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How to analyze food and future requirements for NIR spectroscopy

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Abstract Food has been subject to various characterization methods for centuries. Optical characterization methods like spectroscopy were implemented long ago. Different requirements arise from integrity (contaminations, foreign objects, spoilage) and quality (composition) issues. In our modern world, automated optical tools are being used for several tasks, in particular for testing raw materials and for in-line monitoring of food processing. In the future new options for optical food analysis and inspection will arise. On site testing from “field to fork” drives the development of mobile analysis units for harvesting, transportation, storage and distribution, which will enable a more detailed control during processing. In addition, the consumer is interested in an on-site analysis using household or mobile devices. Here, market opportunities for hardware integrators, software engineering and data service providers are identified.

Keywords: Food analysis, NIR spectroscopy, product development.

1 Introduction

The most common method of analyzing food is organoleptic testing. A person is testing food without any technical means by assessing food with the human organs of perception. Thus, food is characterized by its taste, odor, appearance, color and texture. However, this method is subjective and highly related to the tester’s practical experience and capabilities. In the worst-case this method might be hazardous to the tester's health or even lead to death. Therefore, objective and quantitative methods for analyzing food are in great demand. Various approaches have led to the development of electronic noses [1] and electronic tongues [2], but both still have a far way to go to mimic the human nose and tongue.

Food condition is affected by the complex chemical composition of the food and can be addressed by optical characterization methods like spectroscopy. Especially optical vibrational spectroscopy has the potential for a rapid quantitative detection of food quality and food fraud both in the laboratory and in mobile applications along the food supply chain [3]. Two aspects are generally considered: First, microbiological and toxicological properties must be granted by the producer and distributor. Especially food spoilage due to microbes [4] and other biological [5] and chemical hazards [6] throughout the food chain are the most significant threats to food security within Europe. Here, sophisticated methods are required to meet the very low detection limits. Second, quality issues [7] such as ripeness, freshness and nutritional facts (i.e. sugar, carbohydrate, protein, fat and water) will differ and may change between processing and consumption. A favorable option to evaluate food composition is near infrared spectroscopy (NIR). Technically, the spectral range from 1000 to 2300 nm is most interesting. Relevant data is usually available in the 1100 to 1800 nm range, which can be addressed using standard sensors without compulsory detector cooling.

With the knowledge about suitable optical methods for food quality determination and their potential to go into the field it becomes important to draw a road map for the market entry. Based on the roadmap goals and tasks can be derived for the next steps in development. This concerns both hardware, software and data base. Thereby and by offering suitable solutions the application will be opened not only to dedicated food specialists but for also for common use.

2 How to analyze food

2.1 Basics

Based on a suitable sample preparation method, there is a large variety of analytical techniques available today. These range from biological (e.g. PCR, immunology) to separation (e.g. liquid or gas chromatography), spectroscopic (e.g. mass spectrometry, fluorescence, near-/mid-infrared, NMR), rheological (e.g. viscometry), thermal (e.g. DSC), radiochemical (e.g. isotopic) and electrochemical techniques (e.g. voltammetry) [A. Cifuentes 2012]. Spectroscopic techniques are based on energy selective interaction between electromagnetic radiation and the sample (or in the case of mass spectrometry on energetic filtering of sample constituents). Electromagnetic radiation in the infrared region (λ = 0.78 μm – 100 μm) interacts with molecules and leads to an energetic excitation of the molecules depending on the specific molecular structure. In the near-infrared (NIR, λ = 0.78 – 3.0 μm) and mid-infrared (MIR, λ = 3.0 – 30 μm) region this interaction leads to vibrational and rotational excitations of the molecules. "Nowadays, spectroscopic techniques based on infrared region are one of the most numerous in the food analysis. Thus, infrared spectroscopy is frequently used for quality control of food including analysis of honey ... or muscle food ...". [8]. NIR spectroscopy can contribute valuable information to food quality analysis. However, contaminations at low concentrations (ppm and below) must be detected via more sophisticated methods, which might be too expensive for widespread consumer use.

