Technical

New method to determine correlations in EPDM

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Executive summary

Automatic Continuous Online Monitoring of Polymerization reactions (ACOMP) successfully was used to measure the low and high shear reduced viscosity (RV) of Lion Elastomers' ethylene propylene diene monomer rubber. The analyzed samples came from the production line and were measured quickly to simulate online measurement conditions as best as possible, such as hexane/cement elastomer mixture.

ACOMP generated data that was used to build correlations between the RV and the compositional and physical properties of the EPDM. The correlations showed a linear relationship between the RV measured by ACOMP and Mooney viscosity. Furthermore, the correlations were unique by product grade. In addition, correlations between the RV and the EPDM's percent composition of various monomers and other process chemicals were found.

Because ACOMP can monitor RV and other values of an ongoing reaction in real time, the potential for developing relevant correlations is an expected benefit of continuous data. Through real time data on various polymer properties, combined with these correlations, a new powerful tool for optimized reaction control will result. Ultimately, ACOMP data can be used for both monitoring and enhancing the control of polymer manufacturing at the industrial scale, leading to improved production yields, efficiencies and minimum off-specification product. Additionally, laboratory ACOMP can be used for the discovery and scale-up of new products as they are developed.

TECHNICAL NOTEBOOK
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Automatic Continuous Online Monitoring of Polymerization, or ACOMP, yields continuous data on important characteristics of the reaction and resulting polymers. These polymer properties include conversion, molecular weight, reduced viscosity and shear thinning viscosity.

In this application, the ACOMP detectors that were used included low and high shear capillary viscometers. Other detectors now are being researched for use in ACOMP, such as multi-wavelength ultraviolet absorption spectrometer, a five-angle static light scattering detector, infrared, refractometer, polarimeter (for chiral molecule detection), conductivity, dynamic light scattering and Mie scattering.

Reaction/sample information

Polymer samples were taken approximately every hour over a three-day period at Lion Elastomer's plant in Geismar, La., and transported to Fluence Analytics' laboratory within 12 hours. All analyses were completed within 24 hours of the sample receipt using a Lab ACOMP.

Samples were prepared by diluting them in a pure hexane solvent to achieve a concentration near 5 mg/mL for testing. The concentration was accurately determined after fully characterizing the dried rubber cement concentration of the cement.

Each sample was injected into the ACOMP detector train three times to generate a statistically appropriate average of the characterized reduced viscosity (RV) under both low- and high-shear capillary stress. Once the average RV was determined for each sample, the values were tabulated so that correlations could be observed with respect to the provided Mooney viscosity measurements.

Results

Fig. 1 shows raw detector signals for the low-shear viscometer for the three trials of one EPDM sample. The solvent baseline is clearly seen before the sharp peak, occurring when the EPDM sample hits the detector, which is followed by a quick return to the baseline.

Comparison of this data, along with the high-shear viscometer data to known compositional and physical properties, yields several valuable correlations. **Fig. 2** shows the correlation between reduced viscosity from ACOMP's high-shear viscometer and the Mooney viscosity for two different grades of EPDM rubber during the process of a grade changeover.

This correlation allows ACOMP to target specific grades, potentially allowing for the real time tracking of the Mooney viscosity through constant monitoring of the reduced viscosity.

Furthermore, composition of a polymer can be correlated to the reduced viscosity. **Figs. 3 and 4** show some correlation between reduced viscosity from ACOMP's low-shear viscometer and monomer concentration for two different grades of EPDM rubber during the process of a grade changeover, as evidenced by the groupings in **Figs. 3 and 4**.

The additional insight derived with the correlations of RV to the chemical composition recipes used in this study likely can be utilized to further optimize EPDM production to achieve a target Mooney

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Fig. 1: Raw ACOMP detector signal.

