

## Best Practices for Ingeo™ Processing

This information is intended as a compilation of best practices for handling Ingeo™ polylactide resin and was developed over the course of many years. It is a compilation of tips and recommendations we feel are important for efficient and safe processing of Ingeo resins. For new customers, we encourage a thorough review of the document and invite them to contact their NatureWorks technical representative if there are any questions. For our existing customers, we expect many of them to be fully familiar with these recommendations, but would still encourage a review of this document just in case there are some points that they might have missed which will improve their processing methods. This document discusses best practices from 6 perspectives:

1. General Misconceptions about Ingeo Resins
2. General Safety and Handling Precautions
3. Solids Storage, Handling and Blending
4. Transitioning to Ingeo Resins on Commercial Lines
5. General Machine Design Considerations and Implications when Running Ingeo
6. Process Changes and Troubleshooting

Of course, it is always good for all industrial users to review safety practices, as safety awareness is a constant process of reinforcement. Throughout this document there will be references to other NatureWorks Technical Documents. The most recent versions of all of these documents are available on our website at <http://www.natureworkslc.com/technical-resources>.

### 1.0 General Misconceptions about Ingeo Resins

Because Ingeo resins are categorized as bioplastics, they are often mistakenly thought to have the same set of properties as other bioplastic resins. This is quite a dangerous assumption as every material (whether bioplastic or plastic) has its own unique set of properties. One of the most common misconceptions about the Ingeo polylactides is that they are water soluble. This is not the case. Ingeo is a hydrophilic polymer that will absorb some quantity of water but will not dissolve in water. In this respect, as an aliphatic polyester, it is very much like aromatic polyesters (such as PET) and polyamides (PA). Like these other materials, the level of moisture it absorbs is a function of the temperature and relative humidity. Table one below lists the equilibrium moisture content of water in the associated polymer at 50% relative humidity and 23°C (77°F). Just as PET and Nylon 6,6 are not water soluble, neither is Ingeo.

Polymer	Equilibrium Moisture Content (50% RH, 23°C)
Ingeo	2300 ppm
Polyamide (Nylon) 6,6	23000 ppm
PET	2600 ppm

Another misconception about Ingeo occurs when bubbles are seen in the extrudate. More times than not, the automatic assumption is that this is moisture and that the resin needs more drying time. It is true that under conditions of very high moisture content, on the order of 2% or more, some water will work its way to the end of the die and flash upon exiting the die to cause bubbles. When this occurs, there is also a significant amount of hydrolytic degradation that will cause drastic loss in molecular weight and the extrudate will have a very low viscosity. However, in most cases when the moisture level is high, the moisture will fully react with the polymer chain and simply break down the molecular weight. When this happens, no free water makes its way to the end of the extruder and therefore no bubbles are seen. There may be a notable difference in melt strength because of the loss in molecular weight and there might be an increase in fuming as lactide or oligimers may also form because of the water. However, in most cases, bubbles will not be seen. Generally, the presence of bubbles in extrudate can be attributed to the following causes.

- Low compression ratio of the extruder screw or low feed section temperature will establish a process where the mechanical action of the screw is not effectively squeezing air from the solids bed as the polymer is melting.
- A decomposition product of an additive being used in the process

## **Best Practices for Ingeo Processing**

- A void formation at an interface between the matrix of PLA and some incompatible polymer contaminating the system.
- A rolling melt bank can also cause bubbles in the film by entrapping air at the polymer melt interface. This can be ruled out by examining the die extrudate before it contacts the chill roll.

## **2.0 General Safety and Handling Precautions**

---

### **2.1 Normal Steady State Operations**

- Ingeo resins are thermoplastic materials sold in a pellet form for subsequent processing in a molten state into shaped articles and forms. Therefore, all safety precautions normally followed in the handling and processing of melted thermoplastics should be followed for each of the 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<sup>3</sup> is maintained. The use of process area point source remediation measures such as monomer fume hoods or exhausts near the die or over any open vent ports are typically recommended.
- Molten Ingeo resin 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 Ingeo web 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, molten Ingeo waste should be allowed to cool before being placed into any waste container to minimize fire risks.
- Processors who extrude sheet will have observed that lactide fumes in the warm air near the die will plate out on the cool chill roll. There are additional unit operations in processes in which initially warm volatiles (which may be air, moisture, and lactide) cool down and lactide condenses as a solid. Examples include processes which crystallize and dry high levels of regrind or utilize vacuum venting in extrusion. It is important to protect vacuum pumps or process air heat exchangers, and to clean up relatively white lactide solid where it has plated out before it hydrolyzes with moisture from the environment to become corrosive lactic acid and lactic acid oligomers (typically dark colored viscous liquids).
- When the proper roll temperatures are used and the Ingeo sheet is properly pinned to the casting roll, there should be no plate out on the wetted surface of the rolls. However, during start up and transitions to different gauge products, there may be a period of time where lactide is plating out on the rolls. This can be cleaned by using simple non abrasive spray cleaners such as Simple Green along with some clean cloths.
- For vacuum systems, it is essential to add an in-line cold trap to force the lactide out of the warm volatiles stream at a specific location to protect equipment and to be easier for maintenance to clean and discard the lactide. Care should be taken while cleaning to prevent skin contact through the use of personal protective equipment such as long sleeves, chemical resistant gloves, etc.

