Not just moisture: a review of some commercially successful near infrared applications

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Introduction

On-line measurements for a broad cross-section of the process industries have been very well served by infrared absorption technology for many years. Moisture measurement has tended to dominate because it is a parameter which can be modified by control action on a dryer or conditioning system. Moisture content can have a big impact on product quality and yield and the incentive for process control purposes is self-evident.

The packaging and converting industries have also benefited from this technology and the measurement of plastic film thickness or the coat-weight of adhesives, for example, are equally successful and are now well accepted as offering an accurate, safe and selective technique compared to nuclear technology.

Improvements over the years have centred upon enhancing the quality of such measurements, ensuring that the natural product variations such as colour, particle size and even composition, in the case of foods, do not influence a moisture measurement. Equally important in the converting industry has been the need to design a measurement to be insensitive to changes in coating or film pigmentation levels, or specifically for coatings, variation in the scattering properties of the substrate.

In the background, instrument manufacturers have also had to overcome the more abstract difficulties associated with on-line measurement over which they may have no control, such as product to sensor distance variation, relative humidity and ambient lighting variation and product and ambient temperature variation. Nowadays, high performance, irrespective of these variables is almost taken for granted by users as solved by modern sensor design today. Yet, in fact, in the development of on-line instrumentation, these have all been issues that without question have needed resolution to provide a fast, reliable, robust, non-contact on-line measurement. In reality, only a few manufacturers offer such competence in their sensors and adapted laboratory technology still struggles to offer these facilities which the manufacturer, in a process industry, should be able to take for granted.

Infrared technology has latterly become very well established in the more complex non-moisture applications with instrumentation that still retains the robust, fast, non-contact, continuous nature associated with the simpler measurements.

This paper takes a look at four such examples of using infrared technology in radically different applications in true on-line environments and discusses some of the interesting considerations that were necessary for their successful implementation world-wide.

From Near Infrared Spectroscopy: The Future Waves

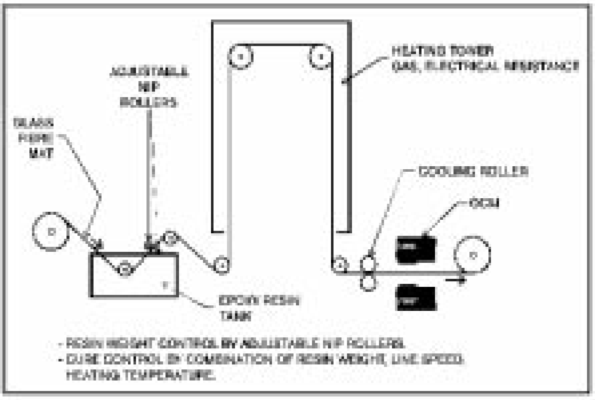

Figure 1. Schematic of epoxy impregnation process.

Measurement of the degree of cure of epoxy resin in fibreglass

Printed circuit card substrates are produced from a fibreglass woven mat that is subsequently impregnated with an epoxy resin. After the impregnation stage (Figure 1) the material passes through a heat curing process to accelerate the polymer cross-linking of the epoxy resin. It is critical in this process to ensure that the finished intermediate product has the correct degree of polymerisation or cure so that when subsequent multi-layer lamination is carried out there is adequate bonding of the layers in the final heat treatment process. Near infrared (NIR) transmission spectra (Figure 2) of the resin at various degrees of cure reveal a disappearing epoxy peak due to C–O stretch in the epoxy ring, at 2.206 μm, consistent with increasing cure level. While the change is obvious in Figure 2 between two extreme samples, the actual requirement is to detect subtle cure variations and, practically, very small changes in absorption. An added complication is the variation in resin weight with time or product type which will clearly influence the size of the epoxy absorption band for a given cure level. An absorption due to the resin occurs at 2.465

