EXTRUSION DIVISION Newsletter

Volume 39
Number 1

Winter 2014

Inside this issue:

Message from the Technical Program Chair for ANTEC 2014

This April the best action in Vegas will be happening off the strip, as SPE presents ANTEC 2014 in sunny Las Vegas at the Rio All-Suites Hotel! The Extrusion Division will have quite an exciting and ambitious technical program, with something of interest for everyone. Our program will include eleven technical sessions that you don't want to miss. Session topics will include:

- Single Screw Extrusion
- Twin Screw Extrusion
- Properties, Structure, Processing
- Dies
- ***Special Session*** - Hot Melt(Pharma) Extrusion
- ***Special Session*** - Dedicated to the memory of Dr. Marino Xanthos
- Joint Session with Flexible Packaging Division

In addition, there will be two Tutorial Sessions. We have added one session each for Single and Twin Screw Extrusion. Look for lively and informative talks by industry veterans, with a panel discussion at the end of the sessions where you can discuss your questions with the experts.

Tuesday evening of ANTEC, the Extrusion Division will hold their Annual Business Meeting and Awards Ceremony. This year we are honored to kick off the meeting with a talk from this year's SPE Businessman of the Year Award Winner, Mr. Baldev L. Boolani. And the meeting will end with our Annual Awards Reception. Always a great opportunity to network with old friends and meet new contacts!

If "what happens in Vegas stays in Vegas" then you don't want to miss what promises to be a great ANTEC program! ANTEC registration is open now. Check it out today at www.4spe.org

David Anzini
Extrusion Division TPC, ANTEC 2014

Don't be left out!

ANTEC 2014

Las Vegas, Nevada

April 28 – April 30, 2014

- Would you like to meet with fascinating, informed and creative colleagues from around the world?
- Would you like to look for partners to collaborate on future projects?
- Would you like to broaden your understanding of the plastics industry?
- **There will be 11 sessions from the Extrusion Division including single screw, twin screw, die design, film and sheet, special sessions on hot melt extrusion and tutorial sessions! Come be a part of the knowledge sharing!**

ANTEC 2014 Las Vegas is for Young Professionals!

We've listened to the feedback our younger SPE members have provided from previous ANTEC conferences. So we just wanted you to know we're offering some new, fun and engaging activities at ANTEC 2014 (April 28-30), specifically for young plastics professionals:

- **Plastics Race** - See Las Vegas through the eyes of a plastics engineer as you team up and roam the Vegas Strip to compete for some awesome prizes!
- **Panel Discussion** - Participate in a lively discussion, ask your industry questions, and gain the knowledge you've been looking for including career tips and tricks relevant to you, not that generic advice you find online.
- **Celebration Dinner** - Network over an enjoyable dinner with your fellow peers, future associates and industry veterans. Prizes, awards and more!
- **Mission Possible 2.0** - Your chance to make ANTEC 2015 and SPE what you want it to be.
- **Speed Interviews** - Sharpen your skills at on-site screening visits with prospective employers.

So come on out, and see the new and improved ANTEC!

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SOCIETY OF PLASTICS ENGINEERS

Check it out online > **www.antec.ws**

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EXTRUSION DIVISION ANTEC 2014 PROGRAM

Monday Morning, April 28, 2014

Monday Morning, April 28, 2014	Properties, Structure, Processing	Moderator: Dr. Maria Noriega, ICIPC
8:30-9:00	Aumnate, Chuanchom	Understanding Processability of PA11 via Rheology
9:00-9:30	Inn, Yongwoo	Flow-Induced Fractionation of Bimodal Metallocene Polyethylene In Capillary Extrusion
9:30-10:00	Laske, Stephan	Influence of Cellulose Fiber on the Foaming Behaviour of a Polypropylene Copolymer
10:00-10:30	Monroy, Roberto	A Method to Characterize Blowing Agent Concentration Effects during Polymer Processing
10:30-11:00	Grade, Noah	Novel Melt Filtration Technology for Challenging Recyclate

Monday Morning, April 28, 2014	Twin Screw I	Moderator: Dr. Jaime Gomez, Coperion K-Tron Pitman Inc.
8:30-9:00	Kluenker, Eike	Degassing of residual monomer during reactive extrusion of PA6: Experimental analysis
9:00-9:30	Huang, Keyuan	Ultrasonic Decrosslinking of Crosslinked High Density Polyethylene: Effect of Screw Design
9:30-10:00	Herken, Tobias	Experimental Analysis of the Material Degradation of PET on a Co-Rotating Twin-Screw Extruder
10:00-10:30	Puch, Florian	Experimental Investigation of the Mechanical Properties and the Morphology of PA6-MWCNT-Composites Depending on the Melt Compounding Conditions
10:30-11:00	Meysami, Mohammad	Continuous devulcanization of scrap EPDM rubber with supercritical CO2: Effect of process parameters on devulcanized rubber properties

EXTRUSION DIVISION ANTEC 2014 PROGRAM

Monday Afternoon, April 28, 2014

Monday Afternoon, April 28, 2014	Twin Screw II	Moderator: Jane Spikowski, PolyOne
1:30-2:00	Maia, Joao	Keynote: A New Extensional Static Mixer Concept for Improved Dispersive Mixing in Twin-Screw Extrusion
2:00-2:30	Fukuda, Graeme	Investigation of Scale-Up Methodologies in Twin-Screw Compounding
2:30-3:00	Lawton, David	Morphological Development of Latex Particles in a Solvent-Free Extrusion Process
3:00-3:30	Gogos, Costas	The Hot-Melt Extrusion Process for the Production of Oral Dosage Formulations and Medical Devices – A Review
3:30-4:00	Gogos, Costas	The Effect of Particle Size of APIs and Extruder Screw Configuration on the APIs' Dissolution Rate in a Co-rotating Twin Screw Extruder
4:00-4:30	Canedo, Eduardo	Engineering Modelling of Laboratory Internal Mixer
4:30-5:00	Tarverdi, Karnik	Using Twin Screw Extrusion Technology to Determine the Effects of Surfactant Concentrations by Melt Blending Organoclays with PET Nanocomposites
5:00-5:30	Mena, Jhorman	Energy Efficiency and Specific Energy Consumption (SEC) in Single Screw Extrusion (SSE) and Twin Screw Extrusion (TSE): Performance and comparison

Monday Afternoon, April 28, 2014	Dies	Dr. Karen Xiao, Celgard
1:30-2:00	Zatloukal, Martin	Keynote: Die Drool Phenomenon in Polymer Extrusion
2:00-2:30	Dooley, Joseph	The Effect of Multilayer Rheology on the Flow Distribution in a Coathanger Style Die
2:30-3:00	Lee, Patrick C.	Effects of Temperature and Viscoelasticity on Film Die Flow
3:00-3:30	Martin, Gerhard	Relaxation Zones in Extrusion Dies
3:30-4:00	Gupta, Mahesh	Comparison of Mesh Partitioning Technique and Level-Set Method for Coextrusion Simulation
4:00-4:30	Metwally, Hossam	Optimization of a Coat Hanger Die Geometry Considering Flow Induced Deformation
4:30-5:00	Gomez, Cathy	Virtual Prototyping Applied to Flat Extrusion Die
5:00-5:30	Sagar, Surendra	Optimizing the Co-extrusion Process for Multi-layer Line

EXTRUSION DIVISION ANTEC 2014 PROGRAM

Tuesday Morning, April 29, 2014

Tuesday Morning, April 29, 2014	Hot Melt Extrusion	Moderator: Dr. Michael Thompson, McMaster University
8:00-8:30	Gogos, Costas	Invited: Discussion of Optimizing the Elementary Steps of Processing in Pharmaceutical Hot Melt Extrusion Processes
8:30-9:00	Ali, Shaukat	Invited: Meeting API's Challenges with Pharmaceutical Polymer by HME
9:00-9:30	Martin, Charlie	Invited: Trends in Pharmaceutical Extrusion
9:30-10:00	Thompson, Michael	Invited: Influence of Formulation on the Twin Screw Granulation of Pharmaceuticals
10:00-10:30	Brown, Chad	Invited: Enabling Pharmaceutical Products Utilizing Extrusion Technologies
10:30-11:00	All Speakers	Panel Discussion — Hot Melt Extrusion

