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Applications for portable NIR spectroscopy in food analysis

The role of NIR spectroscopy for food analysis

Near-infrared spectroscopy (NIR) refers to the study of the interaction between matter and light within the electromagnetic spectrum's near-infrared region which ranges from 750 to 2500 nm [1]. When infrared light interacts with a sample's molecules, the amount of electromagnetic energy that is reflected, transmitted, and absorbed for each wavelength depends on the bond types present in the sample [1]. The C-H, N-H, and OH vibration bonds are the most prevalent in the NIR region, determining the spectra shape of a given substance.

NIR spectroscopy is commonly used to measure and quantify a sample's proximate composition, such as protein, moisture, dry matter, fat, and starch. Additionally, the NIR spectrum reflects its physical properties or characteristics [1]. As a result, when applied to food, the NIR spectra of samples can provide information not only about the food's chemical composition but also about its functionality via a non-destructive, rapid, and clean approach that does not require the use of reagents [2].

The impact of portable instruments

Only recently has NIR technology evolved towards miniaturized devices, making it possible to bring NIR analysis from lab to field. Portable near-infrared (NIR) spectroscopy is an excellent tool for monitoring crop quality and determining optimal cultivation conditions and harvesting time. The importance of controlling food quality cannot be overstated given the high vulnerability of foods to content variation, the need to maintain freshness to prevent quality loss, and the possibility of illegal adulteration. Moreover, the complex nature of the food production, delivery chain and the need to reduce analysis time to a minimum has made portable spectrometers a revolutionary step forward in this field [5] [6].

Examples of NIR spectroscopy for food analysis

Appling computational techniques to the absorption spectra obtained with a NIR analyzer, Parastar et al. were able to discriminate fresh from thawed meat as well as correctly classify chicken fillets according to the growth conditions of the chickens with good accuracy [3]. Using similar tools, Kucha and Ngadi were able to assess the freshness of minced pork meat [4].

These computational methods, usually referred to as 'chemometrics' use a variety of algorithms and statistical techniques such as multiple linear regression, partial least squares regression, and principal component analysis to analyze the data coming from the spectrometer. These methods translate the spectral information into chemical and functional properties associated with the sample [2].

Portable NIR analyzers improve cow health, optimize irrigation and harvesting time

Portable NIR analyzers have been utilized for **on-farm monitoring of feed and forages to assess their quality**. In this process, a sample of the feed is placed in front of a scanner that analyzes it, providing results to the farmer or nutritionist. This allows them to make prompt management decisions regarding the feed, significantly reducing the time required for obtaining results from a few days to a few seconds. For instance, the dry matter content of corn silage in cattle feed can vary greatly from day to day, up to 41% over six months. By making adjustments on the spot, cows receive a more consistent ration leading to an improvement in the general health of the herd. This is observed through changes in blood parameters and a reduction in mastitis, resulting in increased milk production. Moreover, this technology can potentially decrease feed wastage, thereby reducing costs and increasing revenue [7].

Another valuable application field for portable NIR spectrometry is the **on-field evaluation of crops in all phases of produce growth**. Tardaguila et al. have studied the absorption wavelength of 160 individual grapevine leaves from eight different varieties grown under various environmental conditions. They specifically targeted water content assessment to identify an optimization strategy for irrigation in the wine industry [8]. During harvest season, NIR spectroscopy has been used to assess the on-tree ripeness of olive fruits [9], grapes [10], and tomatoes [11] enabling **harvest time optimization** and even automated fruit picking using agricultural robots. Following harvest, spectroscopy techniques in the NIR range could be helpful to farmers, consumers, and quality control officers to conduct a **rapid nondestructive examination of produce quality**. This technique also allowed for the detection of pineapple fraud due to mislabeling of conventionally produced fruits as organic ones [12].

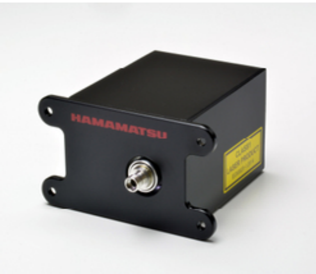
FTIR spectroscopy provides higher throughput and better sensitivity

There are two main methods for **analyzing the absorption spectrum of organic materials** in the NIR spectrum. The first method is **diode-array-based spectroscopy**. This technique uses dispersive grating to separate the light reflected or transmitted from the sample into its wavelength components. Each component is then focused on a different pixel of a linear detector array. This method is considerably fast and can be used for real-time measurements. However, the light throughput of the diode-array spectrometer is inversely proportional to its spectral resolution, which limits its effectiveness. Additionally, the high cost of linear arrays sensitive in the near-infrared region may limit their adoption for certain applications, especially in agriculture and food.