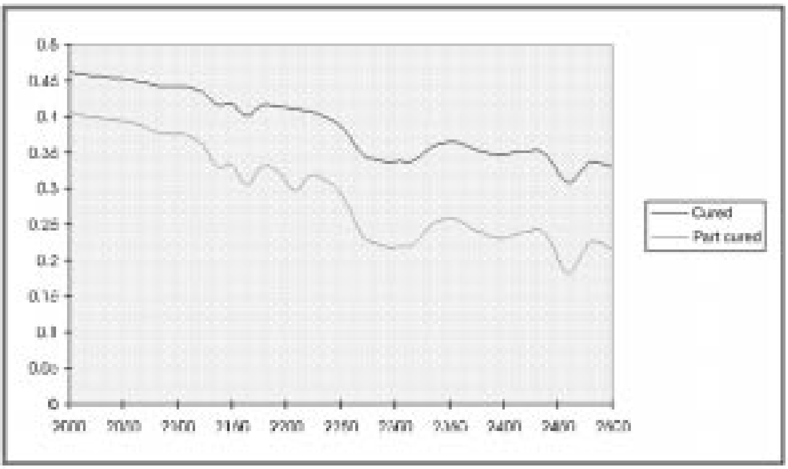

Figure 2. Transmission spectra of epoxy resin at two cure levels.

μm which provides a means of monitoring epoxy resin weight which then allows correction of the cure measurement with resin weight variation.

These spectra show quite fine structure especially for the resin and epoxy peaks with neighbouring absorptions very close by as potential interferers. It is therefore essential to use accurately placed, very narrow band filters to ensure an adequate sensitivity to absorption changes at the epoxy band. Putting the requirements of an on-line measurement into perspective, to measure the degree of cure to the resolution required by the customer, i.e. to within 2 cure units, necessitates photometric performance of better than 0.05% absorption. A further challenge is provided because circuit card material is produced on web widths of up to 1.5 metres and the degree of cure can vary as a function of web width. Since the material is continuously impregnated at line speeds of up to 50 m min^{-1}, it is essential to scan the web providing multiple measurements every second to provide meaningful cross and down web profile information.

Conventional testing by FT-NIR off-line sampling takes several minutes is historical and therefore not very useful since this is a continuous production process. The on-line system compares very favourably with the precision of the off-line method (±1.5–2.0 cure units) despite having an extremely short, 200 ms, sampling time.

The sensor employed for this application is the MM55 Transmission instrument. Infrared light generated by a quartz halogen source passes through a filter wheel rotating at 25 Hz containing optical interference filters. The infrared light passes through the impregnated web and is focused onto a peltier cooled lead sulphide detector. The rapid measurement rate allows near-continuous monitoring of the sheet with excellent resolution of impregnation weight across the web as the sensor is scanned. Data reduction involves a complex algorithm which monitors both epoxy peak disappearance and resin weight and automatically corrects for local resin weight fluctuations, providing a clean signal relating only to degree of cure. Data so collected is normally presented on a computer profile display system in two dimensions to provide a clear picture of cure profile.

It is also interesting to note that the variation in fibre glass substrate matt density and scattering properties (translucency) can cause apparent variation in cure by changing optical pathlengths. It is therefore necessary to incorporate into the optical design of the sensor a method of correcting for this interference. This is achieved by collecting only scattered light and by selection of reference wavelengths that allow continual monitoring of scattering changes.

Measurement precision over a wide range of products from near transparent glasses to strongly scattering, opaque materials have been successfully measured with this arrangement. Figure 3 shows a typical example of a calibration graph of the transmission sensor on the degree of cure measurement.

Practically, the signal derived from the near infrared sensor allows control of degree of cure by closed loop control of the drying tunnel. This particular approach has proved very successful and has now been implemented in many factories within one of the largest circuit card base material manufacturers in the world.

Measurement of very thin coatings on reflective surfaces

Lacquer coatings are often applied to aluminium foils and metallised films in a number of industrial converting applications. Common examples of the end products would be yoghurt lid tops, heat sealed to plastic containers, “end stock” which forms the lid of a two piece aluminium beverage can and the diverse range of packaging materials used for products such as potato chips. In most cases, the lacquer coating provides a protective film to avoid direct contact of the foodstuffs with the aluminium. Typically such coatings may be between 0.5 and 10.0 micrometers. An interesting example which demonstrates the sensitivity and selectivity of the infrared absorption