Tuesday Morning, April 29, 2014	Single Screw I	Moderator: Dr. Deep Samanta, The Goodyear Tire & Rubber Company
8:00-8:30	Campbell, Gregory	Keynote: A Mechanism for Solid Bed Breakup in Single-Screw Extruders - Solid Bed Shape Change
8:30-9:00	Derezinski, Stephen	The Sole Effect of Lateral Stress on Solids Conveying
9:00-9:30	Altinkaynak, Atakan	An Investigation of Melting and Metering Sections in a Single-Screw Extruder
9:30-10:00	Slusarz, Kevin	A Comparison of the Residence Time in Micro-Extruders and Its Effect on Molecular Weight for Low Output Applications
10:00-10:30	Karrenberg, Gregor	A High Speed Extruder with floating Screw Sleeve for Solid-Melt-Separation
10:30-11:00	Martin, Gerhard	Elongation and shear deformation of polymers in a wedge slit

EXTRUSION DIVISION ANTEC 2014 PROGRAM

Tuesday Afternoon, April 29, 2014

Tuesday Afternoon, April 29, 2014	Single Screw II	Moderator: Dr. Gregory Campbell, Castle Associates
1:30-2:00	Spalding, Mark	Keynote: Troubleshooting Gear Pump Assisted Single-Screw Extrusion Processes
2:00-2:30	Huang, Rongzhi	Layer-multiplying Co-extrusion of High Viscoelasticity Ratio Polymers
2:30-3:00	Zhao, Danyang	Experimental Studies on Extrusion Process of Polypropylene Double-Lumen Micro Tube in Medical Applications
3:00-3:30	Harris, Patrick	Multilayer Coextrusion of High Viscosity Elastomer Systems
3:30-4:00	Kneidinger, Christian	Characterizing the Melting Behaviour of Different Shaped Polymer Bulk Solids by Utilizing a Model Epxeriment with an Optical Measuring System
4:00-4:30	Huang, Wenyi	Pressure-Rise Tests for Detecting Particles in Polymers
4:30-5:00	Karrenberg, Gregor	Development of a custom Material Model for 3D-CFD-Simulation of Melting Processes in Polymer Processing
5:00-5:30	Mr. Baldev L. Boolani	SPE Businessman of the Year Award Winner
5:30		**SPE Extrusion Division Annual Business Meeting, Awards Ceremony and Reception**

Tuesday Afternoon, April 29, 2014	Session Dedicated to the Memory of Dr. Marino Xanthos	Dr. Costas Gogos, New Jersey Institute of Technology
1:30-2:00	Gogos, Costas	Invited: Pharmaceutical Hot Melt Extrusion Viewed as a Polymer Extrusion Compounding Process
2:00-2:30	Tzoganakis, Costas	Invited: UV-Initiated Reactive Extrusion for Polyolefin Modification
2:30-3:00	Golba, Joe & Tucker, Chris	Invited: Reactive Extrusion Based In-Situ Polymerization of Nanoclay Filled Polyamide 6
3:00-3:30	Wetzel, Mark	Invited: Experimental Techniques to Characterize Reactive Extrusion Processes
3:30-4:00	Kamal. M, Carreau, P., Ton-That, M-T & Dini. M.	Invited: Re-processing of Poly(ethylene terephthalate) to Produce PET/Clay Nanocomposites
4:00-4:30	Bigio, David & Yang, Chi-Tai	Invited: Modeling of Trace Devolitalization in an Extruder
4:30-5:00	Hyun, Kun Sup	Invited: Foam Extrusion With Physical Blowing Agents — Engineering Approach

EXTRUSION DIVISION ANTEC 2014 PROGRAM

Wednesday Morning, April 30, 2014

Wed Morning April 30, 2014	Tutorial Session: Single Screw Extrusion	Moderator: Tony Neubauer, Materials Processing Consultants LLC
8:00-8:30	Womer, Tim	Sheet Extrusion
8:30-9:00	Noriega, Maria	Material Failure Analysis and Extrusion
9:00-9:30	Xiao, Karen	Screw Simulations
9:30-10:00	Perdikoulias, John	Die Simulations
10:00-10:30	All Speakers	Question and Answer Session

Wed Morning April 30, 2014	Tutorial Session: Twin Screw Extrusion	Moderator: Adam Dreiblatt, CPM Century Extrusion
8:00-8:30	Wetzel, Mark	Simulation as a Tool for Troubleshooting and Improving Twin-Screw Extrusion Processes
8:30-9:00	Andersen, Paul	How to Handle/Process Difficult Materials
9:00-9:30	Dreiblatt, Adam	Optimization of Screw Configurations for Twin-Screw Compound-ing Extruders
9:30-10:00	Martin, Charlie	Twin screw tips, techniques & practices
10:00-10:30	All Speakers	Question and Answer Session

EXTRUSION DIVISION ANTEC 2014 PROGRAM

Wednesday Afternoon, April 30, 2014

Wed Afternoon April 30, 2014	Joint Session: Flexible Packaging/Extrusion	Moderator: Tom Dunn, Flexpacknology & Steve Schick, Teel Plastics Inc.
1:30-2:00	Miller, Mark	Rheology and Slot Die Coating Technology
2:00-2:30	Lin, Yijan	Structure-Property Relationship of Biaxially Oriented Polyethylene (BOPE) Films Made via Double Bubble Film Fabrication Process
2:30-3:00	Hu, Yushan	Polypropylene Based Olefin Block Copolymers as Tie Layers for Multilayer Packaging
3:00-3:30	Borse, Nitin	Developing Cost Effective Co-extruded Film Structures For Flexible Packaging
3:30-4:00	Zatloukal, Martin	Evaluation of Different Heat Transfer Models for 9-layer Film Blowing Process by Using Variational Principles
4:00-4:30	Janas, Marius	A Prediction Model for the Numerical Optimization of a Novel Blown Film Cooling System
4:30-5:00	Dominey, Simon	Equipment and Material Considerations for Microcellular Foaming
5:00-5:30	Dan Falla & Michael Li	Reliable Hot Tack Testing of Polyolefin Films

EXTRUSION HINT

Since we are getting into "dryer season", make sure your dryers are running at their peak performance:

⇒Check the desiccant material – verify the proper is amount is on-line and not contaminated

⇒Verify all heaters are working properly

⇒Clean ALL filters – process, regeneration and after-coolers

⇒Verify they are no leaks in the system – hoses, gaskets, etc.

A dryer must be operating at its best to provide you with the properly dried material for your process!

— Keith A. Larson, ACS Group

SPE Continuous Compounding TOPCON (CCT 2014)

David Bigio

The Society of Plastics Engineers Extrusion Division and the Cleveland/Akron Sections hosted a Continuous Compounding Topical Conference (CCT 2014) on March 11-13, 2014 at Case Western University in Cleveland, OH. This program feature 27 presentations by academia and industry experts and included a review of fundamental mixing technologies on Day 1, continuous compounding core technologies and case studies on Day 2, and advanced/cutting edge technologies on Day 3. The sessions were:

Afternoon session: Fundamentals
Morning session: The Melt Compounder's Extruder Tool Kit
Afternoon session: Case Studies in Melt Compounding and Reactive Extrusion
Morning session: Process Monitoring and Control
Afternoon session: Process Simulation for Design, Scale-Up, and Troubleshooting

All of the speakers (100%) were invited due to their expertise in the field. Our speaker demographics included:

- 3 universities (Case Western Reserve University, Cleveland State University, University of Maryland)
- 5 countries (USA, Canada, UK, France, India)
- State of Ohio
- 17 companies (13 equipment/instrumentation, 2 materials, 1 simulation, 1 consultant)

There were 24 corporate sponsors enhancing the experience of the entire program. A complete list of sponsors is on the SPE website: http://www.4spe.org/sites/default/files/cct14-sponsors.pdf

The program attracted 168 attendees which included the 27 speakers, 24 sponsors and 15 local college students. Attendees traveled from around the USA, Europe and India. Of the attendees, there were registrations for 43 new members to the SPE.

It is the intent of the Extrusion Division in sponsoring Topical Conferences (TOPCON) to expand the interest and knowledge of the Extrusion technology, the interest all practitioners in the field to Extrusion and its many applications, and to build interest in the SPE. In addition to the presentation, Jaime Gomez, from the Extrusion Division and National, led a discussion on the future branding of SPE and how to interest the next generation into participation and membership.

We wish to thank all of the volunteers both from the Akron and Cleveland sections, from the Division and the support of SPE Headquarters, especially Sue Wojnicki and (who was there). It is with their work this all is possible.

