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## Introduction

This information bulletin describes techniques for piece dyeing and finishing knitted fabrics of Ingeo™ fiber (PLA) filament yarns that have been friction-twist textured. Although Ingeo fiber (PLA) yarns exhibit many attributes similar to other synthetic fibers, they are a new, unique category that requires modified dyeing and finishing techniques to maximize their benefits. As Ingeo™ fiber is from a new category of polymer, PLA (Poly Lactic Acid), there is much information that remains to be understood. The information contained in this bulletin is the most current information available and will be updated as more knowledge is gained.

The use of any environmentally hazardous substances during the processing of Ingeo™ fiber fabrics would be contradictory to the environmentally friendly advantages of the fiber and must be avoided.

## Process Flow

To achieve the maximum benefit of the Ingeo fiber fabric tactile characteristics (hand and drape), it is critical to follow the following process flow.

- Pre-Heat Set
- Scour
- Dye
- Reductive Clear
- Dry
- Post Heat Set
- Finish

## Heat Setting

Friction-twist textured Ingeo filament yarns are not heat set during the texturing process, resulting in high shrinkage potential. Setting of the yarns during texturing is not possible due to the low melt temperature (165° C-170° C) of the PLA polymer. Elimination of this initial fabric heat set step prior to any wet processing of the fabric will result in a fabric which is stiff and harsh.

The Ingeo fiber fabric should be heat set to a width 6%-10% less than the “as-knit” fabric width, and with 6%-10% overfeed. The heat set temperature must not exceed 130° C (actual fabric temperature). Very good results have been obtained with heat set temperatures as low as 120° C-125° C, with dwell time of 30 to 45 seconds at effective temperature.

## Scouring

To minimize the risk of poor dye uniformity, dye blotches, stains and dye fastness issues, all knit fabrics should be scoured to remove knitting lubricants and mill dirt or stains that may have accumulated. Fully acceptable scouring can be achieved with nonionic detergents and soda ash. The pH should be maintained in the 4.5-7.0 level to minimize the risk of hydrolysis of the fibers.

### A recommended procedure is as follows

1. Fill with water and raise to 40<sup>o</sup> C
2. Add Kieralon<sup>®</sup> Jet-B Conc (BASF), or Cyclanon ECO<sup>®</sup> (BASF), or Diadavin UFN (BAYER), or Diadafin UNJ (Bayer) at 0.5-1.0 g/l
3. Adjust pH with Soda Ash or MSP (MonoSodiumPhosphate)
4. Raise temperature to 60<sup>o</sup> C at maximum rate
5. Run 10 minutes
6. Cool to 50<sup>o</sup> C
7. Drain
8. Fill with 40<sup>o</sup> C water and Over-Flow Wash for 10 minutes
9. Drain

## Dyeing

Like polyester, Ingeo™ fiber is dyed using disperse dyes. It has to be noted however, that not all Disperse dyes are suitable to dye PLA. DyStar Co. has pioneered the research in determining a selection of Dispersed dyes, from their dye range offering, suitable for the coloration of the Ingeo fiber. This selection is available from the DyStar Co. as the “Ingeo™ fiber Coloration Pack”. Other dye supply companies that have worked on the coloration of the Ingeo fiber are: Ciba, Clariant, Yorkshire Chemicals, and M.Dohmen. Due to the unique crystalline morphology of PLA, the critical temperature range over which dye exhausts (strike rate) on to Ingeo fiber is about 20<sup>o</sup> C lower than that of polyester. As a result of this faster strike rate of Ingeo fiber, the use of levelers is not necessary and is not recommended. The use of carriers is unnecessary and contrary to the environmentally friendly perspective of the Ingeo™ fiber and **must** be avoided. In addition Defoamers that would render the fabric Hydrophobic should be avoided in order to maintain the moisture management characteristics of the

Ingeo™ fiber fabrics.