Different system approaches have been presented for NIR spectral analysis: Classical spectrometers offer superb performance but are bulky, expensive and sensitive towards environmental conditions. Diode array spectrometers with reduced size reveal favorable performance but still the cost issue limits their applications within NIR-based food sensing systems. Low cost devices have been realized using MEMS technologies. Filter based systems (e.g. Spectral Engines, Hamamatsu), digital light processors (e.g. TellSpec), scanning grating (Hiperscan) are some examples for miniaturized systems. However, spectral analysis means more than just applying a spectrometer. The evaluation of NIR spectra requires chemometric modelling based on reference data. In addition, database access is indispensable when ap-

Table 8.1: Hardware parameters of different NIR spectrometer devices.

Device	Size	Expected target price
Lab spectrometer	(30 x 25 x 15) cm^3 = 11 250 cm^3	
Hiperscan SGS 1900	(10.5 x 8.0 x 8.6) cm^3 = 722 cm^3	
Ocean Optics flame NIR	(8.9 x 6.3 x 3.2) cm^3 = 179 cm^3	
TellSpec (DLP based)	(9.0 x 6.0 x 2.5) cm^3 = 135 cm^3	
Next-gen MEMS spectrometer	(5.0 x 5.0 x 3.0) cm^3 = 75 cm^3	500 € @ 10 000 p.a.
IPMS SCS	(1.5 x 1.4 x 1.0) cm^3 = 2.1 cm^3	100 € @ 1 mio p.a.
Mobile phone (tbd)	(1.2 x 1.1 x 0.6) cm^3 = 0.8 cm^3	5 € @ very high volume

plying this method in the field. Therefore, progress in this area must coincide with hardware development.

For a future platform, the following components and requirements in addition to the spectrometer unit itself have to be taken into account: suitable light source(s), appropriate optical coupling with minimal losses, an integrated computational device for data acquisition and post-processing, data base access, internal memory or online data communication. Options could be systems mounted to machines as well as portable devices or even integration into smart phone or tablet. In the latter case size issues become relevant (see Tab. 8.1). The development effort for the next generation MEMS spectrometer is expected to be in the range of 0.5 Million Euros, the integration of SCS including ASIC design is expected at 3 million Euros, the development cost for spectrometer integration into a mobile phone needs to be specified yet.

2.2 Miniaturized spectrometer suitable for food analysis

Much work has been done to miniaturize NIR spectrometers in the past years. In particular, obtaining the relevant spectral range (λ = 950 - 1900 nm) with a suitable spectral resolution (better than 10 nm) while keeping the signal-to-noise ratio (SNR) sufficient (3.5 a.u. corresponding to 12 bit data depth) is a challenging task (here a.u. denotes absorbance unit, which is used in practice – however the ratio between incoming and transmitted radiation is dimensionless).

An example of how small an NIR spectrometer can be realized was demonstrated by Fraunhofer IPMS (see Fig. 8.1) with the stacked-

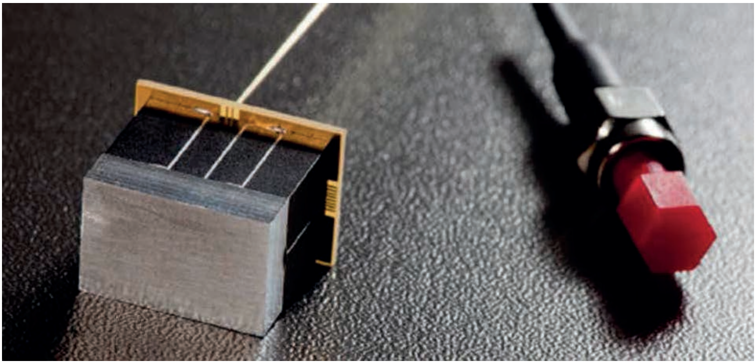

Figure 8.1: Stacked-component-spectrometer (SCS) by IPMS.

component spectrometer (SCS) in the size of a sugar cube using a scanning-grating MEMS chip [9].