RESIN DRYING FOR THE PLASTICS INDUSTRY

By

Tom Baker, ACS Group

Any moisture present during the melt and process stages of plastic resins will dissipate as steam and have a negative impact on the final product being produced, since it can get trapped in or on the surface of the final product. This will cause voids, splay, structural weakness or failure, or poor adhesion of finishes such as plating. Therefore, one must take into consideration the drying process required to remove the moisture before processing.

Plastic resin drying requirements fall under two categories - Non-Hygroscopic and Hygroscopic.

-***Non-Hygroscopic*** resins such as PP, PE and PVC do not absorb any moisture internally, but are subject to surface moisture as a result of exposure to humid ambient air.

-***Hygroscopic*** resins are generally referred to as "engineering resins" such as PET, PC, Nylon, ABS, and many others that absorb moisture internally from exposure to humid ambient air. Non-hygroscopic materials can also become hygroscopic if they are filled, i.e. talc filled polypropylene.

Non-Hygroscopic Resins

Non-Hygroscopic resins having surface moisture only are the simplest to deal with. Often in processes using these resins one does not need to be sensitive to the amount of surface moisture present and no drying is required. In Non-Hygroscopic applications where the presence of the surface moisture may affect the product quality the drying process to remove the surface moisture is actually a simple process.

Hot Air Drying

Warm air has the ability to hold more moisture than cooler air. Therefore, the drying process to remove the surface moisture in Non-Hygroscopic resin is simply heating air and blowing it across the resin you are drying – this is known as Hot Air Drying.

A Hot Air Dryer System is a simple piece of equipment consisting of a heat source, a blower to carry the heated air to the resin, and a drying hopper with a mass flow design.

Hot Air Drying is a single pass design, pulling atmospheric air through the blower intake, heating it on the discharge side of the blower, introducing it into the bottom of the drying hopper sized for one to two hours of residence time, and discharging out the top of the drying hopper back into the atmosphere. The temperature is set lower than the resin's tack point, typically around about 120 to 140 degrees F.

RESIN DRYING FOR THE PLASTICS INDUSTRY

(continued)

When plastic resin is stored in an outdoor silo in a cold temperature climate, and a warming sun comes up, the air in the silo above the resin level will condense its moisture and often cause excessive moisture levels that will affect the process. The lower the level of resin in the silo the more air you have above the resin. The condensation during this action can be extreme to the point of water actually running out of the bottom of the silo.

In this condition, it is not uncommon for a processor to utilize Hot Air Drying not only to dry the surface moisture, but to actually condition the resin to a common consistent temperature to minimize variations in their process.

Hygroscopic Resins

Engineering type plastic resins like PET, Nylon, PC and ABS, and others, are hygroscopic and will absorb moisture internally when exposed to humid ambient air. Each type of plastic resin can hold different levels of moisture within the molecular chain of its makeup. Not only is the potential amount of moisture content of each resin different, the acceptable amount of moisture level for processing varies, and the final product will dictate maximum allowable amounts of moisture present during the process.

Drying hygroscopic resins requires both heat and dry air to remove the moisture. Therefore, the equipment selection becomes more critical than hot air drying. There are multiple designs including Compressed Air Dryers, Vacuum Dryers, and the more common Desiccant Dryer, along with various types of media to provide the dry air.

Drying hygroscopic plastic resins require low dew point air. Simplifying the term dew point, it is the temperature water vapor starts to condense as a liquid on the surface. The ideal low dew point level of process air varies for different resins, but the industry standard is -40 degrees F dew point. The drier the air, the more ability it has of picking up and carrying moisture away.

Regardless of the equipment or media chosen, they all require heating the moisture internal to the pellet to increase the vapor pressure of the moisture so it will release to the surface of the pellet. If you don't achieve this, the pellet will not let go of the internal moisture for removal.

Regardless of the drying process chosen there are four things you must have to get the necessary results. Do not under estimate any of them or you may come up short on your end goal. They are:

1) **TIME** … which is a calculation of the required throughput needed x residence time, dictates the minimum size of a mass flow design drying hopper required.

2) **TEMPERATURE** … is the heat needed to raise the internal temperature of the pellet to the point of vaporizing the moisture within the pellet forcing it to the surface.

3) **AIRFLOW (CFM)** … is the airflow required to carry the heat into the bottom of the drying hopper, and carry the moisture away.

4) **DEWPOINT** … is the dryness of the airflow so it is able to pick up the moisture.

RESIN DRYING FOR THE PLASTICS INDUSTRY

(continued)

Each type of material has its own level of moisture saturation, and a maximum acceptable level of moisture content for processing. The resin, virgin pellet or a regrind flake will affect how it will dry because of the particle shape and density is different.

Most dryers for these resins are sized assuming the moisture content level will be at maximum saturation, and capable of removing a given percentage of that moisture to an acceptable level or below for processing. Although the resin may be saturated with moisture it often is not at its maximum saturation point, and therefore you do not have to remove as much moisture.

In order for a supplier to warrant performance of a piece of equipment for an application one has to assume the maximum moisture content level is present. All manufactures do not design or spec their equipment's capability the same. Anyone offering to dry your resin in less time, or less airflow, is simply not sizing the equipment based on the resins maximum saturation point. There is no magic available.

Compressed Air Dryers

There are Compressed Air Dryers for small throughput applications. These dryers require a Drying Hopper of a mass flow design, sized for the residence time of the specific resin you want to dry. Typical drying times are 2 – 4 hours, and some to 6 hours residence time. The Compressed Air Dryer design includes a heating source to elevate the internal temperature of the pellet to the desired temperature to break the molecular chain so it will release the moisture forcing it to the surface. Compressed air is used to move the heat into the material and away from the material.

Compressed Air Dryers are an open loop process - the compressed air enters the bottom of the drying hopper, expands removing the moisture and exhausts to the atmosphere.

Normal plant compressed air available is capable of 0 degree F dew point as long as there is a dryer on the air compressor. To get the compressed air to -40 degree F dew point, the compressed air dryer needs to have a membrane filter to trap the moisture.

Although a simple design piece of equipment to manufacture and maintain, compressed air is a costly utility to produce. Because of the volume of compressed air required, you will not see these available for larger applications. Sometimes it is in question as to the potential of contamination in the compressed air such as an oil residue ending up in the resin.

Vacuum Dryers

Vacuum Dryers do not use a conventional drying hopper to hold the material during the process. These dryers normally have multiple containers with small amounts of resin. Each container is heated to the elevated temperature to allow the pellets to let go of the moisture. A vacuum is then drawn on the vessel pulling the moisture away.

These dryers are normally restricted in throughput, have many moving parts, require high maintenance because of the many moving parts, and generally are not cost effective. Molding processes require a continuous supply of plastic resin, but since the vacuum dryers dry a small charge at a time in batch form and they need to transfer each charge in preparation of the next batch, vacuum dryers are better suited for batch type drying.

RESIN DRYING FOR THE PLASTICS INDUSTRY

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Desiccant Dryers

Desiccant Dryers, most commonly known as Dehumidifying Dryers are the most widely accepted method for drying Hygroscopic resin in the plastics industry. They provide a very controlled environment of dry dehumidified air, the most cost effective, and proven designs, regardless of the resin or throughput required.

There are various types of desiccant media available. The most common desiccant used in this industry is Molecular Sieve Desiccant because of its strong affinity to absorb moisture. This desiccant is available with specific particle and pore size throughout its make-up, and using the correct size bead and pore make-up does make a difference in your results. It does not change its make-up by absorbing water based moisture, heating and cooling.

Molecular sieve desiccant has an infinite life except for two reasons:

1. Break down by heating, cooling, and vibration
2. Contamination from dust, fines, or volatiles from vapors that form when additives burn off the resin during the drying process.

There are several types of dehumidifying dryer designs using molecular sieve desiccant, but there are mechanical design differences being large desiccant tanks with one tank on process and the other in regeneration and cool down; multiple bed designs, and continuous rotating bed designs. Each has their good and bad points. They all must provide the sufficient amount of dry air, with enough heat and airflow, to the resin for the correct amount of time.

Where you have to be careful is all manufactures will say their dryer is sized to do the job. If you don't know the real moisture content level you are starting with and how much moisture you need to remove, how does the manufacture, or you, know it will do the job unless it is sized to remove the moisture based on the resins maximum saturation point? Waiting until you make a bad product due to moisture is too late to find out.

If you are expecting any dryer that has a minimal volume of desiccant, low heater capacity, low on airflow, or too small of a drying hopper, for the application to thoroughly dry your resin, don't expect it to remove the maximum amount of moisture. You need to achieve those four item requirements…Time, Temperature, CFM, and dew point to assure a dry resin for successful processing.

