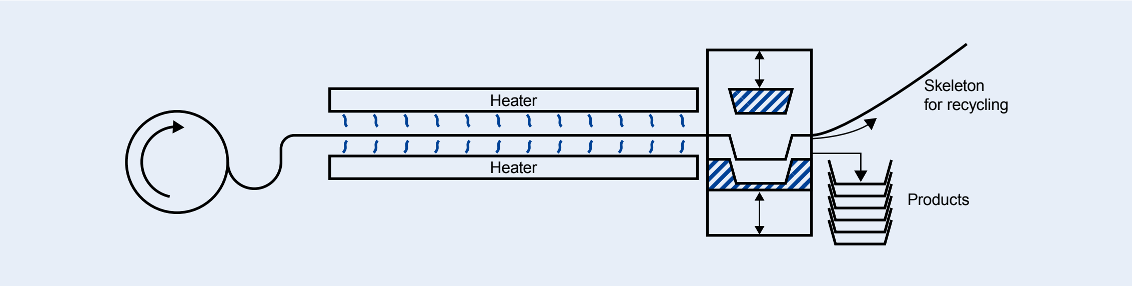

Figure 3 shows a typical thermoforming setup to obtain foamed products.

5.2. THERMOFORMING SETTINGS

The entire sheet thickness needs to be heated to a temperature of approx. 100°C before it can be formed in the mold. Due to the thickness and the structure, complete heating into the core of the foamed sheet takes quite some time. For that reason, top and bottom heaters are required in the TF setup.
When the made product does not require a temperature resistance over ~50°C, a cold mold with a temperature of ~15-20°C can be used. When the product heat resistance up to 100°C is required, it is necessary to crystallize the foamed product in a heated TF mold. Then, the mold should be heated to 100°C, where it is important that the temperature of the whole mold is within 1-2°C.
Exact heating and molding times depends on the sheet thickness and the product geometry. Molding times will be longer when the product needs to be crystallized. Table 5 shows some typical settings.

	Standard product temperature resistance		High heat product temperature resistance	
Sheet thickness	Top and bottom heaters	Mold temperature	Top and bottom heaters	Mold temperature
1-2 mm	250-300°C	15-20°C	250-300°C	~100±1°C
2-4 mm	300-350°C	15-20°C	300-350°C	~100±1°C

Table 5:Typical thermoforming settings for foamed sheet

5.3. RE-USE OF SKELETON

If the Luminy® FOAM 50F and the talc masterbatch are well dried before processing and it is processed under low-shear conditions, it is possible to mix grinded and compacted skeleton material with new Luminy® FOAM 50F pellets and process it into new foamed sheet. The maximum loading of grinded skeleton needs to be carefully validated, but typically 20-40% can be added back in. The addition of the talc masterbatch needs to be adjusted to have a similar talc loading in the mixture as without the grinded skeleton material.
All materials, including the grinded skeleton needs to be dried before processing to levels as recommended in the drying section.