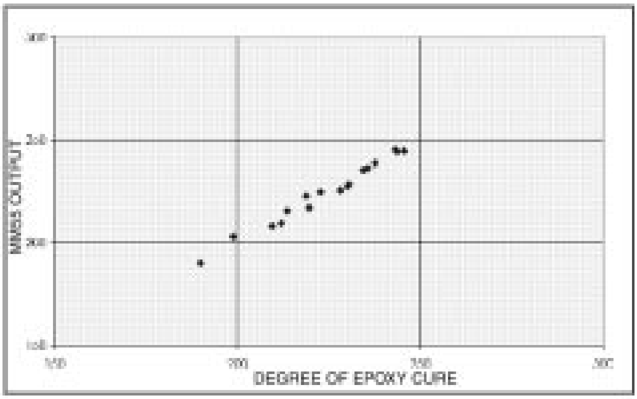

Figure 3. MM55 calibration graph for degree of epoxy cure.

technique is that concerned with beverage can end stock production. Initially aluminium foil of around 200 µm thickness is coated with an organic lacquer, often vinyl based.

However, since this material then undergoes an aggressive forming process by mechanical stamping it is essential to provide a uniformly distributed lubricant layer across the sheet surface prior to the operation, to prevent damage to the presses.

Wax lubricant coatings are applied to the vinyl coated surfaces at extremely low coating weights, at levels of around (50–150 mg m^{-2} or 50–150 nm thickness). The challenge to an on-line infrared measurement is complex for several reasons. To measure the wax coating independently of the underlying, much thicker vinyl coat is important, if meaningful coat weight control is to be achieved. Secondly, thin coatings tend to be locally quite flat in optical terms and can therefore introduce severe optical interference which will, if not corrected for, distort the absorption measurement as constructive/destructive interference changes reflected energy levels in a wavelength dependent way. Finally, aluminium surfaces are far from consistent, ranging from a matt to shiny appearance indicative of changing reflectivity, usually in a wavelength dependent manner.

Differentiation between a vinyl lacquer and a hydrocarbon wax is assisted by the large difference in absorption coefficient at the longer infrared wavelengths around 3.4 micrometers, where there are, of course, much stronger fundamental –CH absorptions. Careful wavelength selection and the use of narrow band interference filters allows this to be achieved with excellent signal to noise performance, vital since this is also an application where scanning a sensor to obtain cross web, profile information is critical. Reflectivity changes in the substrate are measured relatively by analysing reflectance information at additional wavelengths outside the regions of absorption. It is therefore possible to calculate automatically and to compensate for these changes in the region of the lubricant absorption.

The optical design of the sensor is critical for success in this application to avoid disturbance by optical interference. Use of the Brewster angle phenomenon enables effective cancellation of fringe effects. Figure 4 shows that for the two planes of polarised light, upon interaction with an optical surface of refractive index *n*, such as a lacquer coating, reflection intensity will be dependent upon the angle of incidence of the light. It can be seen that at around 56° one of the planes of polarised light shows zero reflection and therefore all of it passes into the coating. Elimination of light reflection from the surface by this means ensures that interference conditions cannot occur and therefore a true absorption measurement can be made. Figure 5 shows a

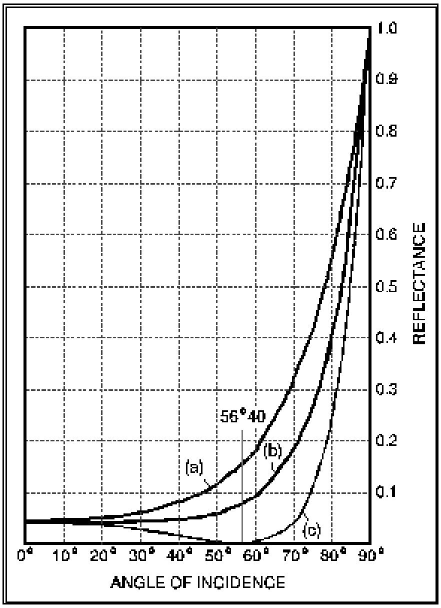

Figure 4. Illustration of the Brewster Angle phenomenon.

schematic of the MM55 Lacquer on Foil sensor specifically designed to eliminate the effects of optical interference that can occur in the coating.