CFM varies between manufactures, and specific dryer applications. Generically, you need approximately 0.8 to 1 CFM per pound of resin being processed. This rating is based on the drying hopper being full of material. There are dryers out there that are sized as low as approximately 0.4 CFM per pound, so keep in mind that CFM is one of the four items you need to have dry material. If you are short on CFM you lose the ability to carry enough heat into the material and moisture away in the residence time. Remember, you want your dryer to supply dry material to your process ALL of the time, not 90% of the time!

RESIN DRYING FOR THE PLASTICS INDUSTRY

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Too much airflow relative to the dryer heater capacity and the size of drying hopper can also work against you. You can fluidize the material in the drying hopper, and therefore not get uniform heating of the pellets, resulting in uneven drying.

The amount of desiccant available is critical because it can only absorb a given amount of moisture in a fixed amount of time. It has to be regenerated at an elevated temperature to drive the moisture out of the desiccant, and it must be cooled back down before it can go back into the process stream or it will not be able to absorb moisture. Without this, you cannot achieve the required dew point.

All of the heater elements need to be working at the design capacity. The process heaters supply the heat source to the resin to be dried. The regeneration heaters provide the necessary heat to regenerate the desiccant media.

Because these dryers are closed loop on the process air, one cannot introduce ambient air into the process side of the airstream. You also have to be aware that the return air temperature coming from the top of the drying hopper back to the dryer must be less than 130 degrees F, otherwise you would put too much heat into the desiccant and it would not pick up the moisture. All high temperature applications, and *all* continuous rotating bed design dyers *require* an after-cooler on the return side of the process air stream.

If you are drying a low temperature resin (normally below 170 degrees) you need to have a *pre*-cooler on process delivery side. This also needs a supply of water and is used to cool the process air down before entering the drying hopper. In this case, the process heater is installed after the cooler and before the drying hopper inlet to ensure proper temperature control.

In summary, there are a lot of things to consider, but it still boils down to TIME, TEMPERATURE, AIRFLOW (CFM or m^3 per hour) and DEWPOINT. If you meet these four items you will have dry material for processing.

TROUBLESHOOTING AND MITIGATING GELS IN POLYOLEFIN FILM PRODUCTS

Mark A. Spalding, The Dow Chemical Company, Midland, MI
Eddy Garcia-Meitin and Stephen L. Kodjie, The Dow Chemical Company, Freeport, TX
Gregory A. Campbell, Clarkson University/Castle Associates, Jonesport, ME

Abstract

The term "gel" is commonly used to refer to any small defect that distorts a film product. Eliminating gel defects from extruded polyolefin film products can be difficult, time consuming, and expensive due to the complexity of the problem and the high levels of off-specification product produced. This paper discusses the identification of gel types, the common root causes for gels, and the technical solutions for mitigating gels in film products produced using single-screw extruders.

Introduction

Troubleshooting extrusion processes where gels are appearing in polyethylene (PE) film products can be difficult due to the number of different gel types that are possible. For these processes, the troubleshooter must be able to diagnose the problem quickly and provide an economically viable technical solution [1]. Because gels can originate from numerous sources, the troubleshooter must be able to identify the characteristics of the gel and recognize the likely possibilities of the source. Process changes must then be performed to mitigate the gel defects.

There are many types of gels [2] and the most common include: 1) highly oxidized polymeric material that appears as brittle black specks, 2) polymeric materials that are crosslinked via an oxidative process, 3) highly-entangled polymeric material (such as high molecular weight species) that are undispersed but not crosslinked, 4) unmelted resin, 5) filler agglomerates from masterbatches, and 6) a different type of resin or contaminant such as metal, wood, cloth fibers, or dirt. A crosslinked resin gel is typically formed during an oxidation process, resulting in the crosslinking of the resin chains and the generation of discolored gels. Highly-entangled gels are typically high molecular weight polymer chains that are entangled and thus difficult to disperse during the extrusion process. When analyzed using a hot stage microscope, this gel type will melt as the stage temperature is increased. When the stage temperature is then decreased, the gel will crystallize, creating the appearance of a gel as a solid polymer fragment. Since these gels are not oxidized they are not associated with color. They are commonly referred to as undispersed or unmixed gels. Unmelted resin exiting with the discharge can sometimes occur, especially at high extrusion rates. These gels will melt during heating with a hot-stage microscope, and typically they will not reform during the cooling phase. Numerous sophisticated methods are available for analyzing gels including epi-fluorescence microscopy, polarized light microscopy, and electron microscopy with x-ray analysis. These methods are discussed in the next sections.

Gels can be generated from many different sources and include: 1) the resin manufacturer, 2) the converting process, 3) pellet blending of resins with significantly different shear viscosities, 4) pellet blending of different resin types, and 5) direct contamination. Modern resin manufacturing processes exclude oxygen from the system and are very streamline such that process areas with long residence times do not exist. As such, crosslinked and oxidative gels are likely not generated by the manufacturer. Improperly designed extrusion equipment and processes, however, are common, leading to the oxidative degradation of resins and crosslinked gels. Several case studies in the next sections show how poorly designed processing equipment can lead to crosslinked and unmixed gel contamination of film products.

The goal of this paper is to describe the different type of gels that are likely to occur in polyolefin film products, techniques for identifying the gel type, and technical solutions to mitigate them from single-screw extrusion processes.

TROUBLESHOOTING AND MITIGATING GELS IN POLYOLEFIN FILM PRODUCTS

(continued)

Protocols for Gel Analysis

Established protocols for gel analysis in polymer films are well documented in the literature [2-4]. For example, gels can be identified using the schematic process [4] shown in Figure 1. Typically a film with defects is visually inspected using a low power dissecting microscope. The gels can be classified based on size, color, and shape, and isolated using a razor blade or scissors. Cross sections of the gels ranging from 5 µm to 10 µm thick are collected at temperatures below the glass transition temperature (T_g) of the film using a cryogenic microtome; i.e., -80°C to -120°C. For optical examination, a thin section containing the gels is placed on a glass microscope slide with a drop of silicon oil and covered with a glass cover slip. Additional sections are collected for examination via hot stage microscopy and for compositional identification if needed.

After collecting the sections, the polished block-face containing the remainder of the gel is retained. In many instances, gels arise from inorganic contaminants such as the metallurgy from pellet handling equipment, extruders, or components from masterbatches. Examination of these inorganic components are best performed with the block-face sample using a scanning electron microscope (SEM) equipped with an energy dispersive x-ray detector (EDX) [5,6]. In some cases, additives or inorganic residues are present in low concentrations within the gels. A method to enrich the concentration of these materials is to expose the block-face containing the gel to oxygen plasma. Etching will preferentially remove the polymer at a much faster rate than the inorganic materials, enriching the inorganic components for elemental analyses. It must be noted that prior to SEM and EDX analyses, a thin conductive coating like carbon is typically evaporated onto the sample to render it conductive under the electron beam.

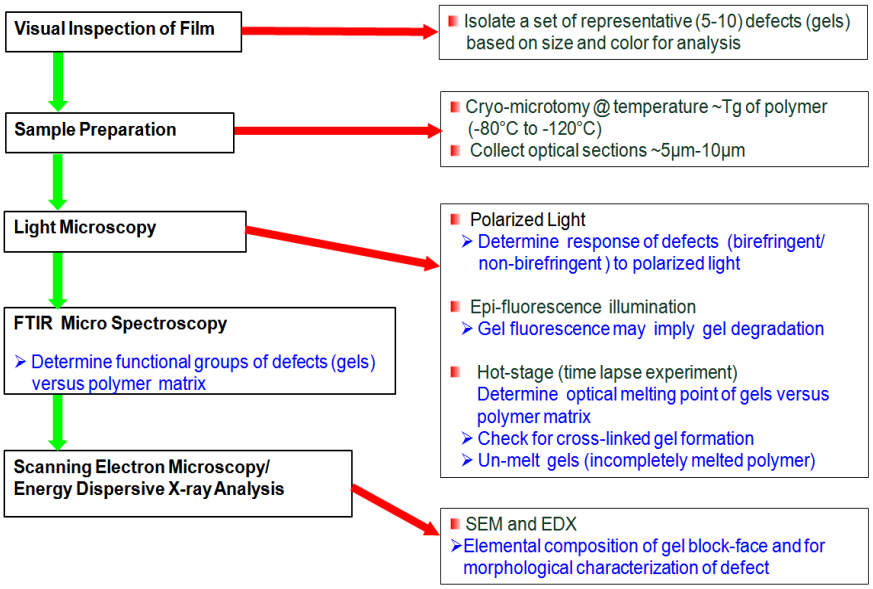

Figure 1. Methodology for characterizing defects in polymer films [4].

