Gel Troubleshooting

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Definition of a Gel

- Film Imperfections or defects developed during forming due to disturbances in the polymer flow
Classification of Film Imperfections

- Contamination
- Un-melt
- Un-mix
- Thermal degradation
Origins
Gels can be created or introduced during

- Polymerization
- Equipment-Screw Design
- Fabrication
Contamination
Gauge Bands/Gels

• Contamination
  – Fiber
Fiber Gel After Melting

Polarized light (w/ 1/4 filter)
Gauge Bands/Gels

• Contamination
  – Fiber
  – Resin Contamination
Resin Contamination

**LDPE**
- Polyolefin
- More Crystalline
- Thermally stable
- Adhesion through oxidation
- 600°F - 630°F processing temperature

**EVA**
- Acid functional groups
- Less Crystalline
- Not thermally stable
- Adhesion through vinyl acetate groups
- 450°F processing temperature
Cross Contamination Solutions

- Better Housekeeping
- Better purging
- Work with resin companies
Gauge Bands/Gels

• Contamination
  – Fiber
  – Resin Contamination

• Un-melt and un-mix
Hot Stage Microscopy

- Slowly heat sample to above melting point.
- Transmit light through cross-polarized filters.
- Observe melting point of film/gel and note any birefringence effects.
Unmixed Gel Characterization

50°C  ->  150°C  ->  50°C

Unmixed Gel
Gauge Bands/Gels

• **Contamination**
  – Fibers
  – Cross contamination w/other Polymers

• **Un-melt & un-mixed**
  – Bad Concentrate
Mixing
Master Batch Concentrates

• Compatibility of base resin
• quality of dispersive mixing
  – agglomerates
Gauge Bands/Gels

• Contamination
  – Fibers
  – Cross contamination w/other Polymers

• Un-melt & un-mixed
  – Bad Concentrate
  – Blends with different melting rate
Mixing
Polymer Blends and Additives

- Thermodynamic compatibility of components (conductivity)
- Large differences in melting points of blend components
- Large viscosity ratios between minor and major components
Gauge Bands/Gels

• Contamination
  – Fibers
  – Cross contamination w/other Polymers

• Un-melt & un-mixed
  – Bad Concentrate
  – Blends with different melting rate
  – Fluff
Contiguous Solids Melting
Gauge Bands/Gels

• Contamination
  – Fibers
  – Cross contamination w/other Polymers

• Un-melt & un-mixed
  – Bad Concentrate
  – Blends with different melting rate
  – Fluff
  – Bad temperature profile
Gauge Bands/Gels

• Contamination
  – Fibers
  – Cross contamination w/other Polymers

• Un-melt & un-mixed
  – Bad Concentrate
  – Blends with different melting rate
  – Fluff
  – Bad temperature profile
  – Inconsistent granular size
Gauge Bands/Gels

- Contamination
  - Fibers
  - Cross contamination w/other Polymers
- Un-melt & un-mixed
  - Bad Concentrate
  - Blends with different melting rate
  - Fluff
  - Bad temperature profile
  - Inconsistent granular size
  - Screw design
Screw Design

• Improper design for the resin can contribute to:
  – SBB causing encapsulation of un-melt solids
  – SBB also causes non uniform mixing history
  – Not providing enough shear to melt resin
Observed Solid Bed Break-up

CONVENTIONAL SCREW
Gauge Bands/Gels

- Contamination
  - Fiber
  - Resin Contamination
- Un-melt and un-mix
- Thermal degradation
Polymer Degradation is a Function of

- Time
- Temperature
- Shear Conditions

All polymers degrade as a function of time, temperature, and shear conditions

Degradation → Gels
Types of Polymer Degradation

• Thermal - High temperatures
• Chain Scission - High stress fields
• Cross-linking - Un-saturation
• Oxidation - Oxygen
Example of crosslinking

- Ethylene Vinyl Acetate (EVA)
  - \(-(\text{CH}_2-\text{CH}_2)_m-(\text{CH-CH}_3\text{COO})-\)

- Temperature causes decarboxylation which result in releasing Acetate, CO\(_2\), and primary free radicals
EVA Degradation

• Creates acetic acid at LDPE process temperatures.
EVA - Effect of Over Heating...

- Ethylene Vinyl Acetate (EVA)
  - $(\text{CH}_2\text{-CH}_2)_m(\text{CH}-\text{CH}_3\text{COO})_m$
- Temperature causes decarboxylation which result in releasing Acetate, CO$_2$, and primary free radicals
Side Reaction

\[
\text{[Chemical structure]} + \text{[Chemical structure]} \rightarrow \text{[Chemical structure]}
\]
Lightly Crosslinked Gel

- **50°C** → **Melt** → **150°C** → **Lightly crosslinked** → **50°C**
What does crosslinked mean?

• Too much energy provided to the polymer.
Too much heat

• Shear
Cross Section of Single-Screw Extruder

- **State of Material**: Fluid | Fluid/Solid | Solid
- **Melt Generator**
- **Melting/Plasticating**
- **Conveying**
- **Output and Metering Zone**
- **Alternative-Mixing**
- **Transition**
- **Feed Zone**

State of material: Fluid | Fluid/Solid | Solid

- **Drive**
- **Gear Box**

Pressure utilize

Z6

- **Output and Metering Zone**
- **Alternative-Mixing**

Z1
Too much heat

- Shear
- Heaters
Too much heat

- Shear
- Heaters
  - T/C not in bottom of well
T/C Location
Too much heat

- Shear
- Heaters
  - T/C not in bottom of well
- Extruder Residence time
HEAT TRANSFER IN AN EXTRUDER

Heat in/Cold out

heaters-coolers

Enthalpy of Resin

cooling

screw

drive

Mechanical Energy
Gel Hang-up at adapter
Gauge Bands/Gels

- Fibers/Contamination
- Cross contamination w/other Polymers
- Un-melt
- Cross-Linked
- Oxidation
Oxidized Gel at 130 C with polarized light (w/ 1/4 filter)
Oxidized Gels

- Formed very rapidly in the presence of oxygen

- Corrective Action
  - Improve stabilizer package
  - Reduce exposure to oxygen during processing
  - Reduce temperature
Oxidation of resin

- Incorrect shut down (not capping die)
- Incorrect start-up with cleaned die
Gauge Bands/Die Lines

- Hang up of gels
- Temperature variation
- Automatic die control
- Static Mixer position
- Hang Ups in flow path
Gel Hang-up at adapter
Solving Gel Problems

• Identify the source of the Imperfection
  – Un-melt
  – Degradation (Cross link or Oxidized)
  – Contamination (Dust, Dirt, Fiber)
  – Agglomeration (Color, Additives)

• Find the Source
  – Resin
  – Equipment
  – Blends/Additives
Tools to detect gel problems

- Hot stage microscopy
- IR analysis
- Chemical Analysis
- DSC unit
Thank You

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Please remember to turn in your evaluation sheet...