Light from the source, after passing through the filter wheel, is polarised to provide the correct plane of light which will suffer no reflection from the lacquer surface. Reflected and scattered light is collected by the mirror and focused onto a peltier cooled, lead selenide detector. The use,

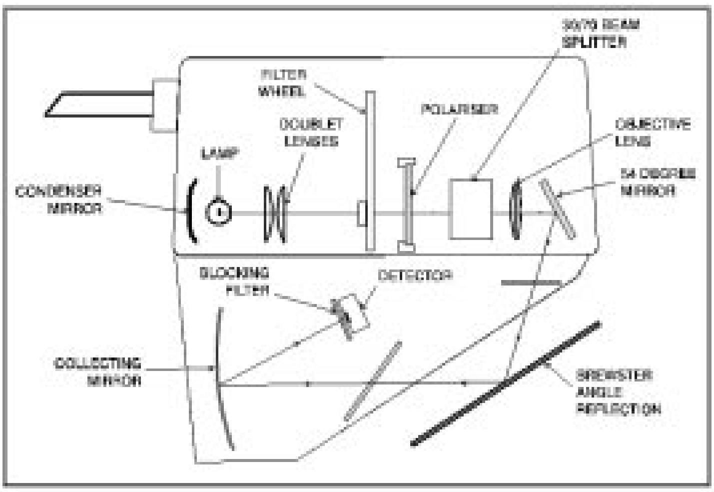

Figure 5. Schematic of the MM55 Lacquer on Foil sensor.

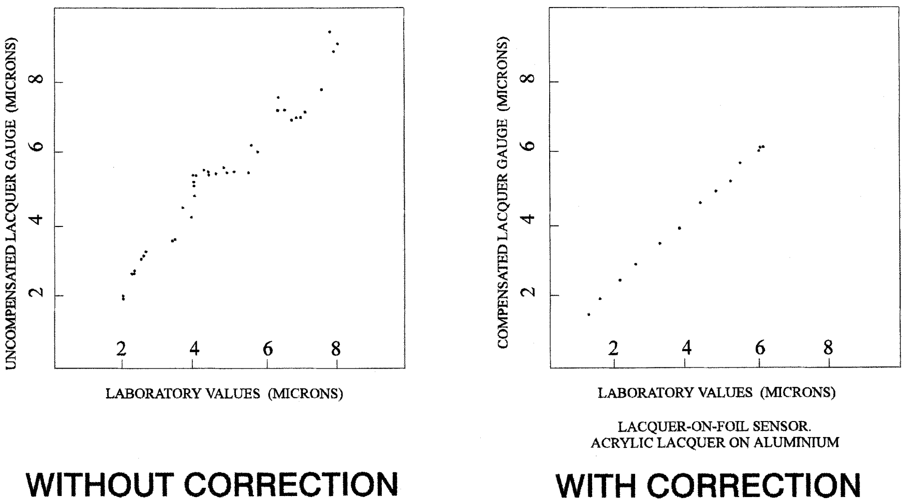

Figure 6(a) and (b). Illustration of the effect of optical interference suppression optics.

or not, of polarisation is dramatic, as shown in the comparative calibration graphs in Figure 6(a) and (b). Performance of the sensor on the wax coating is outstanding as illustrated in Figure 7. The speed of the sensor response allows meaningful cross web information to be achieved and is now in routine use with many aluminium convertors throughout the world.

Alcohol and original gravity in beer

While the use of NIR technology for alcohol and original gravity is recognised, especially using laboratory quality control equipment, its successful implementation on-line has been impaired, principally due to inadequacies of instrument long term stability and the inability to survive the extremely harsh clean-in-place (CIP) conditions commonly used in the industry.

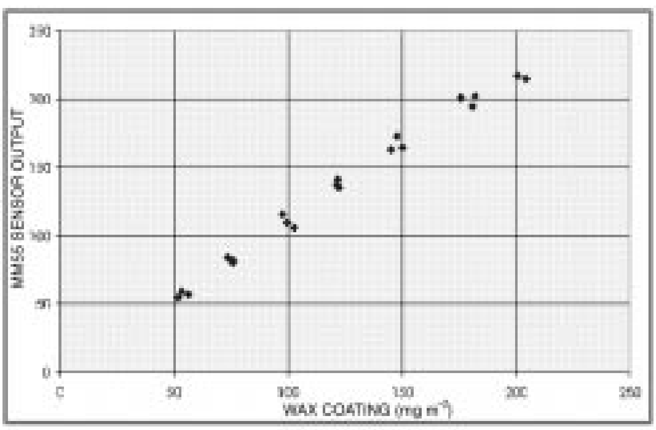

Figure 7. MM55 calibration on wax coating.

