PLA Processing Guide for Spiral Crimped Staple Fibers

Spiral crimped or self crimping INGEO™ Fibers can be made by exploiting morphology differences across the fiber either by utilizing the inherent morphology difference of two different polymers, or by creating a morphology difference in a homopolymer by means of additives, or process manipulation. This can be achieved by the same methods as used for polyester. These methods include:

- Bicomponent technologies such as side by side and eccentric sheath core, which exploit molecular weight and/or stereochemistry differences of each component. Similar effects can be achieved by manipulating other melt spinning process variables (i.e melt viscosity) that cause a differential in the orientation level across the fiber diameter, while using a homopolymer. Additionally, polymer additives like cross linkers or branching agents could also be used to create a similar effect.
- Differential cooling/quenching of solid, hollow, and shaped fibers with eccentric holes.

As the fiber is drawn, the orientation , and/or shrinkage differential between the two sides is magnified, resulting in the formation of helical or spiral crimp once the tension is released and/or the fiber is heatset.

This information is intended for use only as a guide for the manufacture of spiral crimped PLA fibers. Because melt spinning and downstream processing of PLA fibers 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 NatureWorks® PLA resins.

As with most thermoplastics, melt processing and the variability of those conditions will result in minor decomposition. Lactide, a non-hazardous gaseous irritant, is a minor by-product of PLA melt processing. Appropriate air testing should be completed to ensure acceptable Threshold Limit Values (TLV) of less than 5 mg/m^3 are maintained. The use of process area point source remediation measures such as monomer fume hoods or exhausts near the spinneret are typically recommended.

PLA is considered non-hazardous according to DOT shipping regulations. Care should be taken to avoid direct skin/eye contact along with conditions that promote dust formation. Product may cause eye/skin irritation. Product dust may be irritating to eyes, skin and respiratory system. Caused mild to moderate conjuctival irritation in eye irritation studies using rabbits. Caused very mild redness in dermal irritation studies using rabbits (slightly irritating). Ingestion may cause gastrointestinal irritation, nausea, vomiting and diarrhea. For further information, consult the appropriate MSDS for the PLA grade being processed.

2.0 Pellet Storage and Blending Recommendation

PLA resins should be stored in an environment designed to minimize moisture uptake. Product should also be stored in a cool place at temperatures below 50°C (122^{o}F).

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 stored in silos, hoppers etc 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 fiber production area and allowed to equilibrate for a minimum of 24 hours before opening.

During chip transfer, minimal transfer air temperature (~40C) and velocity (~25 m/s for dilute phase transfer systems) are recommended to minimize the potential of generating fines.

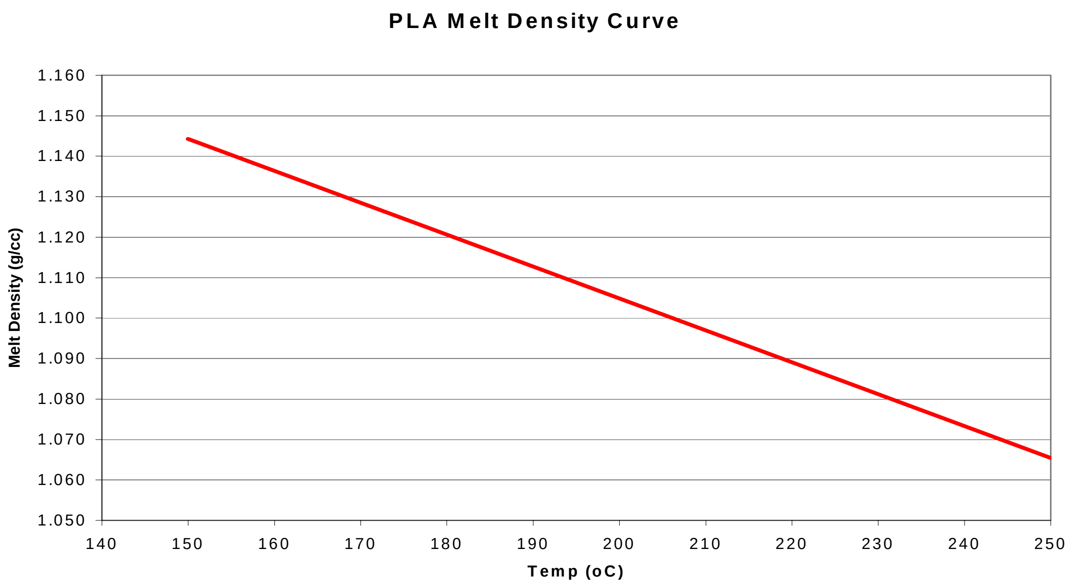
3.0 Resin Properties

6201D and 6251D are the recommended PLA resin grades for spiral crimped staple fibers. Typical properties of 6201D and 6251D are shown in the table on the following page.