### **2.2 Extended Shut Downs**

---

Generally considered as a period of time greater than 2 hours when no polymer is flowing through system and temperatures are greater than the melting point of the polymer.

- A thermal decomposition product of any PLA is acetaldehyde, a material also produced during the thermal decomposition 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

## Best Practices for Ingeo Processing

temperature over longer-than-normal residence times (e.g. 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.

- Under normal extrusion operations, the residence time of the molten polymer is on the order of minutes and therefore no thermal decomposition is expected when the recommended set temperatures are used. It is not recommended to shutdown extrusion or transfer lines while keeping them heated for extended times with PLA still inside the equipment. If equipment is to be kept at temperatures above 175°C for more than 3 or 4 hours, NatureWorks recommends purging PLA from the system with a more thermally stable polymer such as a high melt flow polypropylene which then can easily be purged out with PLA when operations restart.
- Minimize holding time of PLA inside of process equipment in the molten state. Clear equipment rather than keeping on hold for extended periods at elevated temperatures above the melting point (> 175°C). Clear/purge materials to a safe location in case decomposition continues. Be aware that the rate of thermal decomposition increases rapidly with increasing temperature.
- Purge lines and equipment with nitrogen to keep the products of PLA decomposition out of the flammable range. Purge the gases to a safe location. Be mindful of the hazards of asphyxiation associated with the use of inert gases.
- Be aware that the introduction of air into a system that has been kept under heating conditions can lead to potentially explosive conditions and should be avoided.
- Treat line and equipment opening with special caution, particularly if they contain PLA which has been held above 175 °C for an extended period, since highly flammable decomposition gases may auto ignite if above about 175 °C or which can ignite and burn as easily as methane even when cooled below 175 degrees C. Use of flammable gas detection meters is strongly suggested as part of preparation for line and equipment opening and clearing procedures.
- Be sure to keep spark and flame producing equipment out of the process areas until proper clearing of the line has been completed.
- Use proper maintenance tools when clearing or working around equipment containing PLA or PLA decomposition products. Consider use of explosion meters, non spark producing tools, fire resistant clothing and foot wear that is non spark producing depending on the nature of the work being performed. Clothing that will melt and stick to the skin when subject to flame should also not be used in a work place where flammable vapors or liquids can be present.
- Design piping to minimize leaks with leak-resistant gaskets like spiral wound or metal rings. Use low surface area insulation in areas where leaks might be expected. Use temperature limiting heat tracing to avoid excessive hot spots.
- Train both operation and maintenance personnel on the hazards of processing PLA at elevated temperatures and particularly the special hazard of thermal decomposition for prolonged periods. Make sure operating and maintenance procedures contain adequate precautions for these hazards.
- During normal operations, there often are times when the extrusion system needs to be stopped to perform maintenance on part of the line. If this time is short (less than 2 hours) and the system temperatures are in control at normal PLA processing temperatures of less than 240°C, the risk of an unplanned event due to thermal decomposition is very low and purging of the system is not necessary. However, thermal degradation, resulting in a loss of molecular weight accompanied by a drop in viscosity will occur. Upon restarting the line, it is imperative that all safety precautions for starting a line with low molecular weight polymer be followed. This includes keeping away from the front of the extrusion die and keeping all exposed skin protected from hot molten polymer. Anyone in near the extrusion die should also wear full protective face shield as low viscosity polymer can spray from the front of the die upon start up.

### 3.0 Solids Storage, Handling and Blending

---

- 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).
- Best results will always be achieved if Ingeo resin is dried prior to any extrusion or injection molding process.
- For short experimental programs, resin can be processed if a) the product is kept in its original sealed container before use and b) all reasonable efforts are made to minimize exposure of the pellets with ambient air. This can mean anything from keeping resin in covered containers to purging containers with dry air or nitrogen to limit

## **Best Practices for Ingeo Processing**

infiltration of humid air.

