Processing Guide for Biaxially Oriented Ingeo™ Film

This information is intended for use only as a guide for the manufacture of Ingeo biaxially oriented films. Because film orientation and downstream processing of Ingeo films is complex, an experimental approach may be required to achieve desired results.

1.0 Safety and Handling Precautions

All safety precautions normally followed in the handling and processing of melted thermoplastics should be followed for Ingeo 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 Ingeo melt processing. Appropriate air testing should be completed to ensure an acceptable Threshold Limit Value (TLV, based on a Time Weighted Average of 8 hours) 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 Ingeo has a 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. Unlike polyolefins, a molten web of Ingeo will not release as cleanly from a gloved hand so use caution when grabbing any molten stream or patty of polymer.

At ambient temperatures, Ingeo is considered non-hazardous according to DOT (US Department of Transportation) shipping regulations. When handling 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 grade being processed.

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

2.0 Pellet Storage and Blending Recommendation

Ingeo 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

Any of the film extrusion grades (4000 series) are the recommended Ingeo resin grades for biaxially oriented films. Typical properties of one of the grades are shown in the table below.

Typical Ingeo Resin Properties

Resin Property	Nominal Value
RV	3.9 – 4.1
Melt Temperature, °F (oC)	330 – 344 (165 – 173)
Glass Transition Temperature, °F (°C)	130 – 144 (55 – 62)
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,molten resin should not be left in the extruder, polymer filter, polymer transfer lines, dies or any other part of the extrusion system at 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

5.0 Drying

Ingeo 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 resin in carbon steel vessels (see Section 2.0).

In-line drying is essential for Ingeo resins. It is recommended that Ingeo 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 not adversely affect the stability of the polymer but it will increase the viscosity stability over time at elevated temperatures. Processes that have unusually long residence times or result in melt temperatures greater than 240°C should only extrude Ingeo 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's 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 Ingeo resin. Air or nitrogen based desiccant drying systems can be used at the recommended temperatures. Typical drying conditions are shown in the table below.

Typical Ingeo Raw Material Drying Conditions

	Typical Settings	
Drying Parameter	**Amorphous**	**Crystalline**
Residence Time (hours)	4	2
Air Temperature °F (°C)	113 (45)	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 Ingeo 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. Installation of a water cooled after-cooler may be necessary to prevent the drying air temperature from exceeding the recommended set point when drying amorphous materials.

6.0 Melt Extrusion

Prior to introducing Ingeo 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 magnates have been wiped clean. Insure that all hang-up areas such as elbows, transitions 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 Ingeo 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 Ingeo 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 Ingeo into material handling system.
3. Transition to pure Ingeo and purge the extruder again for a minimum 7x the average residences time. Change the screen pack when it becomes obvious that primarily Ingeo is exiting the die. Be sure to flush screen pack completely during the change. Screen pack should be between 80- 125 mesh for optimal performance. The lower mesh screen will result in reduced melt temperatures exiting the die.
4. At the completion of the run, purge all resin from the extrusion system, using a moderate to low melt index PP or PS, immediately after completion of the production run.

Following PET, PA, or HDPE in your system

1. Purge with low MFR (<1) PP at normal PET operating temperatures. Purge for at least 7x average residence time (~30 minutes). Let system empty as much as possible. Clean our hopper as much as possible.
2. Introduce a high melt flow PP (5 - 8 MFR) and change to normal Ingeo operating temperatures.
3. Purge for at least 7x average residence time. Let system empty as much as possible.
4. Alternatively, you can purge with a high flow PETG (similar to Eastman copolymer 6763) for a minimum of 40 minutes. Then reduce temperatures to Ingeo conditions.
5. Turn off extruder and completely clean all hoppers, elbow, slide gates, dryers, hopper loaders bins, hopper loader filters and material conveying lines of residual PET, PA or HDPE and PP. Load Ingeo into material handling system.
6. Transition to pure Ingeo and purge following again for a minimum 7x the average residence time. Change screen pack when it becomes obvious that primarily Ingeo is exiting the die. Be sure to flush screen pack completely during

the change. Screen pack should be between 80- 125 mesh for optimal performance. The lower mesh screen will result in reduced melt temperatures exiting the die.

