Ingeo™ Fibre Apparel Product Guidelines

Dyeing and Finishing

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2. General considerations for dye and finish
3. Dye selection
4. Shades and washing fastness
5. Dye cycles
   - 100% Ingeo™ fibre
   - Ingeo™ fibre / cotton
   - Ingeo™ fibre / wool
   - Package dye
6. Process routes
7. Atmospheric dyeing
Apparel Product Guidelines

1. Introduction to Ingeo™ fibres
Ingeo™ fibre

- The only synthetic fibre commercially available in bulk quantities, that is made entirely from annually renewable raw materials - not oil

[Diagram showing the process of converting corn into Ingeo™ fibre]

Not only from nature - but also back to nature
Ingeo™ fibre

From nature, and back to nature - plus performance:

- Excellent wicking properties
- Moisture management
- Low odor retention
- Does not support bacterial growth
- Hypoallergenic
- Rapid soil release
- Quick drying
- Excellent after wash appearance

And all supported by detailed technical bulletins ……
Ingeo™ fibre

...... selected technical bulletins

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These and more available on www.ingeofibers.com
Apparel Product Guidelines

2. Dyeing - general considerations
General considerations:
1) Lower melt point (170°C)

• Safe, effective, heat setting – 130°C for 30sec
• No high temperature heat set for blends with:
  ➢ Nylon, PET
  ➢ Elastane (~3% with no heat set OK)
General considerations:

2) Sensitivity to high temperature and alkali conditions:

• Safe, effective dyeing - 110°C for up to 30min
• Safe, effective processes developed:
  ➢ Scour
  ➢ Reduction clear
  ➢ Reactive dye
  ➢ Cotton pre bleach
  ➢ etc
General considerations:
3) disperse dyes behave differently from PET on Ingeo™ fibre:

- Not all dyes good for PET are good for Ingeo™ fibre
  - Different exhaustion (some correlation with acetate dyeing)

- Dye shades are different on Ingeo™ fibre compared with PET:
  - Brighter than on PET
  - Higher visual colour yield than PET

DYE SELECTION FOR INGEO™ FIBRE DYEING IS ESSENTIAL
Apparel Product Guidelines

3. Dye selection
Different dyes – different exhaustion

- PLA 110°C, 30min
- PETb 130°C, 30min
Ingeo™ fibre dyeing and finishing

Same dye – different shade

![Chemical structure of dye](image)

![Graph showing absorbance spectra for PLA and PET](image)
Selected dyestuff for high wash fastness - DyStar

**Ternaries**
- Dianix Flavine XF
- Dianix Yellow Brown XF
- Dianix Deep Red SF
- Dianix Blue XF

**Support dyes**
- Dianix Scarlet XF
- Dianix Rubine XFN
- Dianix Turquoise XF
- Dianix Navy XF
- Dianix Black XF
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<th>Ternaries</th>
<th>Support dyes</th>
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<td>Dianix Orange AM-SLR</td>
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<td>Dianix Blue AM-SLR</td>
<td>Dianix Navy AM-G</td>
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<td>Dianix Yellow AM-42</td>
<td>Dianix Black AM-B</td>
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<td>Dianix Dark Red AM-2B</td>
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<td>Dianix Blue AM-77</td>
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</table>
## Selected dyestuff for standard fastness - DyStar

### Ternaries
- Dianix Yellow AC-E new
- Dianix Red AC-E 01
- Dianix Blue AC-E
- Dianix Yellow Brown CC
- Dianix Rubine C-B 150%
- Dianix Blue K-FBL

### Support dyes
- Dianix Flavine XF
- Dianix Red C-4G 150%
- Dianix Violet S-4R
- Dianix Dark Blue SE-3RT
- Dianix Turquoise S-BG
DyStar Colouration Package