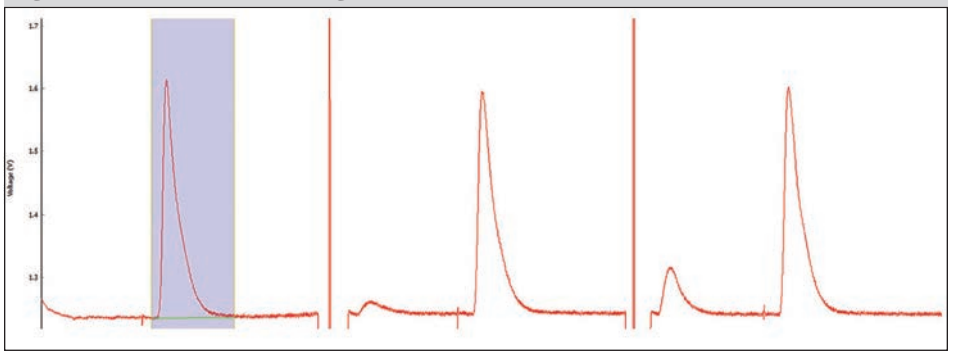


Fig. 2: High shear reduced viscosities vs. Mooney viscosities.

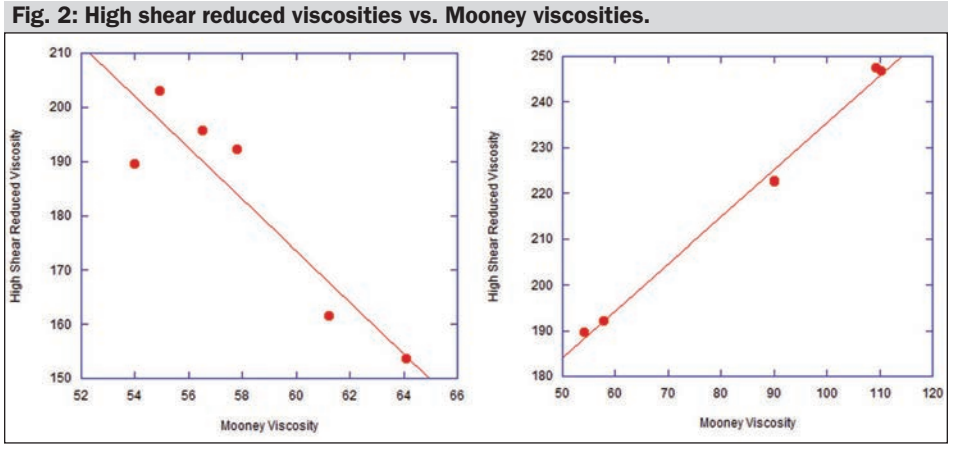
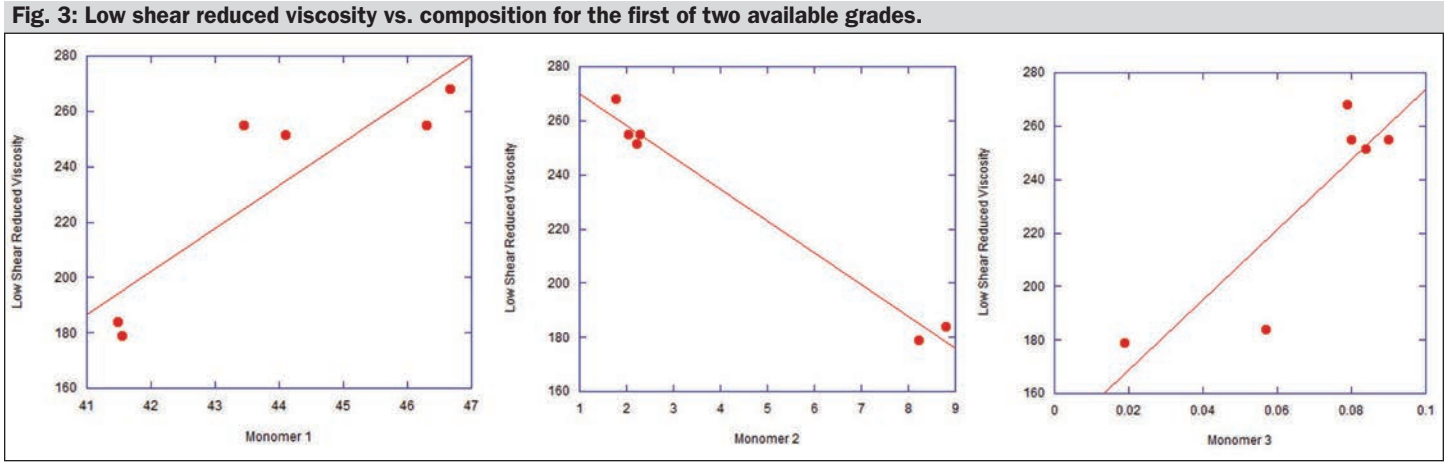
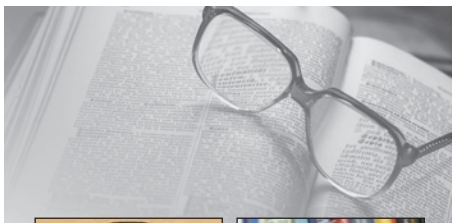
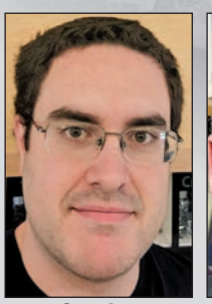


