The Power of Spectroscopy – Moving Out of the Lab to Resolve Production and Processing Challenges

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Spectroscopy has long been a staple of laboratory analysis-measuring the interaction between samples and light to make observations about chemical composition and characteristics. Continuing advances in electro-optics, high-speed array detectors, inexpensive optical fibres and powerful computers have spurred the growth of modular spectroscopy. The size, flexibility and affordability of these new instruments bring previously lab-bound measurements to the production floor, with the ability to positively impact the entire process stream.

As processing lines face ever increasing pressure to speed throughput, while increasing quality and yield, small-footprint modular systems can be rapidly configured for a variety of absorbance, reflectance and emission spectroscopic measurements. Potential applications are as far-ranging as monitoring of dye baths for carpeting, analysis of chemical dissolution processes for pharmaceuticals production and verification of colour and appearance of finished goods.

Operation Gumball–Demonstrating a Range of Optical Sensing Technologies

Consider the simple pleasures of the humble gumball: all the satisfying chewiness of bubblegum and the pleasant crunch of a hard-candy shell, in a sugary, bite-sized sphere. The gumball is an ideal subject to demonstrate a range of spectroscopy techniques, from Raman analysis to reflection measurement. The gumball sorting experiments outlined below demonstrate the power of spectroscopy to solve analysis problems for lab researchers, OEMs and industrial customers. Each measurement can be taken in the lab or scaled to a process environment.

To bring together this range of spectroscopy techniques in an easy to observe format, Ocean Optics, a developer of spectroscopy applications solutions, built a gumball sorter. This sophisticated system comprises multiple spectroscopy measurement stations positioned throughout an 8-foot-tall, 6-sided structure. A series of stainless steel rails mounted to the structure delivers the samples to each station, where spectrometer setups determine gumball sample characteristics such as chemical composition, colour and reflectivity *(Figure 1)*.

Figure 1. Gumball sorter

Additional stations measure absorbance and fluorescence of food colouring and flavourants, transmission characteristics of plastic containers and oxygen content in packaging headspace.

A large hopper holds a collection of 3,000 brightly coloured gumballs (both sugar-rich and sugar-free) and marbles, which look remarkably similar to the gumballs. Each of these sample characteristics provides an opportunity to sort the samples using modular spectroscopy.

As the samples are released from the hopper, they feed through a gravity-driven system of stainless steel rails to their stations. Each station acts as a sort of pass-fail analyser: a measurement is made and the result registers the response in software, which then triggers a second device – a motorised gate – to direct the sample to the appropriate destination *(Figure 2)*. But less important than the logistics of the display are the range and flexibility of the spectral sensing techniques it presents.

Experimental Conditions: NIR Diffuse Reflection

Gumballs and marbles are composed of very different materials, yet their similar sizes, shapes and colours make them difficult to distinguish by sight alone. When properly measured, spectral reflectance can yield much of the same information as the eye, but it does so more quantitatively and objectively. A spectral reflectance measurement can compare two yellow objects, or different textures. It can also offer information about the material from which a sample is made, since light that is not reflected from a sample is absorbed due to its chemical composition, otherwise it is scattered or transmitted.

Figure 2. Gumball station

Reflectance measurements can measure the colour of a sample, or examine differences between objects for sorting or quality control. The samples may be automotive parts, paint, coffee beans, dyed human hair, lizards, or gumballs. In the gumball sorter setup, NIR diffuse reflection spectroscopy is used to separate gumballs from marbles. In this application, the NIR approach focuses on capturing the distinct reflection intensity differences for each sample's coating and avoids trying to detect slight colour differences that one would observe with a visible spectrometer. An NIR spectrometer with response from 900-1700 nm was used for these measurements.

The gumball sorter NIR setup quickly and accurately sorted red, blue and green marbles from gumballs, based on differences in reflectivity. The marbles are glossier and have a higher degree of specular reflectivity, resulting in lower diffuse reflection intensity relative to the gumballs *(Figure 3)*. As evidenced by the spectral features in the gumball spectra, additional information on the chemical composition of the samples including sugar and moisture content could be extracted from the spectra using the appropriate chemometrics models. Rejected marbles were binned separately and the gumballs continued through the sorter for further analysis.