The operating environment is quite hostile. The beer passes through the main at pressures up to 10 bar and at around 2°C. The CIP procedure occurs regularly, perhaps daily, and usually involves flushing with hot (60–90°C) caustic soda solution for perhaps 30 minutes to 1 hour. Apart from being able to withstand attack by caustic soda, the probe must be resilient to the regular rapid thermal and pressure shocks. It is also quite normal for equipment to be hosed down during a cleaning cycle and therefore the sensor design must be impervious to water.

Filter based technology has recently been successfully introduced into this application for two requirements:

a) Monitoring alcohol/original gravity (OG) levels in beer to control the dilution with water of high gravity beers to achieve the final sales strength product.

b) As a quality assurance device to check product prior to canning or bottling, thus allowing rapid rejection of out of specification product.

Filter based technology suits both these requirements very well, because it can provide an extremely rapid response with high accuracy, making many measurements per second, each of which is sufficiently noise free to allow reliable process control to be effected.

Measurement of alcohol at 2.27 micrometers and OG at 2.1 micrometers necessitates the use of a 1 mm transmission cell to achieve the level of accuracy required by the industry. It is also clear that product temperature will have an effect on the shape of the water "window" and needs consideration if a temperature independent measurement is to be achieved. An absorbance spectrum of the principal beer components is shown in Figure 8.

The probe employed, known as Liquidata, operates directly in the beer main, either with a retractor device, allowing removal without interrupting production, or directly attached to a flanged sanitary fitting. Filtered light from the instrument is passed along two sets of fibre optics. The primary system focuses light through a sapphire prism with gold coated surfaces which forms the transmission cell. Use of such exotic materials is necessary to ensure complete resistance to caustic soda attack. The return optic focuses the transmitted light onto a lead sulphide detector. The sensor gains the stability needed for long term process control by diverting some of the outgoing light beam along a near identical set of fibre optics; the secondary or reference optics, which have no contact with the beer stream. The light signals returned from the secondary system are detected by a second detector, spectrally and thermally matched to the primary and maintained at a similar temperature. Signals received from both detectors are then ratioed, wavelength by

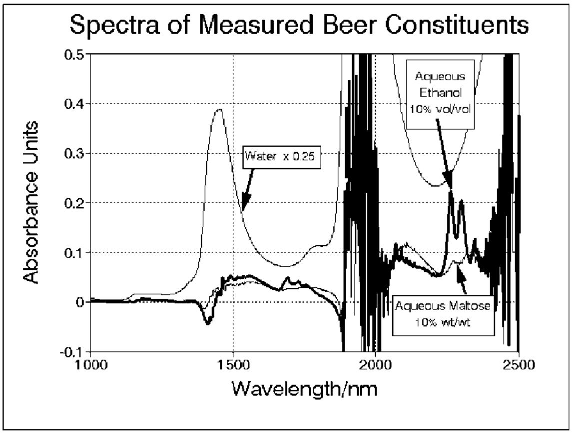

Figure 8. Spectra of measured beer constituents.

wavelength to automatically and continuously compensate for source ageing or filter transmission changes with temperature, which would otherwise cause sensor instability. Subsequent signal processing provides both alcohol and OG or Plato measurement with compensation for product temperature changes. Accuracies achieved in the process environment are respectively better than ±0.03% alcohol and ±0.4 OG units, both 2σ figures. Typically, in the brewing operation many different beer types are processed by a user and so considerable effort has been concentrated on achieving single calibration status for most beer types. Figure 9 shows a typical calibration graph obtained on an abnormally diverse range of beers.

The instrument is built in stainless steel with adequate glanding to protect it from the hostile environment.

Such devices are now installed in many locations with the process in closed loop control, where the signals from Liquidata are allowing the process computer to control blending to extremely high tolerances. Typically a process guarantee on alcohol on any beer will be better than ±0.04% alcohol, 2σ.