Fig. 3: Low shear reduced viscosity vs. composition for the first of two available grades.







Crosley

Drenski

The authors

Matthew Crosley is an application scientist at Fluence Analytics. He continues to advance the use of ACOMP to new polymer chemistries and applications. He uses his expertise in material science to apply ACOMP's proprietary real time monitoring technology to accelerate research and development times and help chemical companies increase efficiency and product effectiveness.

Prior to Fluence Analytics, Crosley worked at the University of Oklahoma, developing a new technique involving silica sol-gel nano-scale thin films that results in a novel end product, with potential applications as bio-sensors, solar cells, and semiconductors. He received his doctorate in physical chemistry from the University of Oklahoma.

Michael Drenski is co-founder and chief technology officer of Fluence Analytics. Prior to founding Fluence Analytics, he worked as the director for instrumentation at Tulane-PolyRMC, an R&D center which is active in fundamental and applied polymer research.

As the CTO of Fluence Analytics, Drenski leads the technology vision and road map, both setting and executing the company's product development priorities. He is listed as inventor on several patents and has authored and co-authored numerous scientific papers.

He holds a bachelor's in physics from Youngstown State University and a master's in physics from Tulane University.

David Ward is chief technology officer and vice president of R&D for Lion Elastomers. He has more than 30 years of experience developing new industrial catalysts and processes. He has led new product development teams in various technologies for the last 25 years, including olefin polymerization, specialty chemical production, biochemical transformation and bio-refining.

Ward earned both his bachelor's and doctorate in chemistry from the University of Waterloo in Canada.

‘Flexible ecosystem’

ATD exec: Supply chain thrives with insight from stakeholders

By Don Detore
Tire Business

HUNTERSVILLE, N.C.—It’s just not your father’s supply chain anymore.

Today, the supply chain that begins on a rubber plantation in Southeast Asia, eventually moves to a tire manufacturing plant somewhere across the globe and finally ends on the wheels of a consumer vehicle has become more complex—and analytical—than ever before.

According to Bill Hancock, senior vice president, supply chain operations, at American Tire Distributors Inc., today’s supply chain works best as a “flexible ecosystem,” one in which every stakeholder understands the role each needs to play and the insights they can share in order to create a successful ecosystem.


Hancock

Hancock said ATD, North America’s largest tire distributor, sees itself as a full-service, end-to-end supply chain solutions provider.

A big part of that, he said, is working with ATD’s manufacturing partners in order to ensure “they have the right demand signals, the right forecast in place, the right visibility that pulls them all the way down to the consumers on what types of product they need to manufacture, and what quantity and where to have it.”

ATD’s advanced analytics platform, he said, helps to provide deeper levels of insights for manufacturers, “so that they can confidently produce what they need to be successful.”

The advanced analytics platform, he said, helps ATD understand the level and type of inventory needed at its 140 distribution centers and mixing warehouses across North America.