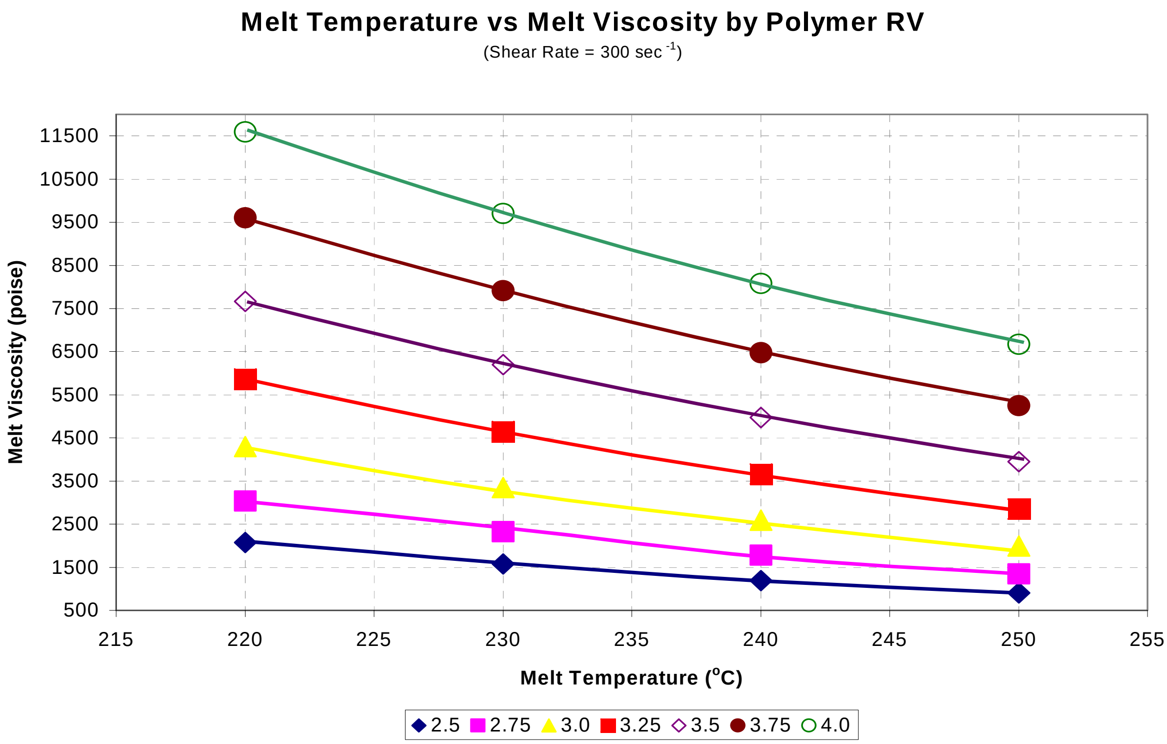
	Nominal Value	
Resin Property	**6251D**	**6201D**
RV	2.4 – 2.6	3.0 – 3.2
Melt Temperature (^{o}C)	165 - 173	165 – 173
Glass Transition Temperature (^{o}C)	55 – 62	55 – 62

Typical PLA Resin Properties for Side-Side Spiral Crimped fibers

The graph on the following page shows the melt density of PLA over a wide temperature range.



Melt viscosity curves at a fixed shear rate are shown in the following graph.



4.0 Materials of Construction

All metal parts in the extrusion process should be constructed of stainless steel to minimize corrosion. Furthermore, PLA should not be left in the extruder, polymer filter, polymer transfer lines, spin beam, spinnerets or any other part of the extrusion system at PLA 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 and spin beam	SUS440C

5.0 Drying

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

In-line drying is essential for PLA resins. A moisture content of less than (50 PPM) is recommended to prevent viscosity degradation. Material is supplied in foil-lined containers dried to less than 400 PPM as measured by NatureWorks LLC 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

PLA. Air or nitrogen based desiccant drying systems can be used at the recommended temperatures. Typical PLA drying conditions are shown in the table below.

	Typical Settings	
Drying Parameter	**Amorphous**	**Crystalline**
Residence Time (hours)	4	2
Air Temperature (^oC)	50	100
Air Dew Point (^oC)	- 40	- 40
Air Flow Rate (m^3/min/kg resin)	> 0.031	> 0.031

Typical PLA Raw Material Drying Conditions

Typical desiccant dryer regeneration temperatures exceed the melt point of PLA 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.