On-line measurements of snack foods

The snack foods business is extremely competitive and one of the consequences of this is the need to continually monitor and improve product quality to maximise opportunity in the market place. Product appearance and taste are important and during the manufacturing process both the oil and moisture content provide strong indicators of these quality aspects. Consequently, on-line measurement is vital since historical off-line analysis, even using NIR, is too slow to effect meaningful process control. The point of measurement in a potato chip frying process, most useful to the manufacturer, is also probably the most hostile for an on-line sensor! Typically, installation would be required virtually immediately after frying on the fryer take out conveyor.

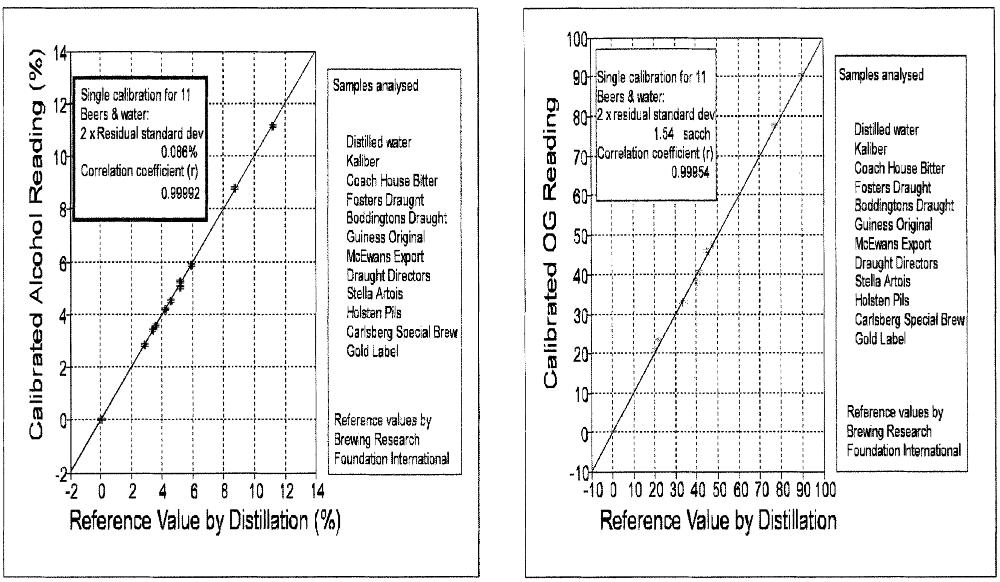

Figure 9. Liquidata calibration graphs for alcohol and OG.

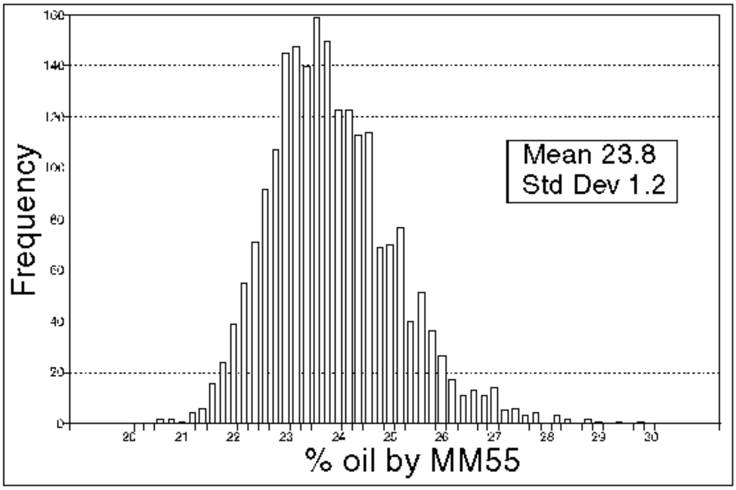

Figure 10. Illustration of the asymmetric distribution of potato chip oil content.

The product is impregnated with hot oil and will be at temperatures in excess of 50°C. Height variations on the conveyor are large and exacerbated by the loosely packed nature of the chips, which would result in the sensor light beam suffering extremely variable penetration through the bed. Volatile oil components will be continually evaporating from the fryer and depositing on cooler surfaces! Ambient temperature conditions can be in excess of 50° C, with high atmospheric humidity.