Comprehensive processing suggestions from DyStar

CD ROM with Colour matching data base
Apparel Product Guidelines

4. Shades and washing fastness
Shades and fastness

AATCC 2A (50°C) fastness on pale / medium shades
Shades and fastness

AATCC 1A (40°C) fastness on medium / dark shades
Apparel Product Guidelines

5. Process routes
Typical Route

Scour / Dye / (Reduction clear)  
↓
Hydro  
↓
Relax dry  
↓
Slit  
↓
Pad soften  
↓
Stenter dry / heat set  
→ 130°C for 20 - 30sec
Typical Route

Knit
  ↓
Slit
  ↓
Pre heat set
  ↓
Scour / dye / (reduction clear)
  ↓
Relax dry
  ↓
Pad soften
  ↓
Dry and post heat set

125°C for 20 - 30 sec

130°C for 20 - 30 sec
Ingeo™ fibre woven fabric dyeing

- Many variations in woven fabrics finishing routes
- Suggested routing for trial start point only:

  Stenter pre heat set
  \[\text{\underline{\text{\arrowdown}}\text{\underline{\text{\arrowdown}}}}\text{\underline{\text{\arrowdown}}\text{\underline{\text{\arrowdown}}}}
  \text{Open width desize/scour}
  \text{\underline{\text{\arrowdown}}}
  \text{Jet dye}
  + reduction clear (dark shades)
  \text{\underline{\text{\arrowdown}}}
  \text{Hydro extract}
  \text{\underline{\text{\arrowdown}}}
  \text{Detwist/open}
  \text{\underline{\text{\arrowdown}}}
  \text{Pad soften}
  + stenter dry / post heat set
  \text{\underline{\text{\arrowdown}}}
  \text{130°C for 20 - 30 sec
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• Many variations in woven fabrics finishing routes
• Suggested routing for trial start point only:
Apparel Product Guidelines

6. Dye cycles

- 100% Ingeo™ fibre
- Ingeo™ fibre / cotton
- Ingeo™ fibre / wool
- Package dye
100% Ingeo™ fibre

Scour

- 50°C
- 60-70°C for 15 minutes
- 50°C

1 g/l Kieralon MFB (BASF)
1 g/l Na₂CO₃
100% Ingeo™ fibre dyeing and finishing - Dye cycles

Dye

- 1 g/l Levegal DLP (Bayer)
- Acetic acid/ sodium acetate to pH 4-5
- Dianix dyes

- 40°C
- 60°C: 1-2°C/min
- 110°C*: 15-30 mins
- 50°C: 3°C/min

* Very heavy shades may be dyed for 30 min at 115°C for maximum colour yield
100% Ingeo™ fibre

Reduction Clear (dark shades)

- 50°C for 15 minutes
- 60-70°C
- 50°C

Chemicals:
- 2 g/l sodium hydrosulphite
- 2 g/l Na₂CO₃
Apparel Product Guidelines

6. Dye cycles

- 100% Ingeo™ fibre
- Ingeo™ fibre / cotton
- Ingeo™ fibre / wool
- Package dye
Ingeo™ fibre dyeing and finishing - Dye cycles

Ingeo™ fibre / cotton
Pre bleach - Peroxide

- 40°C
- 95°C 30mins
- 50°C
- 70°C 20min
- 50°C
- 40°C

3 g/l Na₂CO₃
3 cc/l Hydrogen peroxide (35%)
0.5g/l Baystabil LFS-T
1cc/l Baysolex EXT

0.3cc/l Acetic Acid
Ingeo™ fibre / cotton

Alternative pre bleach - TAED (tetra acetyl ethylene diamine)
Good white base with decreased hydrolysis

40°C
0.5g/l Na₂CO₃
5.0cc/l Hydrogen peroxide (35%)
2.0g/l Warwick T202 (TAED)
1.0g/l Pitchrun L300 (scouring agent)
1.0g/l Neocrystal CG2000 (sequestering agent)
1.0g/l Neorate PLC7000 (peroxide stabiliser)