- Ingeo grades that are not lubricated have a high angle of repose which means that the pellets will not flow easily over each other. In practice, this can lead to poor efficiency of conveying air wands and “rat-holing” around the wand tip. Operator intervention or vibration pads will be necessary when unloading unlubricated grades from gaylord boxes with vacuum conveying wands.
- Conveying of Ingeo pellets can lead to a condition where static charge buildup is observed. You should ensure that all conveying wands and pipes are properly grounded to prevent electrical sparks.
- When forming Ingeo into sheet, be aware that it is a high modulus material. When it is cut or there are web breaks, sharp edges can easily be formed so all Ingeo scrap should be only handled with gloved hands.
- When grinding Ingeo scrap, be aware that it has a much lower Tg than polystyrene and slightly lower Tg than PET. Generally, most sheet product is amorphous unlike polypropylene. Therefore grinders should be operated in such a manner to keep knives and beds cool with maximum airflow to facilitate cooling and prevent melting of the resin. Hole size for grinders will be dependent upon the speed of the rotor, the air flow through the grinder which cools the system and the rate of material feed. If melting occurs, then hole size may need to be increased.
- On oriented film lines, the edge trim at the TDO exit is most easily handled if the trim is cut soon after trimming and then conveyed to holding bins.

### **4.0 Transitioning to Ingeo on Commercial Lines**

---

- The most important step in the purging process when introducing Ingeo on a commercial line that normally runs another polymer is cleaning of all the equipment and piping that handles solid Ingeo, either as pellets or regrind.
- Special attention should be given to conveying lines, the lids of vessels and hopper loaders, drain cutters on bins and loaders and especially grinders. Additive feeders also need to be thoroughly cleaned and inspected for residual materials of previous polymer.
- Proper purge procedures for the extruder, transfer lines and dies are also critical and NatureWorks recommended purging procedures should be followed.
- Different materials contaminating the Ingeo stream will give different types of defects or problems in extrusion
  - Polyester contamination upstream of screen changer will result in rapid pressure rise as polyester will not melt at Ingeo processing temperatures and collect on screen. Even residual dust from PET grinding and slitting operations can contaminate a PLA sheet line and cause visible gels.
  - Polystyrene processes at similar temperatures as Ingeo with similar viscosity and therefore small amount of PS in an Ingeo stream will typically just result in a hazy extrudate
  - Polypropylene contamination will also result in hazy extrudate but may also be accompanied by large gels or fisheyes.
- The sequence of when to add materials and when temperature changes are critical and therefore be sure to follow suggested purging procedures.
- Since clarity of the extrudate is the best indicator of when purging is complete, it is best to delay the addition of any pigment or additive masterbatches until it is obvious that all previous polymer has been purged from the system with Ingeo.
- Molten Ingeo adheres well to metal and concrete surfaces so when purging large lines and collecting patties on the floor or in metal basins, be sure to wet the surface with water to facilitate release. In the case of metal catch basins, a spray mold release can also be used.

### **5.0 General Machine Design Considerations and Implications when Running Ingeo**

---

- All polymer processing lines are designed to process a particular polymer and therefore it is critical to understand differences in the fundamental polymer properties when introducing a new polymer into a equipment designed for another material.
- Polymer density (both solid and melt), thermal conductivity, glass transition temperature, and shrinkage are critical design parameters.
- Ingeo has a much lower Tg than polystyrene and therefore tooling designed for polystyrene needs far less cooling channels than tooling designed for Ingeo, or for that matter PET. Both of these materials need to be cooled to lower

## **Best Practices for Ingeo Processing**

temperatures than PS before they can be ejected from a mold or else they will deform under stress. Both of these materials also have a higher density than PS and therefore there is more mass in the tool than when processing Ingeo. This also adds to the cooling load and can increase cooling time.

- The higher T<sub>g</sub> of PS also allows for running smaller diameter cooling rolls on sheet lines than are needed for higher density, lower T<sub>g</sub>, materials like PET and Ingeo
- Materials of construction should be consistent with the recommendations in the grade of Ingeo being used and the particular polymer process. Be sure to consult the NatureWorks Process Guides and Technical Data Sheets.
- As stated earlier, the safest way to ensure minimal molecular weight loss during extrusion is to dry Ingeo to less than 250 ppm moisture level. For fiber extrusion and extrusion lamination lines, the recommendation is less than 100 ppm moisture.
- While vented or vacuum extruders have been successfully used without pre-drying the Ingeo feed material, their efficiency is highly dependent upon not only the design and operation of the equipment, but also moisture content of the incoming feed material. NatureWorks recommends discussing the use of dryerless extrusion lines with your Technical Service representative before considering the use of dryerless extrusion lines.
- When extruding amorphous Ingeo in a single screw extruder, screw cooling is necessary to prevent polymer from sticking to the screw root and impeding flow. The recommended temperature for the screw cooling water or oil is 80-100°F (26 - 38°C).
- Amorphous regrind up to rates of 30% can be added back into a single screw extrusion process without the use of screw cooling.
- When using regrind at levels greater than 30% and screw cooling is not available, then the regrind should be crystallized. One of the most common techniques is to blend regrind with virgin pellets and feed this blend into the crystallizer to assure good flow and minimal clumping of ground flake as it crystallizes. More information on crystallization of Ingeo is available in the NatureWorks Crystallization and Drying Process Guide.
- Ingeo has a higher density than polypropylene or polystyrene and so Ingeo rolls will be heavier than rolls of same dimensions of these materials. Be sure the load capacity of any lifting device is rated for this increased weight and that personnel do not try and lift loads heavier than recommended local safety guidelines.

