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Real-Time NIR Spectroscopy in Polymer Quality Control

By Del Williams

When manufacturing plastics, rubbers, elastomers, and adhesives, it is critical to monitor polymerization processes for quality and cost at every production stage from material receipt through final inspection.



Instant polymer testing drives quality control and offers the opportunity of immediate adjustments if the process drifts.

Whether polymerization is bulk, solution, suspension, or emulsion, gathering essential data can help to prevent costly quality issues. Otherwise, internal production failures can require rework or scrap, and external failures can lead to warranty claims, recalls and even potential liability.

While traditional testing is critical in determining polymerization factors from simple moisture content to complete chemical analysis, it is time-consuming, labor-intensive and incurs substantial ongoing costs, in terms of the purchase and disposal of reagents and chemicals.

NIR Spectroscopy

Conventionally, testing requires sample preparation and can take 5 to 15 minutes for moisture testing and 24 to 48 hours for more complex chemical testing. Now, a new approach using NIR spectroscopy can provide immediate, real-time, lab-quality readings by using a noncontact method. This method can provide moisture, composition and full spectrum readings for a fraction of the running costs of traditional processes.

"Real-time NIR measurement enables continuous monitoring and optimization of polymerization processes," says John Bogart, managing director of Kett US, a manufacturer of moisture meters, composition analyzers and full spectrum testing equipment. "It provides more timely quality assurance data than a team of QA people, using traditional testing methods, and 100 percent of product can be inspected."



Kett KB270F desktop NIR moisture analyzer.

Instant testing offers superior quality control and immediate adjustment if a process starts to drift out of its target range. Results can improve batch consistency and yield, while helping to eliminate batch failure and reduce material waste.

NIR spectroscopy bounces beams of light off a material and measures how much light is absorbed across certain wavelengths of the electromagnetic spectrum (700 to 2,500 nm). This produces a sort of



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"optical fingerprint," with the device calculating the desired measurements after the meter has initially been calibrated to a lab or production standard.

Filter-Based NIR Systems

NIR devices fall into two general categories: filter-based and full-spectrum. Filter-based products block visible light and only allow certain wavelengths to enter the device. These are simpler than full-spectrum devices and work within a limited range of wavelengths.

"Filter-based products are less expensive, easier to calibrate and to maintain," says Bogart. "The devices are also more robust and typically more stable for operators to use. The drawback is that the instruments are more limited in what they can test."

Traditionally, a slower method of measuring moisture is a loss on drying test, which measures the total material weight change before and after drying. However, these tests typically require a sample to be prepared and brought back to the lab. The test takes at least 15 minutes and up to several hours to perform. It also requires the samples to be altered or destroyed.

NIR moisture meters are the simplest and most economical type of filter-based device. These are often used to inspect incoming raw materials. However, the meters can be used anywhere in the production process where achieving a specific moisture content is important. With these devices, reflected light is filtered to a narrow portion of the NIR spectrum, usually at only one or two wavelengths.

Bulk polymerization does not usually require much more than a moisture meter, since the reaction proceeds without a solvent or diluent. But, moisture meters can also be used with more complex forms of polymer, where the manufacturer may want to measure moisture anyway.

Among filter-based devices, NIR composition analyzers are a step up in measuring capability and can simultaneously measure a few different chemical components in the polymer while being limited to about six or seven wavelengths of light. In addition to moisture, the devices may also be used to measure residual oils or solvents, which can cause contamination.

A third traditional use of this type of device is to measure coating thickness or film thickness. Both desktop and real-time online process measurements can be provided in many cases. Generally, the price point of the NIR composition analyzer is substantially lower than alternative technologies.

Full-Spectrum NIR Systems

Full-spectrum NIR devices can measure more than 500 wavelengths to determine if materials meet a wide range of criteria. Once the device is specifically calibrated for a particular application, the widest number of targeted factors can be measured in real time.

In addition to moisture, polymer manufacturers may measure density, viscosity, melt flow rate (MFR), and functional groups. Such testing is common during emulsion polymerization, which is used to create latexes and synthetic polymer colloids for paint, coatings, rubber, binders, and adhesives.

The testing is also helpful for carrying out suspension polymerization processes, which are often used to produce polymer beads. These processes generally involve an initiator and comonomers, with the final polymers suspended in an aqueous phase that contains additives and residual monomer.

Traditional direct measurements of these parameters can take up to 48 hours for results. The tests are complicated. Various substances must be broken down into their underlying compositions. The tests require meticulous set up and execution to avoid errors.

By contrast, full-spectrum NIR testing is instant and can produce results in real time, without relying on laborious and difficult techniques. Such real-time monitoring of the polymerization process can give the manufacturer the ability to make immediate adjustments to optimize it.

While polymer manufacturers are more familiar with traditional methods, for those concerned not only with quality but also with profitability, selecting the NIR approach that works best for the application, whether it be a moisture meter, composition analyzer or full-spectrum tester, can have a major impact on the bottom line.

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