The next sections will demonstrate various methods of analysis used for common gel types.

(continued)

Oxidized Gels

The most common type of gel is caused by oxidative processes that crosslink the PE chains. The best way to identify this gel type is by observing them with polarized light and ultraviolet (UV) light sources. Transmitted polarized light microscopy represents an effective technique [7] that can be used to investigate structures in crystalline films. For example, black speck gels were contaminating a multilayer film product. The gels were relatively brittle when cut for analysis. The source was unknown. The detail of a gel is clearly visible using transmitted polarized light, as shown in Figure 2a. Close examination of this gel using epi-fluorescence with an ultraviolet light source caused an intense fluorescence emission, as shown in Figure 2b. This type of emission suggests thermal oxidation and crosslinking of the polymer. Micro-infrared analysis of the gel indicated that it contained oxidized PE and maleic anhydride, as shown by the spectrum in Figure 3 (for clarity, this figure can be found at the end of this paper). This material likely formed on the metal surfaces of the extruder and then flaked off during a minor process instability. The material then flowed downstream and contaminated the film as a gel.

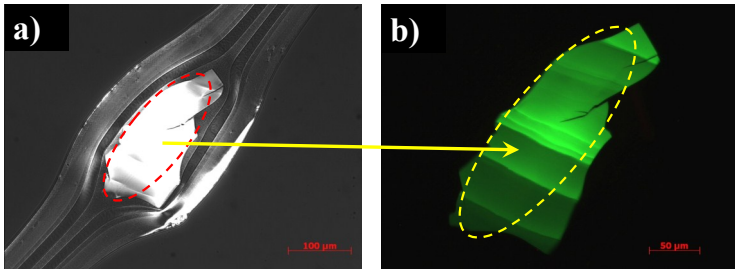

Figure 2. Transmitted polarized light images of a thermally oxidized and crosslinked gel in a multilayer film: a) photograph in polarized light, and b) the gel fluorescing under UV light.

Crosslinked Gels

Crosslinked gels are oxidized gels, but the level of oxidation may not be enough to cause them to fluoresce under UV light. These gels may have a level of crystallinity and thus be birefringent under polarized light. For example, the slightly birefringent gel shown in Figure 4a was studied using a temperature programmable hot stage, polarizing light microscope [7]. The optical melting temperature (T_m) of the gel was measured at 128°C and consistent with the PE used to make the product, as shown in Figure 4b. To determine if the gel was unmixed (highly entangled but not crosslinked), the gel was held above the melting temperature (135°C) and then stressed. A dental tool was used to stress the top of the glass cover slip. Crosslinked gels will appear birefringent, (Figure 4c) in response to the anisotropy of stress distribution in the gel to polarized light. The gel dimensions and shape remained after cooling verifying crosslinking, as shown in Figure 4d. If the gel was highly entangled and not crosslinked, the gel would have disappeared after the stress and cooling were applied.

Gels from Foreign Contamination

The origin of defects causing discoloration in polyolefin pellets can be identified using light and electron microscopy. For example, PE pellets from an in-plant recycle re-pelletizing process contained pellets that were off color and had black specks, as shown in Figure 5a. One of these defects was isolated using the cross sectioning technique, as shown in Figure 5b. The cross section revealed an intense reddish particle that caused the discoloration of the pellet.

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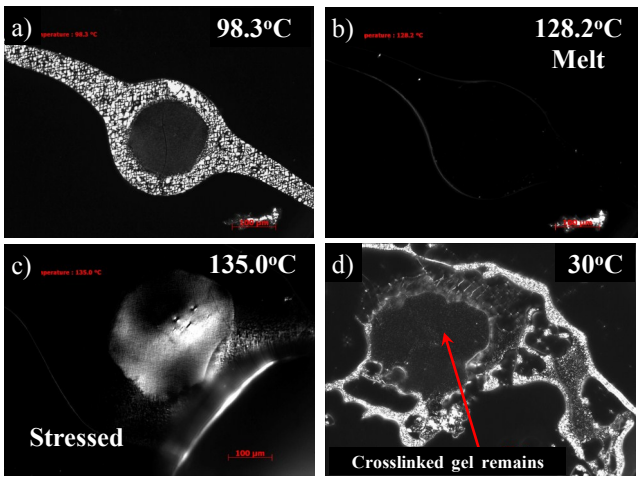

Figure 4. Hot stage microscopy of a crosslinked gel in a crystalline monolayer film: a) below the melting temperature, b) optical melting point at 128°C, c) appearance of birefringence after stressing at 135°C, and d) intact crosslinked gel after cooling to 30°C.

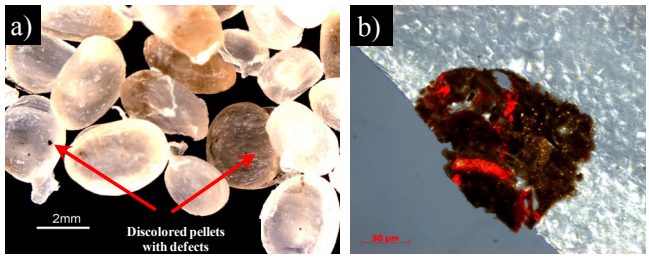

Figure 5. Photographs of foreign contamination in pellets of a re-pelletized reclaim stream: a) photomicrograph of discolored polyolefin pellets containing dark defects, and b) transmitted polarized light micrograph of a pellet cross section containing a defect.

SEM and EDX microanalysis were used to determine that the defects contained primarily iron and oxygen, and it likely was iron oxide. Figure 6 shows a backscatter electron image (BEI) of the pellet block-face sample showing the defect causing the discoloration and the elemental spectrum. Metallic based defects can originate from process equipment, railcars used for shipment, pellet transfer lines, and poor housekeeping. The origin of the iron oxide was likely from a storage bin.

In another example, a multilayer film product was experiencing occasional gels. The gels were isolated and the cross sections were collected as shown in Figure 7a. These gels contained highly birefringent particles that resided in the core layer. The outer film layers appeared amorphous and the core layer was slightly birefringent. The optical melting temperature of the core layer was determined to be 123°C while the birefringent gels melted at 265°C. The melting temperature of 123°C was consistent with the PE resin used to produce the core layer. The higher melting temperature material and micro-infrared analyses of the defects indicate that they were foreign contaminants, and they were identified as a polyester resin. The polyester resin was used in another process in the converting plant, and it inadvertently contaminated the PE feedstock.

TROUBLESHOOTING AND MITIGATING GELS IN POLYOLEFIN FILM PRODUCTS

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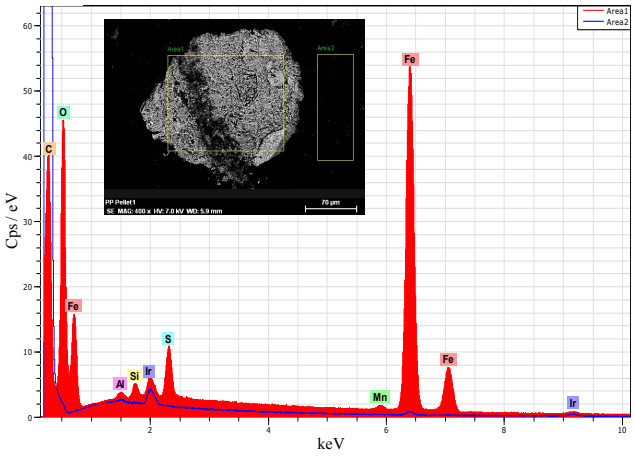

Figure 6. EDX microanalysis of an inclusion in a polyolefin pellet cross section (Figure 5b). The analysis indicated that the particle was likely iron oxide.

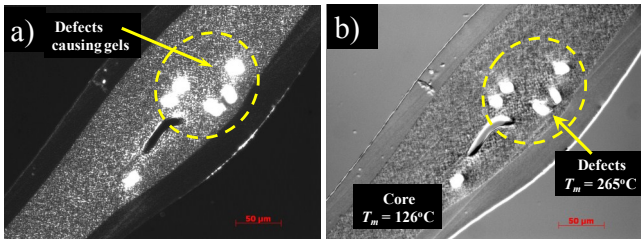

Figure 7. Photographs of gels in the core layer of a three layer film: a) transmitted polarized light, and b) hot stage microscopy was used to determine the melting temperatures of the core resin and defects.