The second method for obtaining an absorption spectrum is **Fourier transform interferometry**. In this method, the incoming light is split into two paths, with one directed toward a fixed mirror and the other toward a movable mirror. When these paths are recombined, an interferogram is obtained.
By performing a Fourier transform of this interferogram, the spectrum of the incoming light can be obtained, and with proper calibration, the absorption spectrum of the sample can be determined. Using this technique, all wavelengths are measured simultaneously, providing **better throughput and higher sensitivity without compromising spectral resolution** (usually referred to as 'Fellgett's advantage'). In this technique, only a single NIR photodetector is used instead of an array, keeping the cost low.

Hamamatsu Photonics' FTIR Engine brings new light to the food industry

Hamamatsu's FTIR engine C15511-01 is a **compact Fourier transform infrared spectroscopic module** with sensitivity to near-infrared light in the range of 1.1 µm to 2.5 µm and USB connectivity. This device features a Michelson optical interferometer and control circuit in a palm-sized housing.



In order to compensate for the light loss due to components miniaturization, engineers at Hamamatsu Photonics have equipped the FTIR engine with a large movable MEMS mirror and a highly sensitive InGaAs PIN photodiode. The special design of this MEMS component neutralizes the influence of external vibration and stray light reflections inside the device. The position of the movable MEMS mirror is continuously and accurately monitored using a dedicated laser system to guarantee the highest wavelength reproducibility.

Generally, Hamamatsu's FTIR Engine delivers **high-sensitivity, high-resolution, and high-speed measurements** comparable to larger and pricey benchtop devices.

There are two measurement methods for infrared spectroscopic analysis using FTIR engines: **"reflection measurement"** and **"transmission measurement"**. Using these methods, we measured the spectra of nuts (almonds, cashews, walnuts) and alcoholic beverages (beer, sake, and brandy).

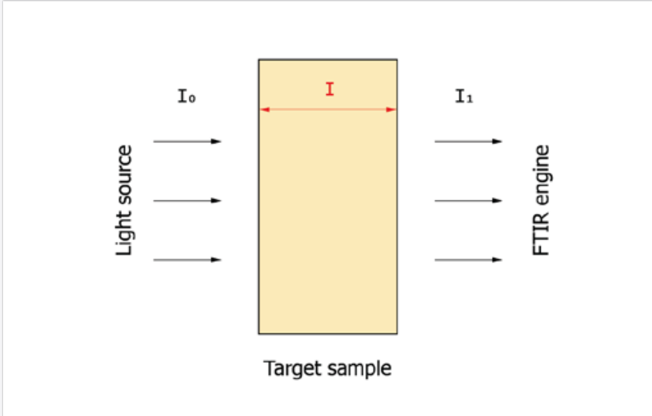
Transmission measurement: comparison of the absorbance spectra of alcoholic beverages and estimation of their alcohol concentration

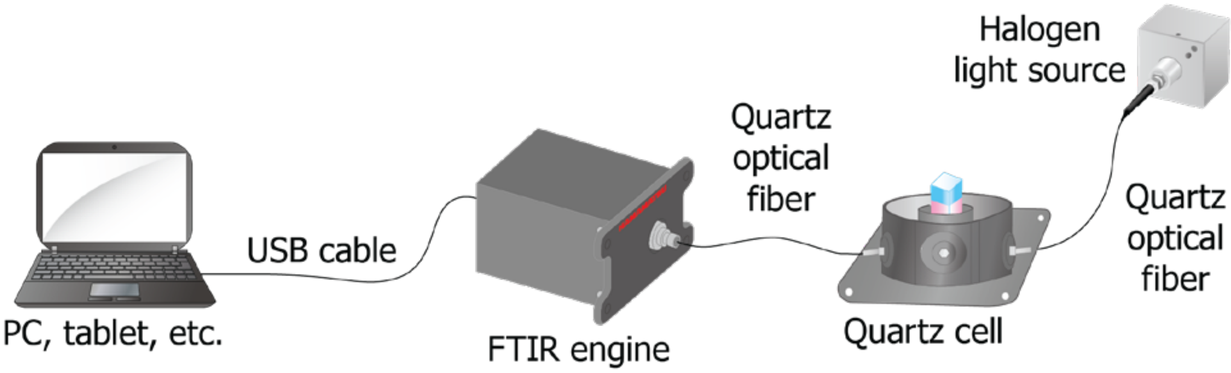
The FTIR Engine C15511-01 was used to observe the differences in the absorption spectra produced by several alcoholic beverages. The liquids were placed into a quartz cell transparent to the near-infrared offering a light path length of 1 mm. A halogen lamp was used as a light source for this experiment. The broadband light coming from the lamp is partially absorbed by the liquid and partially transmitted to the FTIR Engine through optical fibers. The absorption spectra shown in the figure were obtained at room temperature, averaging 128 scans, and subtracting a reference measurement. The shape of these spectra is mainly influenced by the OH-groups in water (absorption wavelengths: 1450 nm and 1900 nm) and the CH-groups in alcohols (absorption wavelengths between 2100 nm and 2500 nm). The spectra of pure water and ethanol were also measured and added to the graph for comparison.