“How do we leverage our hub-and-spoke setup, our national assortment setup to make sure that our customers ultimately get what they want?” Hancock said. “That demand-planning framework is important for both the manufacturer and our customers, because some of our customers have very unique needs. So we’re able to leverage an information-sharing agreement across all of those areas to provide the right visibility and availability.”

A new link in the supply chain has become increasingly important, according to Hancock: the direct-to-consumer piece. It continues to grow as e-commerce becomes the routine rather than the exception.

ATD’s ship-to-home program provides services for all major players selling tires online, according to Hancock.

“If you purchase tires online, there’s a really good chance that it came through our network,” he said. “And when you look at our network, we can provide things that are difficult for others to provide, just based on the number of D/Cs that we have and where they are located.”

He said many of the e-commerce and brick-and-mortar customers are the same.

“We can provide same-day delivery options and next-day delivery options at a price point that is really compelling for those e-commerce providers.”

Hancock said add-on services, such as ATD Express—including partnerships with couriers, on-demand providers and other partial carriers—allows the tire wholesaler “to provide the right level of service and meet those needs” while ensuring it has the capacity required to service larger customers.

Stock levels increase

Capacity has become even more critical in the wake of the COVID-19 pandemic.

There has been some inventory shortages, he said, particularly with light truck tire products. As manufacturers continue to ramp up capacity, stock levels are beginning to increase.

“We’re leveraging our advanced analytics platform with good insights into what the demand looks like and providing those very clear demand signals to our manufacturing partners so that they know exactly what they need to produce and very quickly get back in stock,” Hancock said.

Once the pandemic hit, Hancock said ATD took several decisive steps in order to ensure the safety of its employees and customers.

One of the most successful, he said, is zero-contact delivery that is part of a technology the tire wholesaler rolled out late last year: Traction.

The proprietary mobile delivery app allows customers, for example, to set up delivery alerts and alter the size and/or quantity of a transaction. ATD, meanwhile, can apply credits and process warranties and/or returns seamlessly.

ATD officials said its associates “have been there every step of the way” as the wholesaler continues to operate during the COVID-19 pandemic.

“It’s a very user-friendly experience at that point of delivery,” Hancock said. “And that’s on top of being able to support that zero-contact interface for the customer.”

Hancock said ATD still can provide proof of delivery for customers, “but still give them all of the insights and information customization that they’re looking for.”

The tire wholesaler also has implemented several other provisions as directed by the Centers for Disease Control and Prevention, including:

- Deep cleaning buildings and trucks when an occurrence or diagnosis of COVID-19 becomes known;
- Contacting associates and customers who may have come into contact with an individual who has contracted the disease;
- Changing the way teams interact with each other inside the buildings, including social distancing; and
- Requiring the appropriate personal protective equipment.

“I can’t tell you how proud I am of all of our field associates and support teams,” Hancock said. “It’s been a total company effort.

“These men and women are out there driving our trucks, running our warehouses and staying in direct contact with our customers,” he said. “They have been there every step of way.”

Despite the recent uptick in COVID-19 cases across the U.S., Hancock said he and the company are cautiously optimistic about the future.

“We’re in a customer-centric mindset,” Hancock said. “The way we invest in the technology as a tool, the way we train, coach and onboard our team is to make sure that we are empowering people to work hand-in-hand with our customers, because we know everyone has a different set of needs.

“It’s all about building an environment that allows our customers to interact with ATD on their terms in ways that benefit them and their business.”

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EPDM

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viscosity. It is expected that correlations to lab data, utilizing a much larger process dataset, will be on a grade-dependent basis, for example a range for RV or high-shear RV will be developed to target specific end-performance properties.

Conclusion

ACOMP can directly monitor and characterize low and high shear RV during Lion Elastomers’ EPDM polymerization process. This information is important with respect to characterizing the polymer Mooney viscosity property in real time, and it also is useful in providing insights into the current state of the polymer composition.

This can optimize production rates and efficiencies, including cycle times and product yields. The ACOMP data generated during this study indicates that the ACOMP system is capable of monitoring and controlling polymer manufacturing on an industrial scale, leading to improved production efficiencies and minimum off-specification product.

Fig. 4: Low shear reduced viscosity vs. composition for the second available grade.