6.0 Melt Spinning

Prior to introducing PLA into any melt spinning system, the system should be properly purged to prevent any polymer contamination and spinnability problems from occurring. The purging procedures below are recommended for optimal removal of other polymers.

6.1 PLA Purging Procedure

Following PP in your system (not degraded by cooling and re-heating system)

1. At normal PLA operating temperatures, run a high melt index PP (15- 40 MI) without spinneret in place. Purge for at least 3x average residence time. Let system empty as much as possible.
2. Transition to purge PLA and purge following the same guidelines as step 1.
3. Insert pre-heated spinneret and allow temperature to equilibrate.
   Purge with 6201D and evaluate flow from capillaries. As long as flow is even from each capillary and there is no evidence of contamination, begin spinning.
4. Purge all PLA from the extrusion system, using a high melt index PP, immediately after completion of the production run

Following PET, Nylon, or HDPE in your system

1. Purge with low MI (<1) PP at normal PET operating temperatures. Purge for at least 3x average residence time (~30 minutes). Let system empty as much as possible.
2. Change to normal PLA operating temperatures and run a high melt index PP (15- 40 MI).
   Purge for at least 3x average residence time. Let system empty as much as possible.
3. Transition to purge PLA and purge following the same guidelines as steps 1 and 2.
4. Insert pre-heated spinneret and allow temperature to equilibrate.
5. Purge with 6201D and evaluate flow from capillaries. As long as flow is even from each capillary and there is no evidence of contamination, begin spinning.

Purge all PLA from the extrusion system, using a high melt index PP, immediately after completion of the production run

Important 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 PLA. PET will not melt at PLA operating temperatures and will block screens, if it is present in the system.

2. Brand of PP used for purging is unimportant, as long as it does not thermally cross-link.
3. When handling PLA pellets, the generation of small particles or fines is possible. Conveying pellets slower, such as at a velocity of 25 m/s, will generate fewer fines than at 30 m/s when conveying in dilute phase. Please note that with dilute phase conveying, enough velocity must be maintained to prevent the pellets from plugging the line. Internal and external testing did not show plugging problems at 25 m/s.

6.2 Extrusion

General-purpose single-screw extruders, 24 to 36:1 L/D with feed-throat cooling are acceptable for processing PLA. A mixing tip is generally recommended along with static mixers in the product line to ensure temperature uniformity as well as optimum additive dispersion and melt polymer homogeneity. The following table shows a typical melt profile for PLA.

Extrusion Area	Melt Temperature Setting (^{o}C)
Feed throat	25
Zone 1	230
Zone 2	235
Zone 3	240
Melt pump	240
Spin head	240

Typical PLA Extrusion Conditions

Note 1: Temperatures are only starting points and may need to be altered. Target PLA melt temperatures (after melt pump) should be in the range of 240±5°C (455±9°F).

Note 2: PLA resins should not be processed at temperatures above 250°C (482°F) due to excessive thermal degradation.

6.3 Additives

Delusterants such as TiO2 are best added as a masterbatch at 15-30 wt% in PLA resins and controlled dosing the required amount of dried masterbatch into the feed throat of the running extruder.

6.4 Filtration

PLA resin will be provided pre-filtered to a level of 20 microns. The following pack makeup is recommended:

Loose media (optional - depending upon pack configuration) 200-350 micron shattered metal is recommended for an uncompressed pack cavity fill.

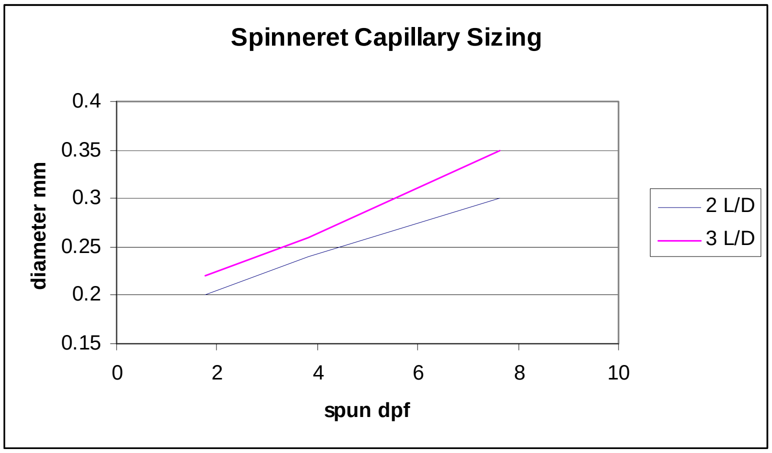
Screens – cascade configuration with appropriate support screens is recommended with finest filtration level of 325 mesh or finer.

