Determining Correlations in EPDM between Compositional Properties, Rheological Properties, and Detectable Trends via ACOMP

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Abstract

Automatic Continuous Online Monitoring of Polymerization reactions (ACOMP) was successfully used to measure the low and high shear reduced viscosity (RV) of Lion Elastomers' ethylene propylene diene monomer rubber (EPDM). The analyzed samples came from the production line and were quickly measured to simulate online measurement conditions as best as possible, i.e. 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 realtime, the potential for developing relevant correlations is an expected benefit of continuous data. Through realtime 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, Lab ACOMP can be used for the discovery and scale up of new products as they are developed.

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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 are now being researched for use in ACOMP, such as multi-wavelength ultraviolet absorption spectrometer (UV), a five-angle static light scattering detector (LS), IR (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's plant in Geismar, LA and transported to Fluence Analytics' lab 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 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

Figure 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.

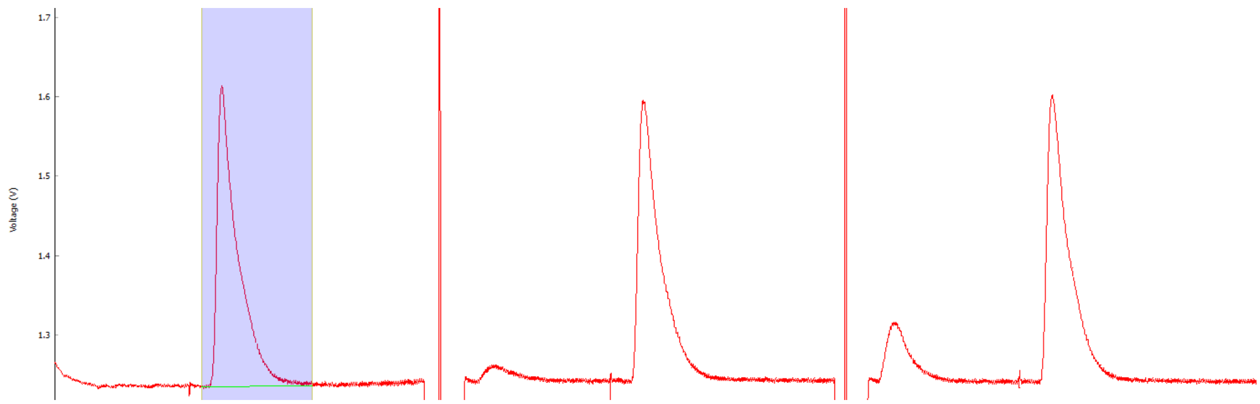

Figure 1: Raw ACOMP detector signal

Comparison of this data, along with the high shear viscometer data to known compositional and physical properties, yields several valuable correlations. Figure 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.

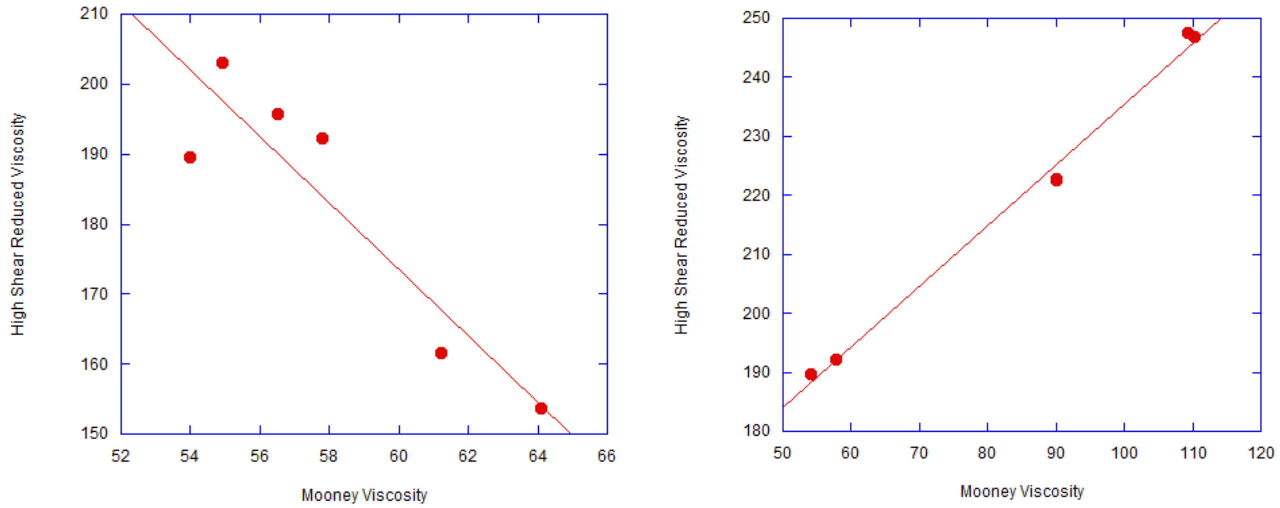

Figure 2: High Shear Reduced Viscosities vs. Mooney Viscosities

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

Furthermore, composition of a polymer can be correlated to the reduced viscosity. Figures 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 below.

