Performance Comparison of Water-Quench versus Air Quench Blown Films

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1. Brampton Engineering
2. Kuraray America
3. DuPont
Outline

- Water quench versus air quench blown film process
  - Outline of extrusion experiment
- Evaluation of film samples
  - Physical attributes
  - Performance
  - Morphology
- Conclusions
Aquafrost™ water quench blown film process

(a) Bubble inflation
(b) Water quenching
(c) Collapsing frame
Water quenched blown film process

- The downward extruded and water quenched blown film system is a unique process that marries the advantages of both a cast and a conventional blown film process.

- WQ process retains the benefits of improved clarity, improved thermoformability and reduced curl from a cast film process while maintaining the balanced orientation and the process flexibility of a blown film process.
Water quench blown film process

Key process variables: Water ring distance from die, water quench temperature and film annealing temperature
Outline of unique extrusion experiment

- Water quench vs. blown film extrusion lines
  - 9 extruder, 9 layer lines in same facility
- Film structures
  - Coextruded 100 µm thick 9 layer structures
- Performance evaluation
  - Oxygen barrier and physical properties
  - Thermoforming and biaxial orientation tests
- Structure-properties relationship
  - Investigation of crystalline structure
**Film Structures**

<table>
<thead>
<tr>
<th>Structure</th>
<th>Layer 1</th>
<th>Layer 2</th>
<th>Layer 3</th>
<th>Layer 4</th>
<th>Layer 5</th>
<th>Layer 6</th>
<th>Layer 7</th>
<th>Layer 8</th>
<th>Layer 9</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>PE</td>
<td>PE</td>
<td>T</td>
<td>PA</td>
<td>EVOH-1</td>
<td>PA</td>
<td>T</td>
<td>PE</td>
<td>PE</td>
</tr>
<tr>
<td>Thickness (%)</td>
<td>15</td>
<td>8</td>
<td>8</td>
<td>15</td>
<td>8</td>
<td>15</td>
<td>8</td>
<td>8</td>
<td>15</td>
</tr>
<tr>
<td>B</td>
<td>P</td>
<td>T</td>
<td>PA</td>
<td>EVOH-1</td>
<td>PA</td>
<td>T</td>
<td>PE</td>
<td>T</td>
<td>PA</td>
</tr>
<tr>
<td>Thickness (%)</td>
<td>15</td>
<td>10</td>
<td>10</td>
<td>8</td>
<td>10</td>
<td>8</td>
<td>14</td>
<td>10</td>
<td>15</td>
</tr>
<tr>
<td>C</td>
<td>PE</td>
<td>PE</td>
<td>PE</td>
<td>T</td>
<td>EVOH-1</td>
<td>T</td>
<td>PE</td>
<td>PE</td>
<td>PE</td>
</tr>
<tr>
<td>Thickness (%)</td>
<td>13</td>
<td>15</td>
<td>8</td>
<td>10</td>
<td>8</td>
<td>10</td>
<td>8</td>
<td>15</td>
<td>13</td>
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</table>

PA = polyamide 6  
EVOH-1 = 38mol% ethylene-vinyl alcohol  
EVOH-2 = a modified 32mol% EVOH  
PE = octene-LLDPE  
T = maleic-anhydride modified PE  
P = polyolefin plastomer

**Key**
- Structure A is PA/EVOH/PA core with PE skins
- Structure B is PA/EVOH/PA core with PA skin
- Structure C is PE/EVOH/PE
PART I RESULTS
Structure A is PA/EVOH/PA core with PE skins
Structure B is PA/EVOH/PA core with PA skin

Haze (%)

Structure A

15.5

4.1

Structure B

17.8

7.4
Water vapor barrier of flat films

Structure A is PA/EVOH/PA core with PE skins
Structure B is PA/EVOH/PA core with PA skin
Oxygen barrier of flat films

Structure A is PA/EVOH/PA core with PE skins
Structure B is PA/EVOH/PA core with PA skin
Evaluation of crystalline structure

Above: Birefringence micrographs from trial – Structure A

Right: monolayer nylon films
WAXD analysis of Structure A

Scattering Angle (Two Theta, Degrees)
Intensity

- PE
- EVOH
- Nylon
Part I Summary

- WQ flat films have
  - Lower haze, water vapor and oxygen barrier in both symmetric and asymmetric structures

- AQ flat films have
  - Higher haze, water vapor and oxygen barrier in both structures

- Films produced at various water quench temperatures, water tank positions and annealing temperatures had insignificant differences in OTR

- Optical Microscopy and Wide-Angle X-Ray Diffraction (WAXD) analysis.
  - The results indicated that the polyamide, EVOH and PE in the AQ samples have a higher degree of crystallinity than their WQ counterparts.
PART II RESULTS
Forming experiments

Forming tests conducted on batch ZED former and Multivac horizontal FFS former.
Barrier of formed packages was tested at 20°C / 65% RH OUT, 100% RH IN.
Oxygen Barrier – Formed Packages

![Bar chart showing OTR (cc/[package.day]) for Structure A with AQ and WQ.