Widespread success in the application has been achieved with the MM55 backscatter sensor. The sensor is constructed in stainless steel with a sapphire optical window. Optical design of the instrument gives it excellent tolerance to product height variation over ranges of at least 200 mm (8") without loss of performance or impact on signal to noise. The instrument employs the dual detector technology, as previously described, to compensate for ambient temperature variation and long term changes in the source lamp. In the case of the reflectance sensor, the filtered NIR light is sampled by the secondary detector immediately prior to the interaction with the product. This clearly provides excellent compensation for any opto-electronic changes in the sensor, thus ensuring long term stability.

The sensor is maintained at a reasonable operating temperature (<50°C) by the use of Vortec cooling. A sintered stainless steel air purge device maintains a constant stream of air across the sensor window to keep it free from oil build up. Despite the intense environmental difficulties, extremely good results are obtained which allows for closed loop control of the frying operation. While it is necessary to average signal output over periods of seconds, the measurement is very much continuous and the short term noise reflects the true variation in product oil content.

An interesting example of the benefit of an on-line measurement is provided by an analysis of the MM55 sensor output on a fryer. Plotted in histogram format, it shows an asymmetric distribution of oil content over a period of time (Figure 10), with the bias being towards chips having a higher oil content. This can be rationalised with an understanding of the frying process. The shortest residence time for a chip in the fryer will be those instances when the chip is carried through the fryer at the speed of the conveying system, and these will have the lowest oil content. However, it is more likely that due to circulation in the fryer, some chips will remain in for longer

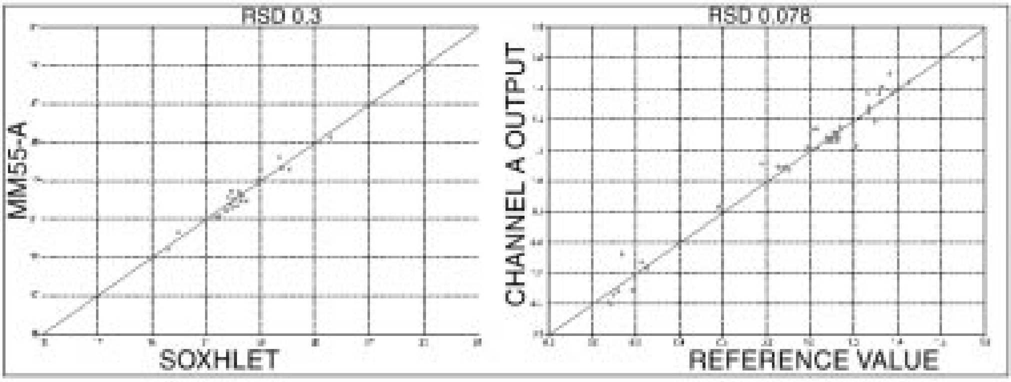

Figure 11. MM55 calibration graph for oil and moisture in potato chips.

periods and will therefore "collect" more oil. It is also, of course, the case that those chips remaining in the fryer for too long, will be low in moisture content and potentially over-cooked causing discoloration. Such intimate knowledge of the process can only be gleaned by a truly continuous fast infrared measurement which then provides the means to improve the production technology.

Calibration graphs for both oil and moisture from on-line data are shown in Figure 11. Many hundreds of these sensors are now employed world-wide to good effect and have contributed significantly to the consistency of the "crunch" in your favourite snack food!

Conclusion

On-line NIR measurement is an already well established technique and has been successfully provided for over the last 25 years by companies who have specialised in industrial instrumentation. There are now thousands of dedicated industrial on-line instruments providing real benefits to users across a broad spectrum of the manufacturing sector.

Moisture measurement has dominated industry needs over the years and continues to be important. However, on-line NIR is increasingly being applied in a staggering range of non-moisture applications, the diversity of which has been illustrated by the above examples. New applications continue to arise and the demand for on-line measurement is ever increasing. It should therefore be reassuring to a potential user that robust and proven on-line instrumentation, highly tolerant to the usually difficult process conditions, already exists, ready to meet these new challenges.