## **6.0 Process Changes and Troubleshooting**

---

- The viscosity of Ingeo is much more sensitive to changes in temperature than other thermoplastics such as polypropylene and polyester. Therefore when making temperature adjustments to fine tune a process, much smaller changes are required to get the desired response. As a rule of thumb, it is best to make one half of the temperature change than you would when processing polypropylene or PET.
- This sensitivity to temperature means that care should be taken to minimize air flow around dies, particularly flat dies, which could make it difficult to control gauge.
- Just as with other polymers, the quality of the melt exiting the die or nozzle of an injection molding machine will determine the quality of the final product. In complex polymer processes such as biaxially oriented film or sheet, it does not make sense to start MDO or TDO stretching until the quality of the cast sheet is acceptable.
- Specific recommendations and troubleshooting guides are available for the various processing operations from NatureWorks

*©2015 NatureWorks LLC. All rights reserved. The information provided herein is provided in good faith, but no warranties, express or implied, are provided with respect thereto. It is the responsibility of the users of Ingeo resin to determine the proper conditions of use on their particular equipment and at their particular facilities. No rights under any patent of NatureWorks LLC or any third party is granted or implied.*

## Best Practices for Ingeo Processing

### 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 175°C), 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

## **Best Practices for Ingeo Processing**

intended use, and then take appropriate steps to protect the environment and the health of our employees and the public.

### **Customer Notice**

---

NatureWorks encourages its customers and potential users of its products to review their applications from the

standpoint of human health and environmental quality. To help ensure our products are not used in ways for which they were not intended or tested, our personnel will assist customers in dealing with ecological and product safety considerations. Your sales representative can arrange the proper contacts. NatureWorks literature should be consulted prior to the use of the company's products.

#### **NOTICE:**

No freedom from infringement of any patent owned by NatureWorks LLC or others is to be inferred. No information in this publication can be considered a suggestion to infringe patents.

The technical information, recommendations and other statements contained in this document are based upon tests or experience that NatureWorks believes are reliable, but the accuracy or completeness of such information is not guaranteed. Many factors beyond NatureWorks control can affect the use and performance of a NatureWorks product in a particular application, including the conditions under which the product is used and the time and environmental conditions in which the product is expected to perform. Since these factors are uniquely within the user's knowledge or control, it is essential that the user evaluate the NatureWorks product to determine whether it is fit for a particular purpose and suitable for the user's method of application. In addition, because use conditions are outside of NatureWorks control and applicable laws may differ from one location to another and may change with time, Customer is solely responsible for determining whether products and the information in this document are appropriate for Customer's use and for ensuring that Customer's workplace, use and disposal practices are in compliance with applicable laws and regulations. NatureWorks LLC assumes no obligation or liability for the information in this document.

NATUREWORKS MAKES NO WARRANTY, EXPRESS OR IMPLIED, REGARDING THE INFORMATION CONTAINED HEREIN OR ITS PRODUCTS, INCLUDING BUT NOT LIMITED TO ANY WARRANTY AS TO ACCURACY OR COMPLETENESS OF INFORMATION, OR ANY IMPLIED WARRANTY OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE.

**NOTICE REGARDING PROHIBITED USE RESTRICTIONS:** Unless specifically agreed to in writing by NatureWorks, NatureWorks LLC will not knowingly sell or sample any product into any of the following commercial or developmental applications (i) components of or packaging for tobacco products, (ii) components of products intended for human or animal consumption, (iii) any application that is intended for any internal contact with human body fluids or body tissues, (iv) as a critical component in any medical device that supports or sustains human life, (v) in any product that is designed specifically for ingestion or internal use by pregnant women, (vi) in any application designed specifically to promote or interfere with human reproduction, (vii) in microbeads, including those used in personal care/cosmetic applications, or (viii) to manufacture bottles or bottle pre-forms in North America.

For additional information please contact NatureWorks via our [website](#) on the tab called [FAQ's](#) or by clicking [here](#).



15305 Minnetonka Blvd., Minnetonka, MN 55345