7. At the completion of the run, purge all resin from the extrusion system, using a moderate to low melt
8. Index PP, immediately after completion of the production run.

Notes:

1. It is critical that all drying and conveying/receiving systems be free of all PET or PP and is vacuumed to ensure that there is no remaining polymer dust, before adding Ingeo. PET will not melt at Ingeo operating temperatures and will block screens, if it is present in the system.
2. The brand of PP used for purging is unimportant, as long as it does not thermally cross-link.

6.2 Extrusion

A general-purpose single-screw extruder, 24 to 36:1 L/D with feed-throat cooling is acceptable for processing Ingeo resins. 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. The following table shows a typical melt profile for film extrusion.

Typical Ingeo Extrusion Conditions

Extrusion Area	Temperature Setting, °F (°C)
Feed throat	113 (45)
Zone 1	355 (180)
Zone 2	375 (190)
Zone 3	390 (200)
Melt pump	390 (200)
Die	375 (190)
Desired final melt temperature	390 – 420 (200 – 215)

Note 1: In some instances where the extruder contains a screw not specifically designed for Ingeo, a reverse temperature profile has proven to be beneficial.

Note 2: Temperatures are only starting points and may need to be altered. Target Ingeo melt temperatures (after melt pump) should be in the range of 210±10°C (410±20°F).but in all cases, the resin should not be processed at temperatures above 240°C (464°F) due to excessive thermal degradation.

6.3 Additives

Colors, slip and anti-block agents are best added as a masterbatch at 10-30 wt% in Ingeo resins and controlled dosing the required amount of dried masterbatch into the feed throat of the running extruder. A significant component of the value of NatureWorks' Ingeo product, for both NatureWorks, LLC and Customer, lies in Ingeo resin being from more environmental friendly and sustainable feedstocks and processes. There are substances that could be used with Ingeo that could substantially diminish this value, therefore, NatureWorks, LLC and our customer agree that preservation and enhancement of Ingeo's product sustainability positioning and performance attributes requires limitations on the materials that can be added to, blended with or used to further process Ingeo-based products.

NatureWorks has established this list of prohibited substances to provide objective guidance and to ensure continuing improvement in the sustainable nature of our technology, processes and products. NatureWorks may from time to time modify this list to include additional substances that represent a significant risk. This list identifies a number of substances that may

pose a significant risk to human health or the environment. Please contact your NatureWorks, LLC representative to obtain the most recent list of substances banned from addition to Ingeo resins..

6.4 Filtration

Ingeo resins are highly filtered during their manufacturing process to remove gels that could cause disruption in the orientation process. Since the chemical nature of Ingeo is such that gels are unlikely to form during extrusion, high filtration at the extruder is unnecessary and in fact can be harmful. The large candle filters such as those typically used in many OPP extrusion lines significantly increases the residence time of the Ingeo at melt temperature. In addition, the increased back pressure generated on the screw increases the melt temperature of the Ingeo. The combination of increased melt temperature and residence time is detrimental in many ways. First, it can lead to a loss of molecular weight of the resin, which will reduce melt strength and possibly reduce properties. Even without a loss in molecular weight, the increased melt temperature by itself will cause a loss in melt strength and lead to difficulty in primary sheet casting. Finally, the yellowness index of the material can increase to unacceptable levels with long residence times and increased melt temperatures. The replacement of a large candle type gel filter with a simple screen changer equipped with screens in the range of 60 to 120-mesh is strongly suggested.

6.5 Sheet Casting

Extruder web is generally cast on a single chill roll. Due to the slow crystallization of Ingeo, water quench baths are generally not required. The cast roll temperature should be cool enough to chill the web, but warm enough that the molten web will adhere to the roll when pinned. Excessive cooling will result in a slipping of the web on the roll and more importantly, a build-up of residual lactide monomer which could affect the visual properties of the final film. Chill roll temperature should be between 35 and 60°C (95 – 140°F).