95°C 40mins
40°C
50°C
20min
70°C
50°C
40°C

Neutralise with acetic acid if needed
Ingeo™ fibre dyeing and finishing - Dye cycles

**Ingeo™ fibre/cotton**
Disperse / Direct - one bath

- 10 g/l sodium sulphate
- Dianix/Sirius dyes
- 1 g/l Levegal DLP (Bayer)
- 2 g/l Persoftal L (Bayer)
- Acetic acid/sodium acetate to pH 5

Temperature and Heating Rates:
- 40°C
- 60°C (1-2°C/min)
- 110°C (15-30min)
- 100°C (1°C/min)
- 80°C (3°C/min)
- 50°C
**Ingeo™ fibre dyeing and finishing - Dye cycles**

**Ingeo™ fibre /cotton**
Disperse / Direct - one bath + after treat for darker shades

<table>
<thead>
<tr>
<th>Time (min)</th>
<th>40°C</th>
<th>Sirius B* over 5min</th>
<th>NaOH** over 10min</th>
<th>Rinse</th>
<th>Neutralise + Acetic Acid</th>
</tr>
</thead>
</table>

* % Sirius B = 1.0 + (1.5 x % of total direct dye)

*Minimum 1.5%*/ maximum 7.0%

** cc/l NaOH (40%) = 1 + amount of Sirius B (in %)

*Minimum 2.5%*
Ingeo™ fibre dyeing and finishing - Dye cycles

Ingeo™ fibre/ cotton (start point for trials only)
Disperse / Reactive - stage 1

- 40°C
- 60°C 1-2°C/min
- 110°C 30mins
- 3°C/min
- 50°C
- 60-70°C 15mins
- 50°C

Dianix dyes

1 g/l Levegal DLP (Bayer)
2 g/l Persoftal L (Bayer)
Acetic acid/sodium acetate to pH 5

2 g/l sodium hydrosulphite
2 g/l Na₂CO₃
Ingeo™ / cotton (start point for trials only)
Disperse / reactive - stage 2

- Sodium sulphate
- Remazol RGB dyes
- NaOH
- Acetic acid to pH 6
Apparel Product Guidelines

6. Dye cycles

- 100% Ingeo™ fibre
- Ingeo™ fibre / cotton
- Ingeo™ fibre / wool
- Package dye

NB – there has been limited work on dyeing of Ingeo™ fibre blended with wool, and the information provided is based on our best knowledge to date. It can only be taken as guidelines for development work.
General considerations -
Ingeo™ fibre and wool are highly compatible

- Both fibres dislike high temperature and alkali combinations:
  - The pH dyeing conditions for Ingeo™ fibre are good for wool
  - The lower dyeing temperature of Ingeo™ fibre affords more protection to wool than PET blend dyeing

- Stress / strain curves are similar:
  - Indicates compatibility in most stages of processing
General considerations for Ingeo™ fibre / wool blend dyeing:

- Disperse dyes will cross stain on wool
  - Can cause fastness issues
  - (Similar issues on dyeing PET / wool blends)
  - After treatment to minimise cross staining is vital (particularly for dark shades)
- Wash fastness is limited to 40°C tests (e.g. AATCC 1A / C06, A2S)
### Ingeo™ fibre / wool blends

**Suggested dark shade recipes for Ingeo™ / wool - standard fastness**

*(start point for lab trials only)*

<table>
<thead>
<tr>
<th>Black</th>
<th>Navy</th>
</tr>
</thead>
<tbody>
<tr>
<td>• Dianix Flavine XF 0.19%</td>
<td>• Dianix Flavine XF 0.18%</td>
</tr>
<tr>
<td>• Dianix Yellow Brown CC 1.45%</td>
<td>• Dianix Turquoise S-BG 0.42%</td>
</tr>
<tr>
<td>• Dianix Turquoise S-BG 0.29%</td>
<td>• Dianix Dark Blue SE-3RT 2.36%</td>
</tr>
<tr>
<td>• Dianix Dark blue SE-3RT 2.20%</td>
<td>• Isolan Dark Blue 2S-GL 1.08%</td>
</tr>
<tr>
<td>• Isolan Black 2S-LD 1.13%</td>
<td></td>
</tr>
</tbody>
</table>
Ingeo™ fibre / wool blends