6.5 Heating Systems

To allow for the required temperatures to be obtained in spinning, typically vapor heat transfer system medium changes are required. Dowtherm® J / Therminol® LT or a comparable vapor HTM which has an atmospheric boiling point of 200°C or less while remaining within specific system pressure design limits is generally recommended. Operation of the HTM system at a temperature as close as possible to the actual melt temperature (235±5°C) is recommended to provide an adiabatic spinning system. For vacuum assisted systems, typically heat transfer medium changes are not required as long as the system vacuum can be operated at a level to provide vaporization and uniform heating at the suggested temperatures (230-240°C). Prior to being placed into service, spin packs should be heated to 250^{o}C to allow for some temperature loss during spin pack installation.

6.6 Spinnerets

Recommended capillary dimensions for a fiber with a solid round cross section range from 0.2-0.35 mm diameter, typically with a 2 to 3:1 L/D ratio. Larger capillaries may be necessary for fiber's greater than 6 dpf. The following guide can be used to estimate spinneret requirements based on spun product dpf:



Capillary dimensions for modified cross sections will deviate greatly from solid round fibers. They should be designed to meet the desired fiber shape, while providing adequate pressure drop to ensure good denier uniformity and adequate draw down or stretch ratio to facilitate good spinning performance.

6.7 Quenching

Filaments should be quenched with air at controlled temperatures and velocities to ensure good denier and orientation uniformity. Typical quenching conditions are shown below, but quenching conditions need to be optimized by product depending upon the denier, spinneret design, and cross section.

Quench parameter	Typical Target
Quench Air velocity (m/s)	0.26 – 1.5
Quench Air Temperature (^oC)	10 – 20
Monomer Exhaust Velocity (m/s)	.26 – 2.0
Spacer or Shroud Length (mm)	50 -100

Typical PLA Quenching Conditions

A monomer exhaust system is preferred to prevent the buildup of residual lactide around the spinneret face and quench screen.

6.8 Take Up

PLA can be spun over a wide range of take up speeds, but typically runs between 1100 and 1850 m/min.

7.0 Drawing

7.1 Creeling and pre-tensioning

7.1.1 Creel Size

Creel size is largely dependent upon the crimper size and the tension rating of the drawstands. PLA can be run at similar tow densities in crimping as PET, with typical densities ranging from 65 to 72 thousand dtex per cm of crimper width.

7.1.2 Creel Tensioning

Creels should be designed to provide a minimal tension level on each subtow in the creel. If the tension is too great, typically greater than 0.2 g/den, the tow could begin drawing before or during immersion in the pre draw bath, causing excessive denier variation along with the possibility of broken fibers and high wrap rates. In addition, the tension level between individual subtows in the creel should be uniform. An adjustable pre tension stand is preferred prior to the pre draw bath to assist in equalising the tension between subtows.

7.2 Pre Draw Bath

Pre-draw baths should be sufficiently long to enable saturation of the tow with moisture and finish and to initially raise the tow temperature to 25-50^oC. The tow should not be heated above PLA's Tg (58^oC), other wise the tow could begin drawing prematurely, which would lead to the same types of problems described in the preceding section. Also nip rolls are recommended on the pre draw rolls to minimize tension on the towband before drawing.

7.3 Drawing

As mentioned earlier, a stress differential is developed across the fiber as it is drawn since the higher molecular weight material requires a higher force to draw than the lower molecular weight material. This differential is what causes the crimp to form. The crimp is further developed as it undergoes drying and heatsetting since there is also a shrinkage differential between the two molecular weights. For successful spiral crimp formation with PLA, drawing must be done in a single step. The crimp level is dependent upon drawing temperature. Low temperatures yield higher crimp levels, while high temperatures reduce the level. If drawing temperature exceeds ~85^oC, the crimp will not form at all. This temperature is not an absolute maximum, but a general rule since the orientation level and differential of the as-spun material, and draw ratio will also impact the level of crimp.

7.3.1 Temperatures

Drawing temperatures should be maintained between 75^oC and 85^oC to ensure that spiral crimp is formed without stress whitening the fiber. Drawing at cooler temperatures could produce higher crimp levels, but stress whitening would occur. Drawing at higher temperatures will eventually result in no crimp formation.