A monomer exhaust system is preferred to prevent the buildup of residual lactide around the die.

6.6 Pinning

Electrostatic pinning is required for Ingeo, preferably with a moving wire to eliminate streaking. Low pressure air pinning can be used at the edges to reduce neck-in and improve edge stability.

7.0 Orientation

7.1 Machine Direction

Orientation of Ingeo in the machine direction is done on conventional MDO roll stack. Ingeo does tend to neck in during drawing so nipped rolls are required on the slow and fast draw rolls. A reduced gap also decreased the amount of neck in. Typical MDO draw conditions are listed in the table below

Preheat rolls	45 – 65° C (112 – 150°F)
Slow draw roll	55 – 70° C (150 – 160°F)
Fast draw roll	70 – 75° C (160 – 170°F)
Annealing roll	45 – 55° C (112 – 130°F)
MDO stretch ratio	2x – 4 x (4042D or 4043D) 2x – 3 x (4032D)

7.2 Transverse Direction

Orientation in the TD is also achieved at much lower temperatures than either PP or PET. Typical conditions are listed in the table below for a thermally stable film that is heat set. A heat shrinkable film will require quick quenching after stretching instead of an annealing zone.

Preheat section	65 – 70° C (150 – 160°F)
Draw section	70 – 85° C (160 – 185°F)
Annealing section	125 – 140° C (260 – 285°F)
TDO stretch ratio	3x – 5 x (4042D or 4043D) 3x – 4 x (4032D)

7.3 Web Handling and Slitting

The stiffness of Ingeo is similar to OPS and quite different from either OPP or even OPET.Be careful of sharp edges particularly on thick cast sheet. 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 when producing film above 5 mils. Edge trim should be cut with a chopper near the slitters and air conveyed to a grinder. Reground edge trim Ingeo can immediately be added back to the virgin at levels up to 30% with minimal impact on extrusion performance. Any regrind should originate from otherwise prime film and not contain any contamination. Regrind and edge trim should be dried to the guidelines outlined in the drying section.

Safety and Handling Considerations

Safety Data Sheets (SDS) for Ingeo biopolymers are available from NatureWorks. SDS's 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. SDS's are updated regularly; therefore, please request and review the most current SDS's before handling or using any product.

The following comments apply only to Ingeo biopolymers; 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

Ingeo biopolymers 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. Pellets or beads may present a slipping hazard.

No other precautions other than clean, body-covering clothing should be needed for handling Ingeo biopolymers. Use gloves with insulation for thermal protection when exposure to the melt is localized. 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.

Good general ventilation of the polymer processing area is recommended. At temperatures exceeding the polymer melt temperature (typically 175ºC), 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 (or goggles) to prevent exposure to particles, which could cause mechanical injury to the eye. If vapor exposure causes eye discomfort, improve localized fume exhausting methods or use a full-face respirator.

The primary thermal decomposition product of PLA is acetaldehyde, a material also produced during the thermal degradation of PET. Thermal decomposition products also include carbon monoxide and hexanal, all of which exist as gases at normal room conditions. These species are highly flammable, easily ignited by spark or flame, and can also auto ignite. For polyesters such as PLA, thermal decomposition producing flammable vapors containing acetaldehyde and carbon monoxide can occur in almost any process equipment maintaining PLA at high temperature over longer residence times than typically experienced in extruders, fiber spinning lines, injection molding machines, accumulators, pipe lines and adapters. As a rough guideline based upon some practical experience, significant decomposition of PLA will occur if polymer residues are held at temperatures above the melting point for prolonged periods, e.g., in excess of 24 hours at 175°C, although this will vary significantly with temperature.

Combustibility

Ingeo biopolymers 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 option is to recycle into the process 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. Disposal must be in compliance with Federal, State/Provincial, and local laws and regulations.

Environmental Concerns

Generally speaking, lost pellets, while undesirable, are benign in terms of their physical environmental impact, but if ingested by wildlife, 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 environment.

Product Stewardship

NatureWorks has a fundamental duty to all those that 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, and then take appropriate steps to protect the environment and the health of our employees and the public.

Customer Notice

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