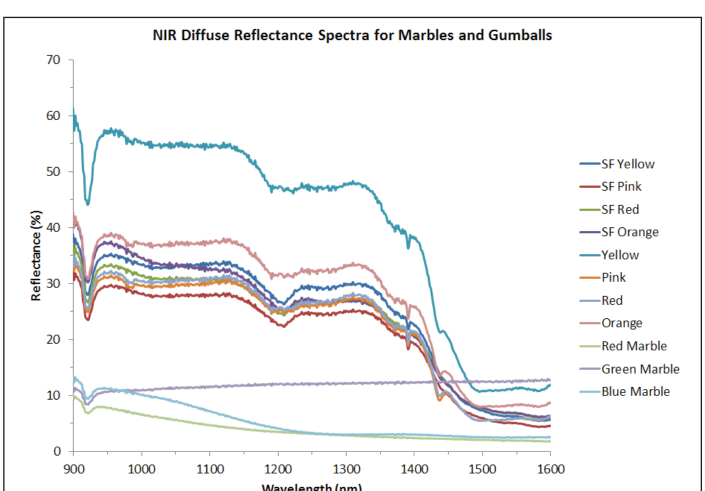

Figure 3. Marbles vs. gumballs spectra

Experimental Conditions: Raman for Chemical Analysis

Raman spectroscopy is a very useful technique for determining chemical composition, especially because of the strong specificity of the Raman signature of many materials; it just requires the right equipment to sift through the available information. Raman is especially useful for quantifying mixtures like gumballs (the coating and the gum inside have different chemical composition), as even minor spectral differences are discernible.

In the gumball sorter, a modular Raman spectrometer setup was used to distinguish sugar-rich from sugar-free gumballs. Samples were captured in a Raman sample holder equipped with a Raman fibre optic probe. Excitation light was provided by a 785 nm laser and the Raman spectrum was collected with a high sensitivity spectrometer.

The sugar-free gumballs have a distinct Raman shift near 881 cm^{-1} that corresponds to the peak for sorbitol *(Figure 4)*, a sugar alcohol often used as a sugar substitute in diet foods and sugar-free gum.

Using NIR excitation light for Raman spectroscopy at 785 nm minimises fluorescence signals, which dramatically simplifies chemical identification and fingerprinting. In addition, Raman can capture data from a sample contained in plastic or other materials (like packaging) that are optically transparent to the wavelengths of interest.

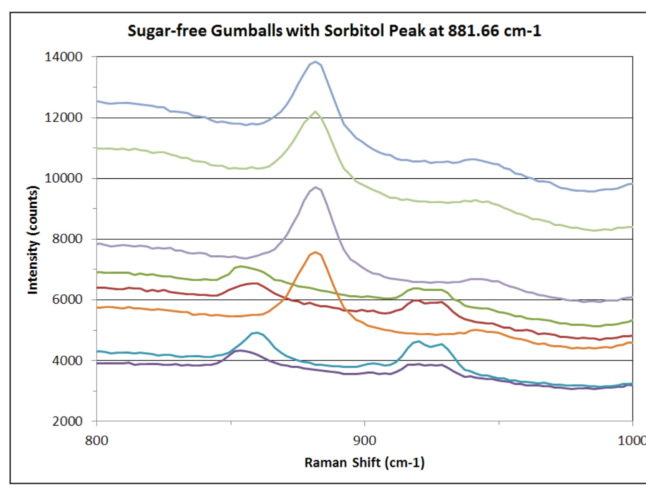

Figure 4. Sorbitol spectra

Experimental Conditions: Reflected Colour of Sugar-rich and Sugar-free Gumballs

Colour assessment with the human eye can be highly subjective. Changes in lighting, viewing angle and in the observer himself can all influence the perception of colour. Detailed spectral information can bring quantitative objectivity to the assessment of colour. Small-footprint modular spectroscopy systems are readily configured for quality control experiments such as verification of colour and appearance of raw materials and finished goods. With the gumball sorter setup, three experiment stations were used to demonstrate the flexibility in design approach that is available with sensing tools for colour measurement.

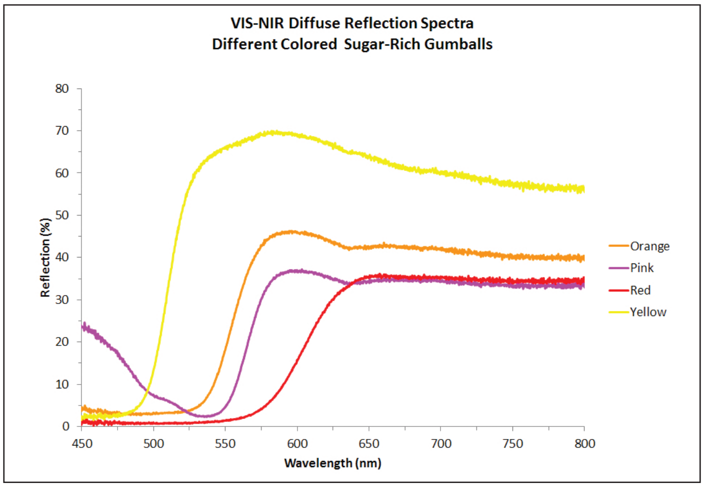

Figure 5. Colour spectra of sugar-rich gumballs.