Another common contaminant that produces gels is fibers, as shown in Figure 8. In many cases, these contaminants are cotton fibers from clothing and gloves or cellulosic fibers from packaging materials. Fourier transform infrared (FTIR) spectroscopy is one of the best techniques for determining the chemical functionality of organic based defects in PE films. The infrared absorbance characteristics of the defect were determined using FTIR spectroscopy, as shown in Figure 9 (for clarity, this figure can be found at the end of this paper). The broad absorption bands near 3600 cm^{-1} to 3100 cm^{-1} are due to hydroxyl (-OH) stretching vibrations, the C-H vibration stretch is near 2916 cm^{-1} to 2851 cm^{-1}, and the ester carbonyl group absorption is near 1734 cm^{-1}. Based on the infrared absorption characteristics, the defect in the PE film is a cellulosic fiber with degraded PE resin.

Once the contaminant is identified, the troubleshooter must determine how the material entered the feedstock stream. Process controls must be identified and implemented to mitigate the contaminant source.

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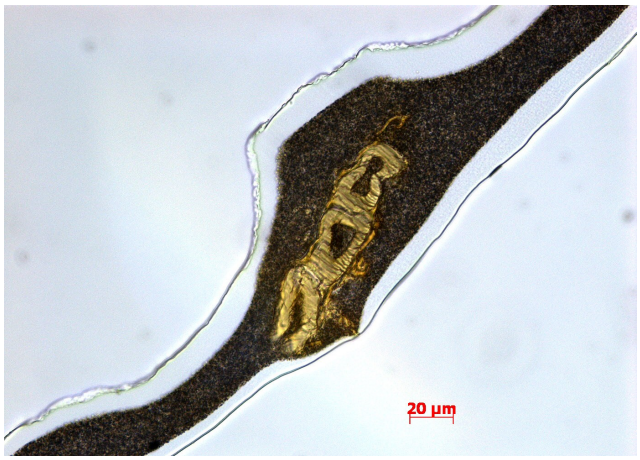

Figure 8. Transmitted bright-field image of PE film containing a fibrous gel.

Case Studies

Oxidized gels, carbon specks, and unmixed gels can be created inside the extrusion processing line. Crosslinked gels and black specks occur due to regions in the process that are stagnant and have very long residence times in the extruder. Unmixed gels and solid polymer fragments occur because the resin was not subjected to a high stress level during processing. This section provides several case studies where these types of gels occurred. The technical solutions to mitigate the gels are then presented.

Gel Showers in a Cast Film Process

Crosslinked gels can form in stagnant regions of screw channels, transfer lines, and dies. The time required for these gels to form range from about 30 minutes for linear low density polyethylene (LLDPE) resin up to 12 days for low density polyethylene (LDPE) resin. Stagnant regions can occur at entries and exits of mixers [1] and barrier sections, and they can occur when the metering channel of smooth-bore extruders is not controlling the rate. In these cases, a section upstream of the metering section is rate limiting, causing portions of the metering section to operate partially filled [8,9]. When these channels operate partially filled the main flow is on the pushing side of the channel while the trailing side operates void at first. After a period of time, clean resin gets into the void regions and rotates with the screw for long durations. Eventually the resin will degrade, forming crosslinked gels. Slight process upsets can dislodge this material, allowing the material to flow downstream creating a gel shower in the film.

A film plant was extruding a LDPE resin into a specialty product using a cast film process [8,9]. Due to high demand, a new 88.9 mm diameter, 33 L/D extruder was installed in the plant. Soon after startup the product was acceptable and high quality. After 12 days, the line began to experience intermittent discharges of crosslinked material (gel showers) and carbon specks. Photographs of these gels are shown in Figure 10. In some cases, the gel showers were observed 2 to 3 times per day and would last from 1 to 5 minutes. The gels were clearly crosslinked and were brown in color. The extrudate temperature was higher than expected for the process. The intermittent gels resulted in production downtime due to purging and in numerous customer complaints. A high and costly level of quality control was required to remove the gel contaminated product from the prime product. Due to the high amount of downtime and the high levels of quality control needed, the operation of the new line was considerably more expensive than planned.

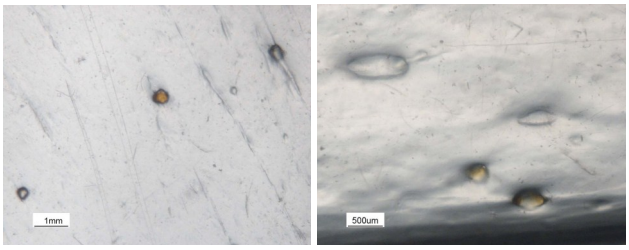

Figure 10. Photographs of crosslinked gels in a LDPE film.

It was hypothesized that the extruder was operating partially full due to the low specific rate during operation. To determine if partially filled channels were the root cause of the reduced rate, high discharge temperature, and degraded material, screw rotation was stopped and the screw was removed while hot from the extruder. Examination of the polymer on the screw indicated that in the meter section about half of the channel width on the trailing sides of the flights for all but the last diameter were filled with a dark colored, partially carbonized LDPE resin, indicating that these regions were stagnant. The reduced flow rate caused these regions to be partially filled, creating void regions on the trailing side of the channel. Some of the resin adhered to the trailing side of the screw in the void regions and stayed there for extended time periods, as shown in Figure 11. The resin adhering in the void regions eventually degraded into the dark-colored, crosslinked material. Small process variations dislodged some of this material and caused the intermittent gel showers that contaminated the product. Moreover, compacted solids were found wedged in the channel at the entrance to the barrier section. The wedged material was caused by the relatively large width of the entering solid bed being forced into the continually decreasing width of the solids channel of the barrier section.

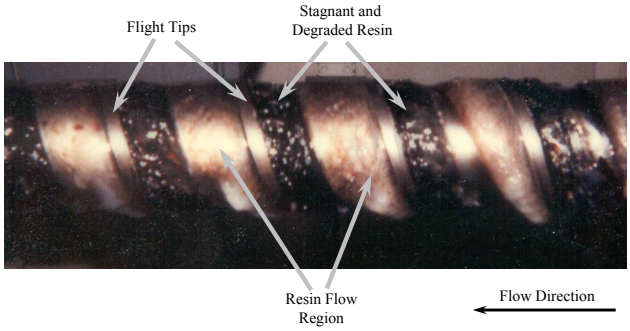

Figure 11. Photograph of a removed screw showing the resin flow and degraded resin due to stagnant regions [9].

The technical solution to eliminate this problem was a simple modification to the entry of the barrier melting section. For this modification [8], some of the metal in the melt conveying channel was removed along with a portion of the barrier flight, allowing some solid material to enter the melt channel and reducing the restriction at the entry. By reducing the restriction, the rate limiting step of the process changed from the entry region of the barrier section to the metering section. After the modification was made, the gels were eliminated from the process.

Unmixed Gels

As stated previously, unmixed gels are highly entangled species that are molten when they are discharged from the die, but solidify first upon cooling to produce a gel that appears as a solid polymer fragment. These types of gels are easily removed from an extrusion process by subjecting all molten resin to a one-time high level of stress near the discharge of the extrusion screw. This stress is easily applied using a Maddock-style mixer with a relatively tight clearance on the mixing flight.

(continued)

A film process was producing a monolayer film that had a low level of gels. The gels were tested using hot stage microscopy and identified as highly entangled species (unmixed gels). These gels melted and then disappeared when heated and stressed via pressure smearing using a dental tool, as shown in Figure 12.

The unmixed gels were removed by increasing the stress level in the Maddock mixer. The stress level was increased by decreasing the clearance on the mixing flight. The stress level required to disperse unmixed gels depends on the resin and the level of chain entanglement. In past experiences, the stress level required to disperse PE unmixed gels is about 100 to 200 kPa.

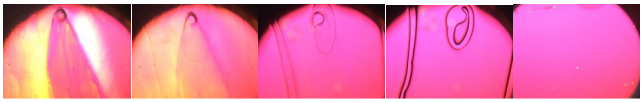

Figure 12. Photographs of an un-mixed gel at select temperatures using a hot-stage microscope. The un-mixed gel melted at about 135ºC. When the gel was smeared by moving the glass cover slip, the stress was enough to disentangle the polymer chains such that the gel would not reappear upon cooling.