The SCS is realized by stacking two key components: the MEMS/detector part and the spacer/mirror part. The basic idea behind this concept is to connect necessary spectrometer substrates (including micromechanical and optical functional elements) in a stacked manner. This enables a convoluted light path between both substrates [10].

Compared to the much larger commercially used system Hiperscan SGS1900, the IPMS SCS performs alike (Fig. 8.2 left) and exhibits a spectral resolution of less than 10 nm (Fig. 8.2 right) [9].

2.3 Food analysis using scanning grating technology

Application related measurements have been performed on selected use cases. One of these use cases is milk, representing a prominent example of food adulteration, especially in the Asian region. The scanning grating technology of IPMS has been tested for this application and measurement results are shown in Fig. 8.3, clearly giving access to fat content monitoring.

Further milk samples were deliberately spoiled with a variety of common adulterants in the Asian region (e.g. fructose, glucose, vegetable oil and detergent). First chemometrical models were developed in cooperation with Fraunhofer IOSB. The results of a principal com-

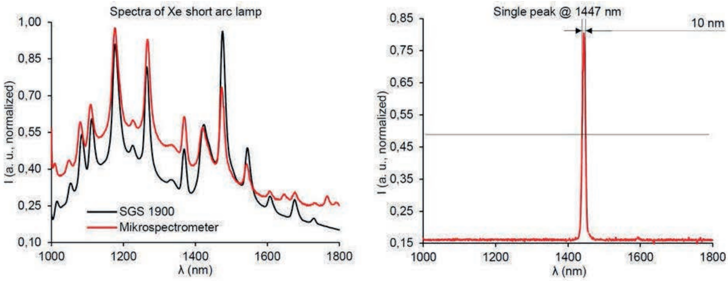

Figure 8.2: Performance comparison of IPMS SCS (microspectrometer) with Hiperscan SGS1900 (left) and spectral resolution (< 10nm) of the IPMS SCS (right).

ponents analysis (PCA) for the case of milk adulteration are shown in Fig. 8.4.

Common adulterants of milk measured with scanning-grating based NIR spectroscopy can be discriminated after PCA analysis.

3 Future requirements for NIR food analysis

3.1 Existing and future requirements

Several systems for food analysis have already been presented for selected applications (e.g. by Ocean Optics, TellSpec, Consumer Physics and others). However, to meet the customer demand in different application fields further research and development is still required. For example, the available spectral range must coincide with the relevant optical bands, especially for H2O (λ=1450 nm/1960 nm), C-C, C-H, C-O (1680 – 1820 nm). Whereas size, weight and usability are fair for systems on the market, price is still a big issue. Before considering development goals, the specific requirements of some use cases must be taken into account. Four different market segments with differing market entry conditions are discussed:

In line metrology – mounted devices

State of the art spectrometers are in use in this field, e.g. in harvesting control and meat processing. Further applications may be addressed