■ Equation of absorbance

$$A = -\log_{10}\left(\frac{I_1}{I_0}\right) = cI\varepsilon$$

A : absorbance
c : sample concentration
I : optical path length of quartz cell
I_1 : transmitted light level
I_0 : incident light level
ε : molar absorption

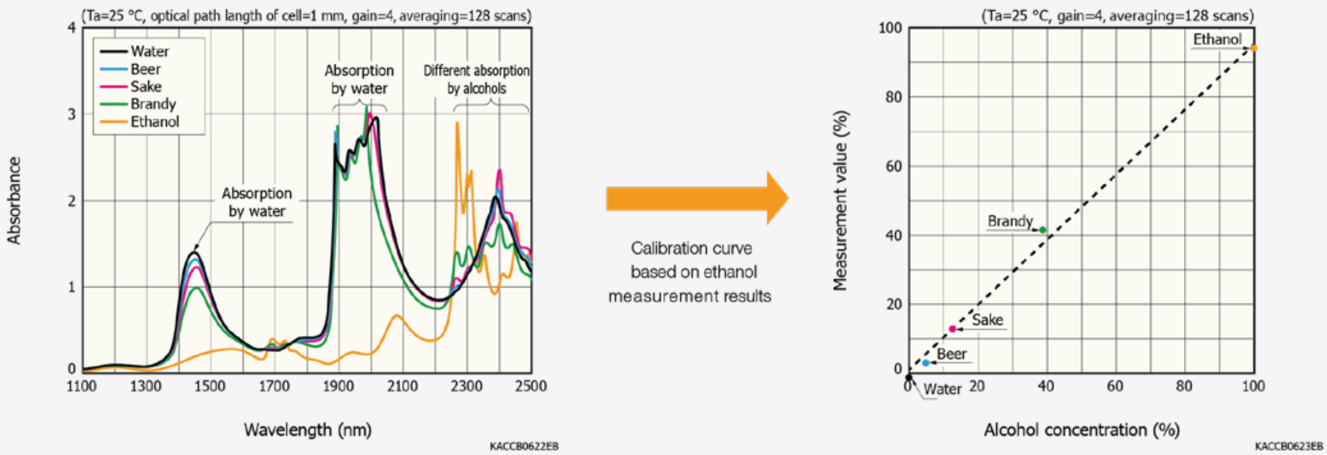




Additionally, absorption peaks at 2300 nm were used to estimate the alcohol concentration in each of the beverages. This measurement showed values in accordance with the actual presence of alcohol in the liquids, confirming how accurate estimation is possible using this compact device and method.

■ Absorption spectra of alcohols (measurement example)

■ Alcohol concentrations (measurement example of 2300 nm band)



Diffuse reflection measurement:
nuts classification using near-infrared spectroscopy

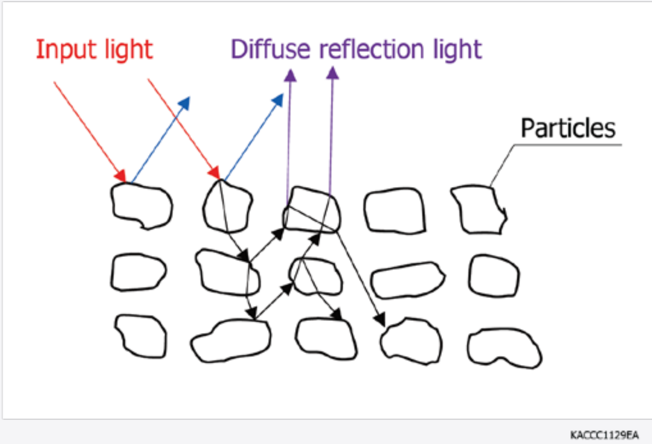
While the portion of light irradiated onto a sample is regularly reflected by its superficial particles, the rest penetrates the sample. Here, the light is repeatedly diffused through refractive transmission, light scattering, and surface reflection until it makes it out of the sample to be measured. The diffuse reflection spectrum obtained through this measurement is similar to the absorption spectrum of the sample.