A similar problem with solid polymer fragments occurred for a thermoplastic polyurethane (TPU) resin [10]. For this case, a combination of a lower compression ratio, a longer barrier section with a very small barrier flight clearance, a Maddock mixer with a small mixing flight clearance, and deeper metering channels allowed the TPU resins to extrude at twice the rate and provide high quality extrudates that were free of solid polymer fragments.

The shear stress that the material experiences for flow across the mixing flight of the Maddock mixer can be estimated using Equations 1 and 2. The shear stress level is responsible for breaking up the entangled species. This calculation is based on screw rotation physics [1].

$$\dot{\gamma}_M = \frac{\pi(D_b - 2u - 2\lambda)N}{(u + \lambda)} \qquad (1)$$

$$\tau_M = \eta\dot{\gamma}_M \qquad (2)$$

where $\dot{\gamma}_M$ is the average shear rate for flow over the mixing flight in 1/s, N is the screw rotation rate in revolutions/s, η is the shear viscosity at the temperature of the mixing process and at shear rate $\dot{\gamma}_M$, D_b is the barrel diameter, u is the undercut distance on the mixing flight, λ is the main flight clearance, and τ_M is the shear stress that the material will experience for flow over the mixing flight.

Carbon Specks in a Film Product

Carbon specks can be generated in the extruder channels and in downstream transfer lines and dies if stagnant regions are present. In general, these regions are not very large like those in Figure 11. Instead, they are thin coverings that occur at the flight radii or at entry and exits of mixing devices [1]. In general, the region will first create small crosslinked type materials that adhere to metal surfaces. With additional residence time, the crosslinked material will form a thin carbon layer of highly oxidized material. When the layer breaks away from the metal, it is discharged as black specks in the PE film. These specks will fluoresce under UV light.

TROUBLESHOOTING AND MITIGATING GELS IN POLYOLEFIN FILM PRODUCTS

(continued)

A LLDPE blown film line was experiencing black specks in the product. In order to locate the source, a Maddock solidification experiment [11] was performed where a small amount of a red color concentrate was added to the feedstock resin, after the red color appeared in the extrudate screw rotation was stopped, and the resin was solidified in the channels. A photograph of the experimental sample [12] is shown in Figure 13. Here a thin layer of carbonaceous material was formed at the pushing flight due to the formation of Moffat eddies [13]. Moffat eddies are recirculation or vortices that occur at sharp corners as shown in Figure 14. When fluid is put in motion with top driven cavity flow the main circulation is shown in Figure 14. A secondary circulation is set up in the stationary corners of the channel, creating a low velocity helical eddy that is outside the high velocity flows of the main part of the channel.

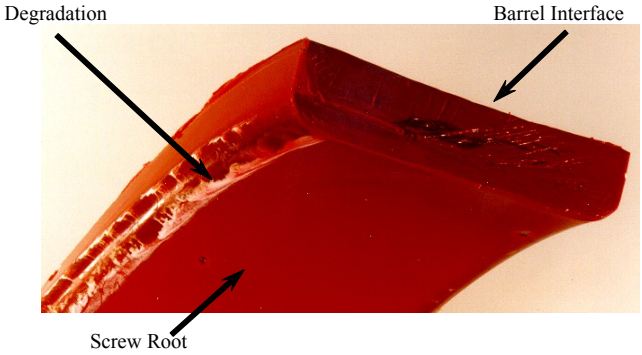

Figure 13. Photograph of degradation at the pushing flight for a screw running LLDPE resin [12].

The Moffat eddies that created the degraded resin occurred because the flight radii were too small for the depth of the channel. If the flight radii would have been larger, the Moffat eddies would not have occurred and thus carbon deposits would not have formed.

The Society of the Plastics Industry, Inc. (SPI) guidelines state [14] "unless otherwise specified the root radius will not be less than 1/2 of the flight depth up to 25 mm radius." Many screws are often designed, however, with flight radii that are very small and approach values that are between 10 and 20% of the channel depth. Previous research [12] has indicated that the SPI guideline as a minimum is appropriate for many resins. But for thermally sensitive resins, radii up to 2.5 times the depth are optimal. Flight radii sizes are shown in Figure 15. When a new screw with radii equal to the depth of the channel was built and installed into the blown film line, the black specks were essentially eliminated.

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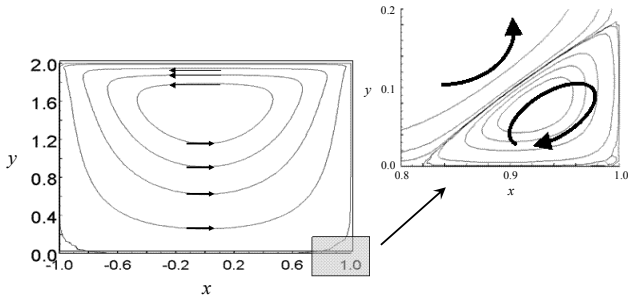

Figure 14. Two dimensional flows in a screw channel with an $H/W = 1$ (channel depth / channel width). The arrows show the recirculation flows. The shaded area in the lower right corner is expanded to show the Moffat eddy [1].

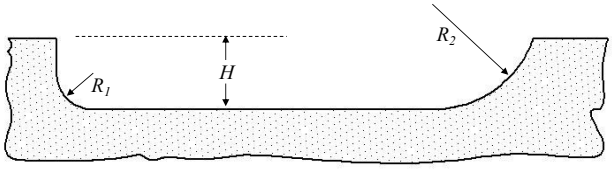

Figure 15. Schematic of small (R_1) and large (R_2) flight radii.

Filler Agglomerates

Some specialty films are produced using masterbatches with high levels of mineral fillers. The filler materials must be compounded with a properly designed process such that fillers are not agglomerated prior to dispersion into the base resin. If agglomerates are produced and contained in the masterbatch, then they are essentially impossible to disperse in the filming process, leading to optical defects in the film. For example, a compounding operation for making a specialty resin from a high impact polystyrene (HIPS) resin and specialty filler chemical was not designed properly. Here the filler chemical was partially agglomerated prior to the melting process in a twin-screw extruder. As shown in Figure 16, the resin was colored black and the filler chemical was white. These white agglomerates could not be eliminated in the final plasticating process (injection molding in this case) and created defects in the product. The goal for this type of application is to produce masterbatches that are free of filler agglomerates since the final film making extrusion process is incapable of dispersing them.

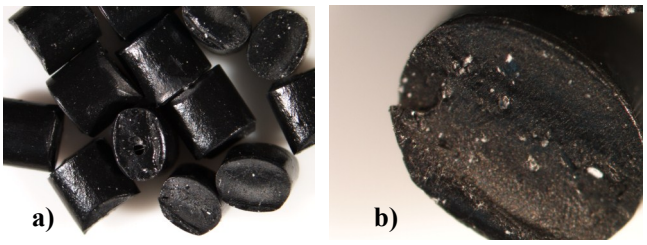

Figure 16. Photographs of specialty HIPS resin pellets made using a poorly designed process. The white specks are filler agglomerates: a) 1x magnification, and b) 4x magnification.

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Discussion

Gel defects are common in PE film products, and they can originate from many different sources, causing a reduction in the product quality and sometimes stopping production. Gel types, identification protocols, and mitigation strategies were presented in this paper. Mitigating or eliminating gels quickly via the best technical solution will reduce costs to the plant and maximize profits.

The equipment and techniques required to diagnose properly many of the gel types can be expensive and require highly trained people. Many small converters will not be able to afford the development of these types of capabilities. Most resin suppliers, however, have the capabilities and are willing to aid customers on the identification and mitigation of the gels.

Summary

This paper describes the different type of gels that are likely to occur in polyolefin film products, techniques for identifying the gel type, and technical solutions to mitigate them from single-screw extrusion processes.