- AQ: 0.32
- WQ: 0.26]
Forming Performance Structure A

Multivac 4 pocket set-up of 135 x 125 x 85mm
Heating time 1 second
Forming time 2 seconds
Gauge Distribution – formed films

Water quenched films had superior gauge distribution

AQ – Structure A

WQ – Structure A
Forming Performance Structure B

Multivac 4 pocket set-up of 135 x 125 x 85mm
Heating time 1 second
Forming time 2 seconds
Part II Summary

- **Oxygen barrier of formed package from WQ film lower than AQ film**
  - WQ film has better thermoformability than the AQ film and hence the formed package of WQ film is able to retain its barrier properties
  - Shape of the formed package from the WQ film was more appealing than that of the AQ film

- **Consistent with lower crystallinity, the water-quench technology allows barrier films to form more evenly at lower forming temperatures and to form over wider temperature range.**

- **Note that Structure C films (without PA) did not form well at this depth on horizontal FFS Multivac™ former**
  - Nylon is required to reach deep draw ratios with even gauge distribution
PART III RESULTS
Biaxial Stretcher

Test Conditions
Preheat 100°C for 20 seconds
Orientation at constant strain rate to 2 x 2 or 3 x 3 orientation
Biaxial orientation – AQ and WQ - Structure A

MD & TD Stress
Biaxial orientation – AQ and WQ - Structure A

Total Stress

![Graph showing stress vs. draw ratio for AQ and WQ in Structure A.](image-url)
Biaxial orientation – AQ and WQ - Structure B

Total Stress

![Graph showing stress vs. draw ratio for AQ and WQ.]
Biaxial orientation – WQ - Structure B
Total Stress at Condition 1 & 2

![Graph showing total stress at condition 1 & 2 for WQ-1 and WQ-3. The x-axis represents draw ratio, and the y-axis represents stress (MPa).]
Biaxial orientation – AQ - Structure C
Total Stress with EVOH 1 vs. 2

![Graph showing stress vs. draw ratio for EVOH 1 and EVOH 2](chart.png)
Summary of biaxial orientation test results
3 x 3 draw ratio at 100°C – 20s

<table>
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<tr>
<th>Structure</th>
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<th>B</th>
<th>C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Extrusion Process</td>
<td>AQ</td>
<td>WQ</td>
<td>AQ</td>
</tr>
<tr>
<td>Yield Stress (MPa)</td>
<td>12.2</td>
<td>8.0</td>
<td>18.0</td>
</tr>
<tr>
<td>Ultimate Engineering Stress (MPa)</td>
<td>29.3</td>
<td>24.8</td>
<td>39.4</td>
</tr>
<tr>
<td>Modulus of resilience (MPa)</td>
<td>0.96</td>
<td>.43</td>
<td>1.1</td>
</tr>
<tr>
<td>Modulus of Toughness (MPa)</td>
<td>39.4</td>
<td>29.4</td>
<td>57.2</td>
</tr>
</tbody>
</table>

AQ films had higher resistance to deformation than WQ films.
Oxygen barrier before and after orientation

OTR at 30°C / 85%RH

OTR (cc.20mics/sq.m.day)

Unoriented

Oriented

A-AQ
A-WQ
B-AQ
B-WQ
C EVOH-1
C EVOH-2
Part III Summary

- Structure A resistance to deformation of AQ films was higher than the WQ films. Total stress and modulus of toughness also higher.
- Structure B AQ and WQ films replicated results of Structure A.
  - Conditions of water ring position, water ring and annealing station temperature had no significant effect on orientability.
- C EVOH-1 and C EVOH-2 structures oriented similarly with low resistance to deformation.
  - Significantly lower than both the structures containing nylon (A and B).
- Barrier of all films after orientation almost equal.
  - Suggests that heating and then orientation of the films created conditions of annealing and strain induced crystallization that tended to produce similar morphologies and thus barrier of the oriented films.
Conclusion

- Properties of water quenched films were quite different from the air quenched films
  - Water quenched films had lower resistance to orientation, improved thermoformability and reduced haze.
- Barrier of the water quenched films to oxygen and moisture was lower than that of the air quenched films before forming.
- Differences in key physical properties was correlated to variation in polymer crystallinity
- Barrier testing of oriented films and formed packages suggest that orientation tends to equalize crystallinity of the EVOH
Acknowledgements

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Thank you

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