■ Equation of absorbance

$$\frac{K}{S} = \frac{(1-R)^2}{2R} = \cosh[\log_{10}(1/R)]-1= \log_{10}\left(\frac{I_1}{I_0}\right)$$

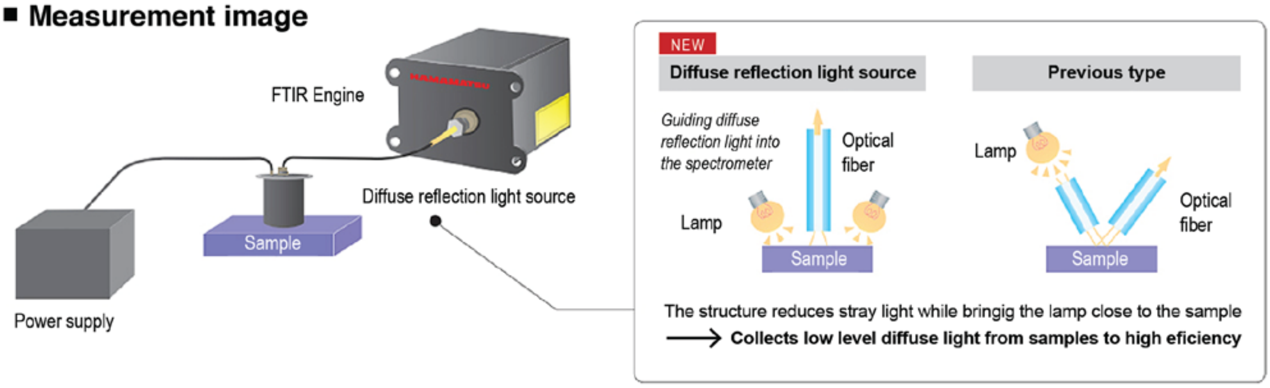
K/S: Kubelka-Munk
S : scattering coefficient
K : absorption coefficient

R : Reflectance $= I_1/I_0$
I_1 : transmitted light level
I_0 : incident light level



Diffuse reflection signals are generally weaker than those obtained through transmission. For this reason, one of the main challenges when using this method is improving illumination efficiency. In a traditional configuration, the broadband light from a single halogen lamp is directed to the sample using an optical fiber. Hamamatsu Photonics has recently designed L16462-01, an innovative **light source optimized for diffuse reflection measurements**. This device is equipped with multiple lamps in close proximity to the sample at a specific angle. The light diffused from the sample is collected by an optical fiber and directed to the NIR spectrometer. This configuration greatly **improves the measurement signal-to-noise ratio** minimizing the influence of stray light.

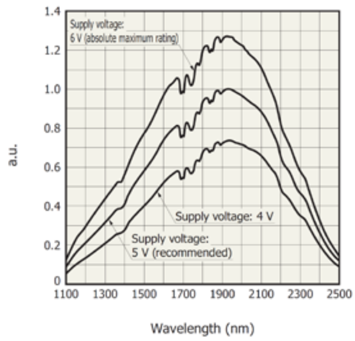
■ Measurement image

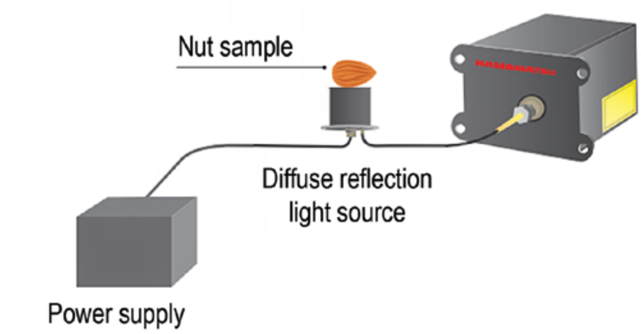


Food allergy is a condition where genetically susceptible individuals experience an unfavorable immune response after consuming certain food components. This reaction can result in immediate or delayed symptoms, which can be severe or fatal [13]. This immunological disorder has emerged as a significant worldwide concern over the last few decades, affecting at least 8% of children and 5% of adults in Western nations. It places a considerable strain on the healthcare system and can severely restrict daily activities [14]. Many kinds of nuts, including walnuts (Juglans regia), cashews (Anacardium occidentale), and almonds (Prunus dulcis) are listed as allergens by the European Regulation 1168/2011 and need to be added to the ingredients list whenever present in food [15]. For these reasons, the detection and classification of nuts are a necessity for the food industry.