### A typical dye procedure is as follows:

1. Set bath 40<sup>o</sup> C
2. Add 6 g/l MSP (MonoSodiumPhosphate)\*.
3. Add Dispersant (such as Basol WS liquid by Bayer)  
Dispersant (follow manufacturer’s recommendation on level)  
Also the addition of a fiber to metal lubricant is recommended. However, as with defoamers, not diminishing the hydrophilic character of the Ingeo fiber should be considered. Milease-T (initially from ICI) or similar in chemistry auxiliaries have been found to be suitable for this application.
4. Run 5-10 minutes to ensure good mixing
5. Add dyes
6. Check pH and adjust to 4.5-5.0 as necessary with MSP or Soda Ash
7. Raise temperature to 60<sup>o</sup> C at 2-3<sup>o</sup> C/minute
8. Raise temperature to 110<sup>o</sup> C at 1<sup>o</sup> C/minute (for dark shades such as Navy and Black raise temperature to 115<sup>o</sup>)
9. Run for 30-45 minutes depending upon dyes used and shade desired
10. Cool to 60<sup>o</sup> C at 1-2<sup>o</sup> C/minute
11. Drain
12. Fill with 50<sup>o</sup> C and Over-Flow wash for 10 minutes
13. Drain

\* Normally, 2 g/l of MSP is sufficient to lower the pH to about 5.0. However, it was noted that during the dye cycle of the Ingeo™ fiber fabrics the pH of the bath would progressively drop to an undesirable level. It was found that an additional 4 g/l of MSP would provide the buffering needed to maintain a pH level of 4.3 to 4.8 during the entire dye cycle.

## Reductive Clear

To remove surface dyes in order to obtain the best wet fastness properties possible, a reductive clearing scour is recommended after dyeing.

**A recommended Reductive Clear procedure is as follows :**

1. Raise temperature to 40<sup>o</sup> C at maximum rate
2. Add from the addition tank:

<b>For Light to Medium Colors</b>	<b>For Dark Colors</b>
2 g/l Cyclanon ECO	1.5 g/l Soda Ash
2 g/l MSP	2 g/l Hydrosulfite Con.
3. Raise temperature to 60<sup>o</sup> at 2<sup>o</sup> C
4. Run for 15 minutes
5. Cool to 50<sup>o</sup> C at 3<sup>o</sup> C
6. Drain
7. Over-Flow wash at 50<sup>o</sup> C for 10 minutes
8. Over-Flow wash at 30<sup>o</sup> C for 5 minutes
9. Drain and Unload fabric.

## Post Heat Setting

After dyeing, fabrics with friction-twist textured Ingeo fiber filament yarns will have to be heat set again. The fabric temperature must not exceed 130<sup>o</sup> C.

Factors influencing the post heat setting conditions for fabric width and overfeed include the following:

- Wet width of fabric
- Desired finished construction (stitches/inch and wales/inch)
- Smoothness of fabric after dyeing
- Desired drape of finished fabric
- Desired hand of finished fabric
- Desired bulk in finished fabric

As can be seen from the above, conditions for the final heat setting of the fabric are set by some experimentation before committing the entire dye lot.

## Fabric Finishing

Moisture management and flammability are two inherent attributes of PLA fabrics and must not be inhibited by the introduction of any chemicals or auxiliaries during the dyeing or finishing process. Until the effects of various additives are fully understood, the addition of any additives (hydrophilic or hydrophobic) should be done in consultation with NatureWorks LLC technical representatives.

Dyed fabrics of PLA yarns can be finished in the same manner as other synthetic fibers. Like other synthetic fibers, consideration of final finishing procedures should be taken into account when designing the initial fabric construction.

Any fabric surface finishing process **must** take into account the low melt temperature (165<sup>o</sup> C-170<sup>o</sup> C) of the Ingeo fiber. Excessive energy from surface treatment can result in a fabric that is harsh and stiff due to fusing of filaments from the generated heat.

## Trade References

Additional information concerning dyes, dye bath auxiliaries and the dye process can be obtained from NatureWorks LLC web site ([www.natureworkspla.com](http://www.natureworkspla.com)).

### 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 170°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 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.

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