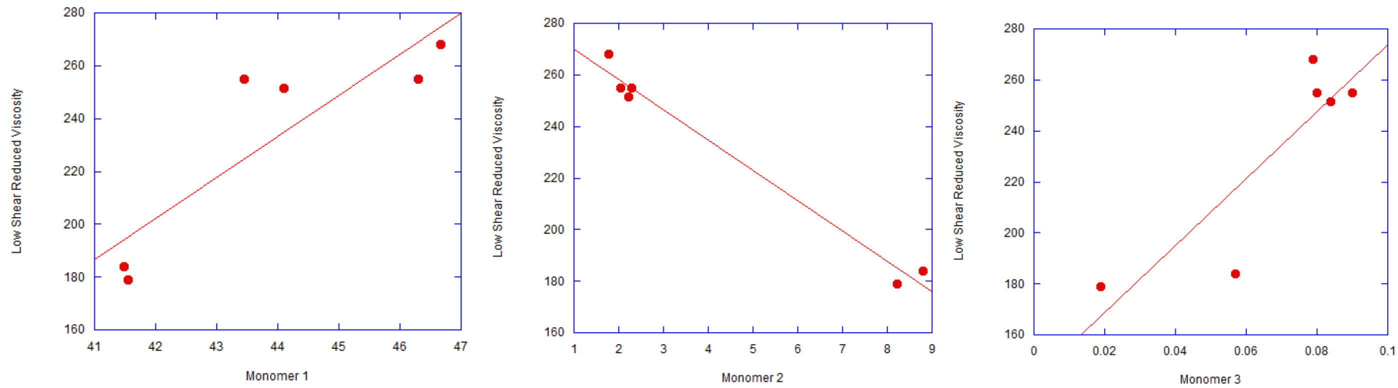

Figure 3: Low Shear Reduced Viscosity vs. Composition for the First of Two Available Grades

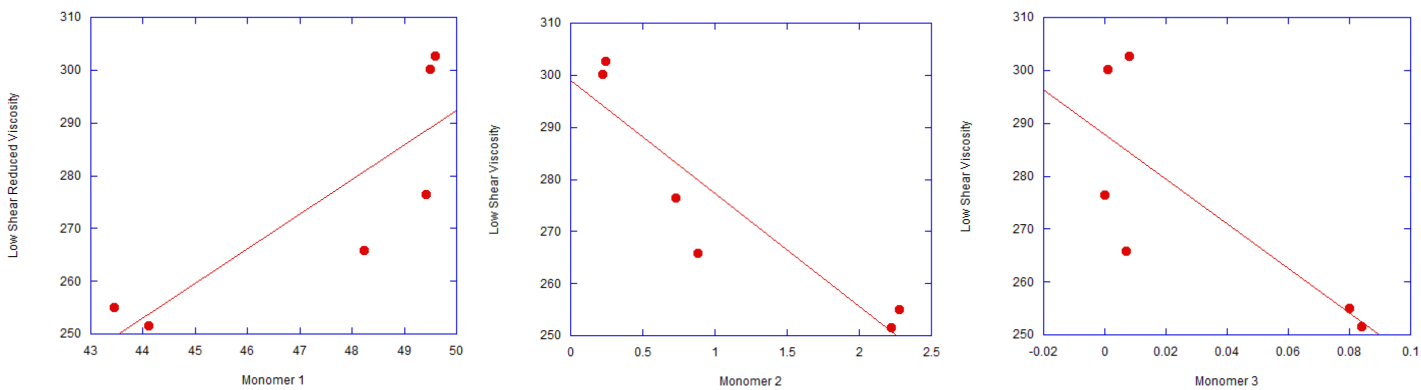

Figure 4: Low Shear Reduced Viscosity vs. Composition for the Second Available Grade

The additional insight derived with the correlations of RV to the chemical composition recipes used in this study can likely be utilized to further optimize EPDM production to achieve a target Mooney viscosity. It is expected that correlations to lab data, utilizing a much larger process dataset, will be on a grade dependent basis, i.e. 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 is also 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.

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