Hamamatsu used NIR spectroscopy to study and classify the absorption spectra of almonds, cashews, and walnuts. The measurements were obtained using FTIR Engine C15511-01 and the new lamp L16462-01. The nuts were placed over the light source without any preliminary preparation and an average of 128 scans was performed to obtain the absorption spectrum of each sample.

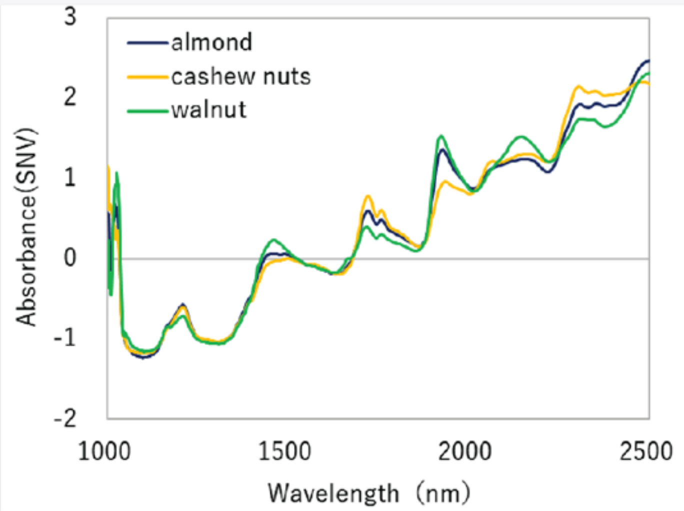
■ Lamp L16462-01 spectrum when used with FTIR engine C15511-01 (typical example)

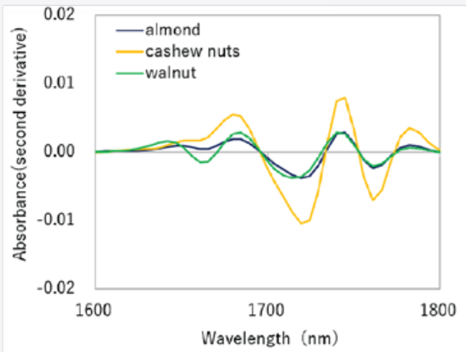


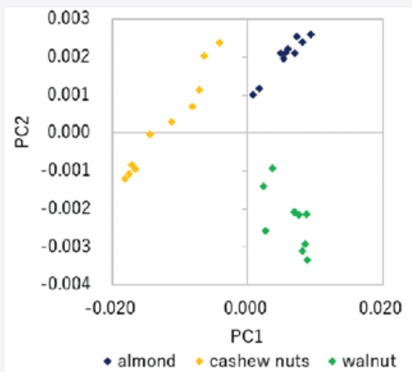


The obtained spectra were characterized by peaks at 1600-1800nm, caused by the first overtone of CH stretching from lipids and protein. The differences among the various spectra were more evident when observing the second derivative of the spectra. Classification of the different kinds of nuts was possible through the principal component analysis method.

■ Almonds, cashews, and walnuts absorption spectra measurements using FTIR Engine C15511-01 and lamp L16462-01







Conclusion

The potential applications of NIR infrared spectroscopy in the food industry have been widely documented by numerous scientific publications for several years. The advent of portable instruments is now moving the analysis from lab to field reducing dramatically the time for results from days to seconds. Most notably this hardware miniaturization driven by Hamamatsu's MEMS technology happens without compromising on sensitivity or resolution.

New computational techniques are continuously being developed to analyze and compare the absorption spectra and estimate the content of specific chemical compounds in food. These methods are making the technology more and more accessible to non-technical users across the industry.

Portable FTIR analyzers can be a valuable tool to address many vital challenges in the food industry. For example, they can help improve crop yield thus providing an alternative to deforestation when facing an increased food demand. Integrating these technologies into farming can limit water waste when optimizing irrigation and limiting food waste across the supply chain. Finally, FTIR analyzers can contribute to the improvement of our food quality making it safer and healthier for us and for all the animals depending on us.

Contact us to test our FTIR Engine or visit our product page to learn more about this technology, its applications, and all the other spectroscopy solutions offered by Hamamatsu Photonics.

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