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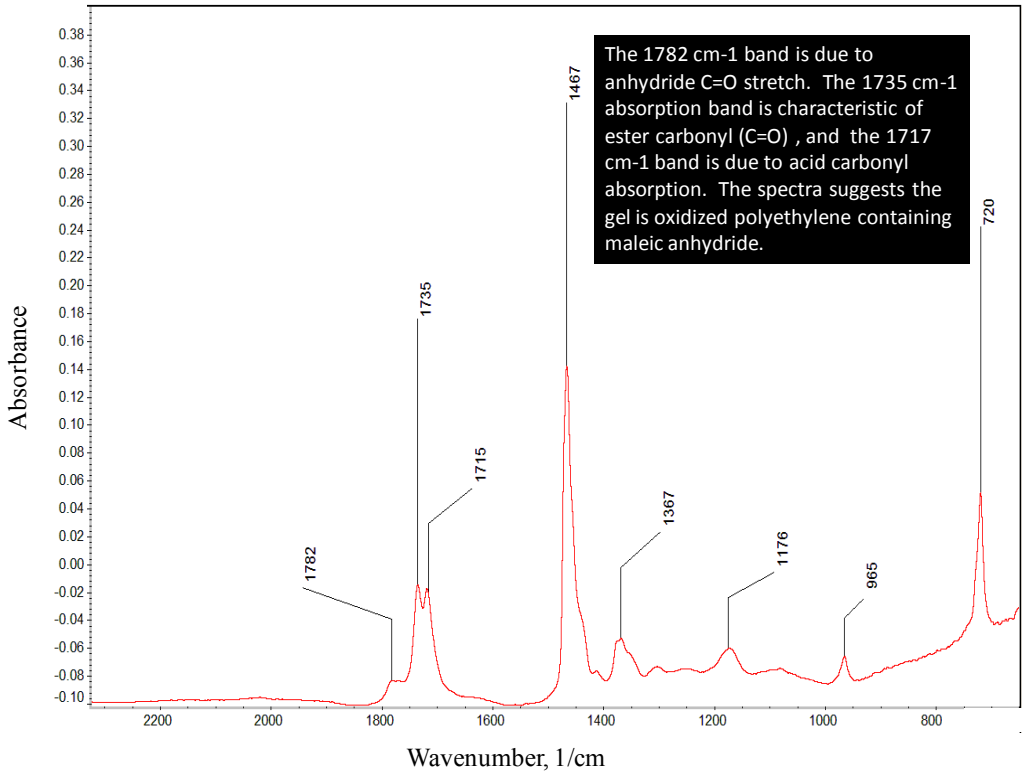

Figure 3. The micro-infrared spectrum of gel shown in Figure 2. The spectrum suggests it is an oxidized polyethylene gel containing maleic anhydride.

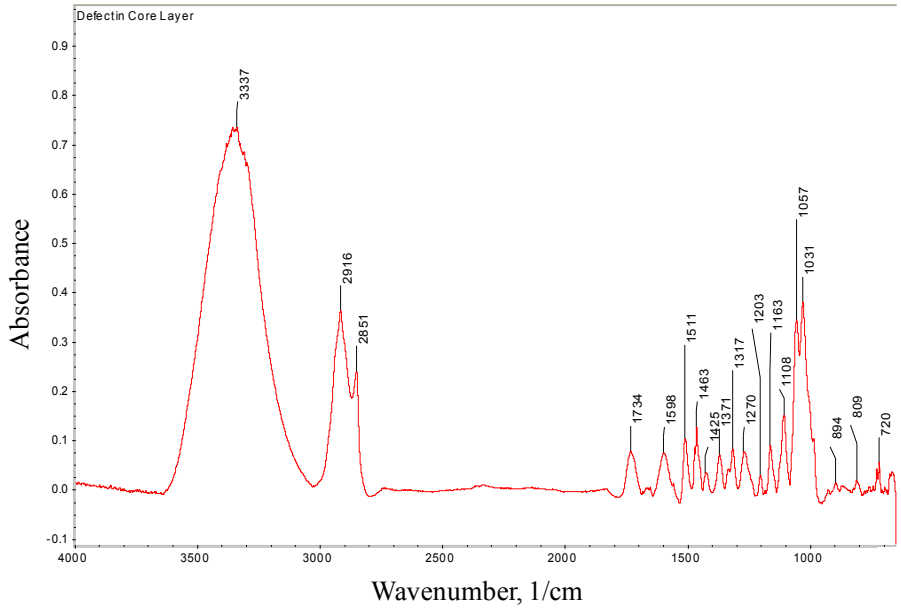

Figure 9. FTIR spectrum of defect in a polyolefin film. The spectrum indicates that the gel is cellulosic fiber and degraded PE resin.

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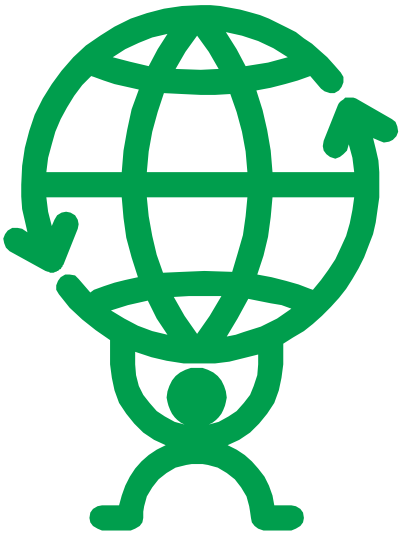

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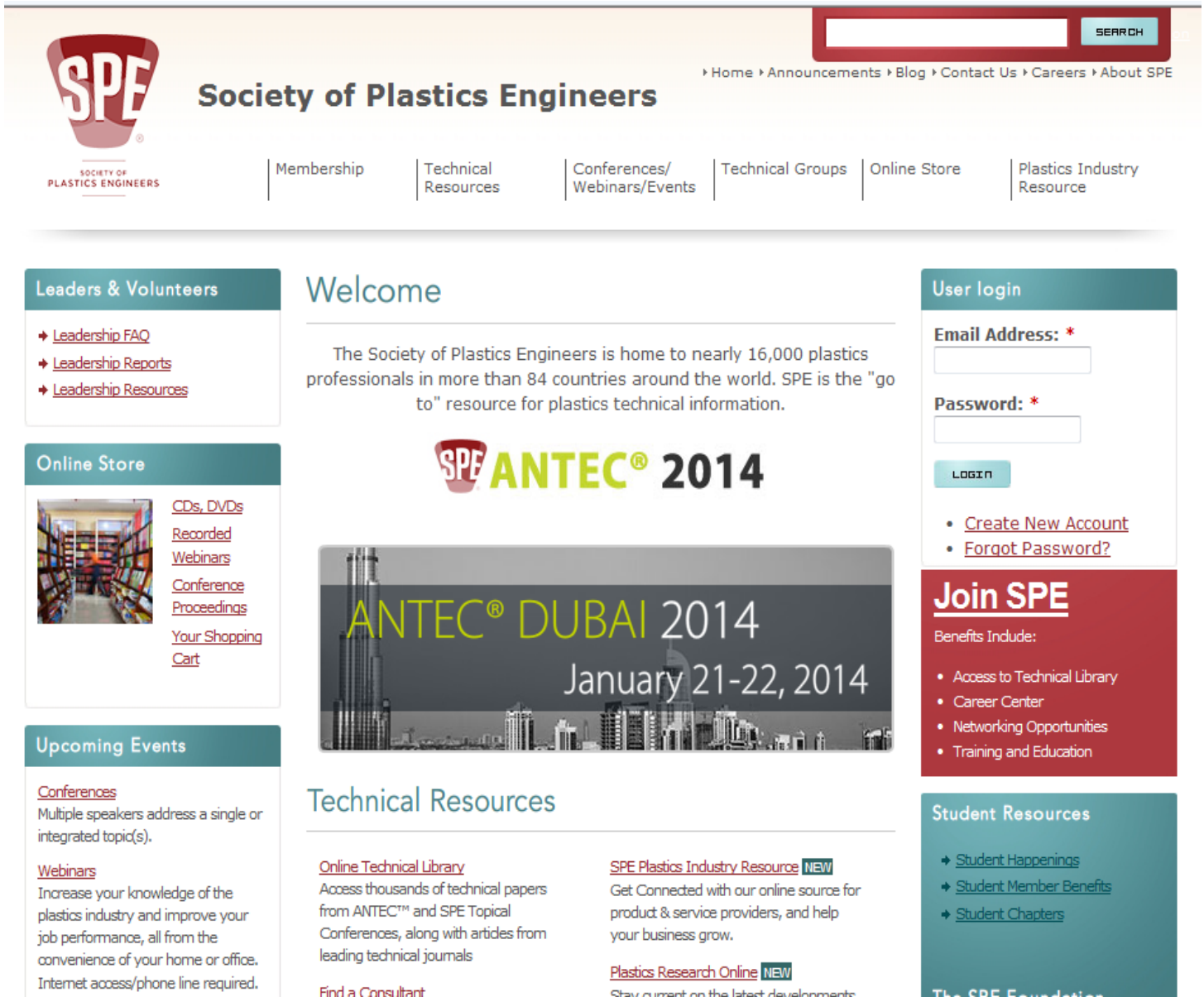

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