7.3.2 Draw Ratios

For spiral crimped fibers, PLA must be drawn at lower draw ratios relative to mechanically crimped products since drawing temperatures must be maintained below 85^oC, and all drawing must be done in a single drawing step. Typical draw ratios are $\leq$ 2.5:1. Drawing above this level causes stress whitening. However, draw ratio can be increased by reducing the orientation of the as-spun material. Reducing the melt viscosity (lower RV, lower spinning speed, higher melt temperature and/or moisture) and/or reducing the quenching rate can reduce the as-spun orientation. Reducing the orientation will allow increased draw ratios before the onset of stress whitening.

7.4 Finish application

Finish should be applied before entering the dryer or heat setter. Applying finish after the dryer will de-stabilize the tensile properties of the fiber with time. Finish should be selected based on downstream processing requirements. Goulston Technologies, Inc or Takemoto Oil and Fat Company LTD can recommend and provide finishes for PLA that have been proven for a variety of applications. Depending on subsequent processing and finish types, application levels range from 0.35% up to 0.8% finish on fiber.

7.5 Tow Transfer Systems

Traditional transfer methods used to transfer spiral crimped PET from the drawline to the heatsetter should be adequate for PLA.

7.6 Heatsetting

Hot through air dryers are recommended for drying and heatsetting. The dryers should have multiple zones with a cooling zone at the dryer exit. Temperature control is critical, as the objective is to expose the fiber to temperatures as close to the melt point as possible without actually melting the fiber. Recommended drying temperatures for PLA range from 120 to 140^{o}C.

7.7 Cutting

The temperature of the tow before entering the cutter should be maintained below 50^{o}C to ensure that the crimp does not get pulled out due to the tension at cutting. Tension stands are recommended prior to cutting. Conventional rotary cutters are acceptable.

7.8 Baling

Conventional polyester balers are acceptable for baling PLA. The bale density required to produce stable bales will vary with denier and finish type. Bale densities range from 0.27 – 0.40 g/cm^3 (17 – 25 lb/ft^3). Typical pressures applied in baling range from 50 to 100 psi. Low melt point binder fibers require lower ram pressure to minimize the potential of fiber sticking together in the bale.

7.9 Fiber Storage

Baled fiber should be stored in cool, clean, dry location to prevent hydrolysis and contamination. Environmental conditions of 25^{o}C, 55% RH are recommended.

8.0 Fiber Properties

The table below shows some typical PLA fiber properties.

	Product Description		
Fiber Property	2.5 dpf hollow	4 dpf hollow	7 dpf hollow
Denier	2.6	4.7	7 – 8.2
CPI	4-12		6 - 8
Tenacity (g/den)	2.6 – 3.3	2.7	2.2 – 2.8
Elongation to break (%)	55	120	75 - 135
Finish on Fiber (%)	0.25-0.35		
Hot air shrinkage (130^{o}C, 10 mins)	$\leq$ 5	$\leq$ 4.0	$\leq$ 4.0

Safety and Handling Considerations
Material Safety Data (MSD) sheets for PLA polymers are available from NatureWorks LLC. MSD sheets 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, such as OSHA (U.S.A.), MAK (Germany), or WHMIS (Canada). MSD sheets are updated regularly; therefore, please request and review the most current MSD sheets before handling or using any product.

The following comments apply only to PLA polymers; 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
PLA polymers 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. 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. Pellets or beads may present a slipping hazard. Good general ventilation of the polymer processing area is recommended. At temperatures exceeding the polymer melt temperature (typically 170ºC), 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 if there is a potential for exposure to particles which could cause mechanical injury to the eye. If vapor exposure causes eye discomfort, use a full-face respirator. No other precautions other than clean, body-covering clothing should be needed for handling PLA polymers. Use gloves with insulation for thermal protection when exposure to the melt is localized.

Combustibility
PLA polymers 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 options include recycling into the process or sending to an industrial composting facility, if available; 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. (For example, in the U.S.A., see 40 CFR, Part 261, “Identification and Listing of Hazardous Waste.”) All disposal methods must be in compliance with Federal, State/Provincial, and local laws and regulations.

Environmental Concerns
Generally speaking, lost pellets are not a problem in the environment except under unusual circumstances when they enter the marine environment. They are benign in terms of their physical environmental impact, but if ingested by waterfowl or aquatic life, 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 ocean or any other body of water.

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

Customer Notice
NatureWorks LLC encourages its customers and potential users of its products to review their applications for such products 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 LLC literature, including Material Safety Data sheets, should be consulted prior to the use of the company's products. These are available from your NatureWorks LLC representative.