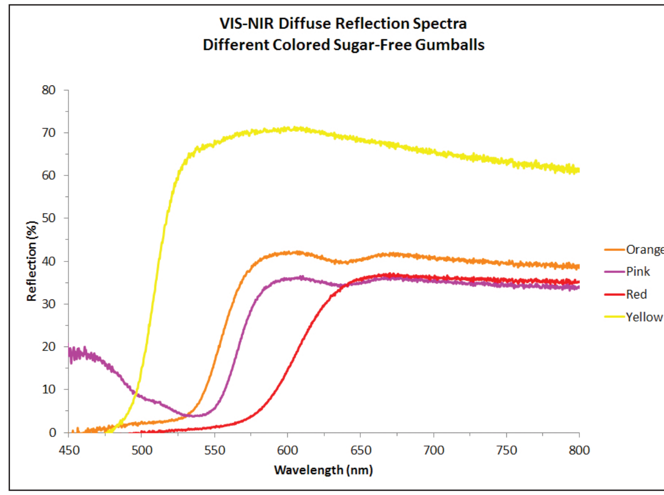

Figure 6. Colour spectra of sugar-free gumballs.

The sugar-rich gumball colour station was anchored by a spectrometer distinguished by its low stray light and high thermal stability performance.

The colour of the sugar-rich gumballs was easy to observe, although there is visible variability from gumball to gumball *(Figure 5)*. This variability is deduced as being a result of batch differences in manufacturing (a 'close enough' standard is probably all the manufacturer was aiming for), fading of the coating colour with exposure to air and light, and some 'bleed-over' of colours from adjacent gumballs as they collide while moving through the sorting process.

A linear CCD-array spectrometer with a tungsten halogen source for illumination and a fibre optic reflection probe for sampling collection was used for analysing colour in sugar-free gumballs.

Similar to the observed results for sugar-rich gumballs, the relative colour characteristics of the sugar-free gumballs were readily apparent. Interestingly, the colour of coatings for the sugar-rich and sugar-free gumballs revealed differences in relative intensity that varied from colour to colour *(Figure 6)*.

Colour measurement was also used to demonstrate an 'at-line' measurement. As the gumballs exited the process stream, they were collected into containers based on colour and sugar content, for additional colour quality assessment. Here a spot check of gumball colour was performed using a compact microspectrometer.

A good microspectrometer delivers great performance despite its device size, which is ideal for embedding into process streams or OEM devices where one or more wavelengths are being monitored and a highly reproducible result is desired.

The Significance of Gumball Sorting

Although this simple gumball sorter isn't designed for high throughput manufacturing or likely to end up on a factory floor, every instrument and each spectroscopy technique it demonstrates can today be implemented into a process stream, used in a lab or field setting or incorporated into another sensing device. Modular spectroscopy is especially suited for process environments, where monitoring of incoming raw materials and finished goods is critical. Flexibility in detectors, light sources and sampling optics allows for deeper implementation of instrumentation into the process stream, and makes it much simpler to optimise setups. Multiple-point sampling and redundant sensing are easily integrated into on-line applications.

Modular spectroscopy brings reliability, ease of use, convenience and increased productivity to critical process measurements. Multi-wavelength spectroscopic sensing has dramatically decreased in cost in recent times when considered on a per-measurement basis. Miniature spectrometer systems can be custom configured and are often much less expensive than traditional benchtop instruments. Furthermore, these systems add flexibility so that reworking them for a changing process is not a particularly expensive proposition.

How Many Lamps Does an AAS Laboratory Need

This problem is familiar to every AAS user: For each element, an individual hollow-cathode lamp is needed. Multi-element analyses take an enormous amount of time and work. Flexibility, too, is significantly restricted, new elements require new lamps. With the High-Resolution Continuum Source (HR-CS) AAS technology used in the contrAA® range from **Analytik Jena,** these disadvantages of conventional AAS have become a thing of the past: a xenon lamp generates a continuous emission spectrum of consistently high intensity across the entire relevant wavelength range. Thus it is possible to analyse all elements of a sample directly, using a fast sequential procedure. Thanks to alternative wavelengths, it is possible to extend the measuring range towards higher concentrations, doing away with the need for time-consuming dilutions.

In conjunction with a high-resolution double monochromator and a CCD line detector, it is possible to visually represent the absorption spectrum of each sample. Users 'see what they are measuring' and are thus able to detect and correct any interferences promptly and easily.

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