Scour (DyStar)

- 50 °C
- 60 °C for 15 min
- Rinse at 50 °C

1 g/l Diadavin® UNJ (Bayer/Sybron) or Kieralon® MFB (BASF)
2 g/l soda ash.
Ingeo™ fibre / wool blends

Dye (DyStar)

1) 1 % Avolan UL® DLP (Bayer/Sybron)
   1 g/l Avolan IS® (Bayer/Sybron)
   pH 4.5-5 acetic acid
2) X % Dianix dyes
3) Y % Supralan / Isolan dyes

40 °C
1-2 °C/min
30 min
110 °C
3 °C/min

1 2 3
Ingeo™ fibre / wool blends

Aftertreat (DyStar)

1.5 g/l Blankit® IN  
pH 8.5-9

rinse at 50 °C

rinse at 30 °C

rinse at 50 °C
Apparel Product Guidelines

6. Dye cycles

- 100% Ingeo™ fibre
- Ingeo™ fibre / cotton
- Ingeo™ fibre / wool
- Package dye
• Limited experience
  ➢ Winding
    – Density as PET ~ 0.35gm/cc
    – Minimal yarn shrinkage = minimal increase in package density after dye
  ➢ Dyeing
    – Suggested dye cycle (start point for trials only) as follows
Ingeo™ fibre yarn package dye cycle

(scour, reduction clear etc - as for fabrics)

NB this is a suggested dye cycle as a start point for trials only

Flow rate: 3 min in to out, 2 minutes out to in

Similar cycle is also appropriate for top dye
**Ingeo™ fibre yarn package dye cycle**

- alternative system with MSP (Mono sodium phosphate) buffer
  (scour, reduction clear etc - as for fabrics)

*NB this is a suggested dye cycle as a start point for trials only*

- MSP to pH 5.0 - 5.3
- Plus additional 4 g/l MSP
- 2.0 g/l Basol WS
- 0.25 g/l Milease T (Lubricant)

**Flow rate:**
- 3 min in to out,
- 2 minutes out to in
Apparel Product Guidelines

7. Atmospheric dyeing
• Limited lab work suggests:
  - Selected dyes give adequate exhaustion at 100°C, 30min
  - Only pale to medium shades
  - AATCC 1A fastness

• Trichromatic combination as start point for trials only:

  - Dianix Deep Red SF
  - Dianix Yellow Brown XF
  - Dianix Dark Blue SE-3RT

  Significant for sweater yarn and garment dyeing
Ingeo™ fibre - atmospheric dyeing

• Process cycle

- Scour – as for 100% Ingeo™ fibre
- Dye - as for 100% Ingeo™ fibre, but hold at 98°C for 30min instead of 110°C
- Reduction clear - pale to medium shades, so not normally needed .... or mild process, eg Cyclanon ECO (DyStar)
IMPORTANT NOTES:
1) The information provided in this document is given in good faith based on the best knowledge of current technology.
The information provided can only be taken as a start point for trials to establish production routes for Ingeo™ fibre products.
Cargill Dow cannot be held responsible for any claims arising from the information contained within this document, howsoever caused.

2) Yarns produced from different merges, and from different suppliers of INGEO(TM) PLA fibers, may vary in dye uptake, which can influence the reproducibility of shades. It is therefore recommended that before carrying out any production dyeing on new merges or fiber from new suppliers, that the dye recipe is verified by a laboratory check.
This is a similar procedure to other man made fibers, and so similar practices should be adopted to control color reproducibility at the final coloration stage.

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