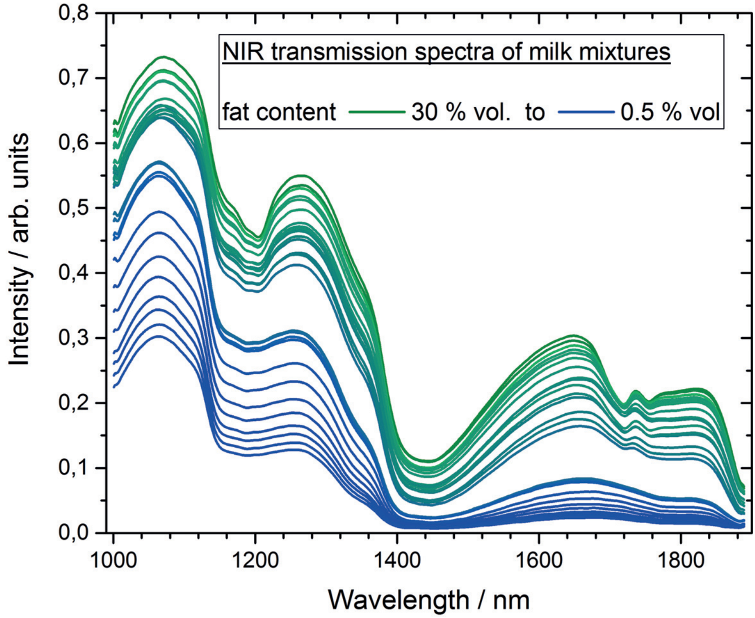

Figure 8.3: NIR spectra (acquired with scanning grating technology in diffuse reflectance mode) of milk mixtures with fat content in the range from 0.5–30 % vol.

with decreasing price and reasonable total cost of ownership. Besides, sterilization and cleaning in process must be taken into account.

Handheld analyzers – expert systems

A suitable system integration including light source, data interface and power management is required. Rough environmental conditions must be considered. Developments in this market area are already in progress.

Integrated devices – consumer use

Spectral analyzers may be integrated e.g. into scales at the point of sales or into kitchen equipment like mixers or refrigerators. A major issue beside the data base will be the proper measurement of the relevant data. Here user guidance for inhomogeneous objects combined with intelligent software may help for proper measurement quality.

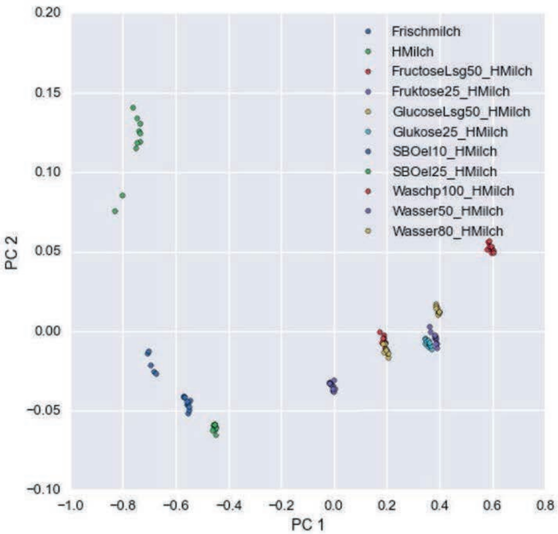

Figure 8.4: PCA of adulterated milk samples, FhG-report: MEF-SensoSpek 2015, in cooperation with H. Schulte (Fraunhofer IOSB).

Mobile spectrometers – everybody
An NIR spectral analyzer in a mobile phone or similar device may serve more applications than just food. Also here a dedicated software app must ensure proper use.

Concerning the food market optical NIR-spectroscopy has a huge potential as smart monitoring technology in the whole food value chain (see Fig. 8.4).

Certain requirements arise for appropriate NIR spectroscopy systems for a potential field of application within the food value chain, which are summarized in Tab. 8.2.

3.2 Opportunities for spectrometer development

Starting from existing systems and devices and based on multiple discussions with potential customers from different market segments, it can be derived that a growing market for NIR food analyzers exists.

Figure 8.5: Food value chain and potential fields of application for optical NIR-spectroscopy as smart monitoring technology.

In-line monitoring is driven by increasing requirements due to legislative changes [11]. There is a growing demand to access food quality, e.g. freshness and ripeness of fruit, at the Point-of-Sales. Finally, several mobile phone manufactures and their optical system integrators have signaled interest in NIR spectroscopy but the cost requirements are tough to meet. Within the next years business opportunities arise for those who provide systems within an acceptable cost and price range.

3.3 Development goals in the field of MEMS based solutions

Based on feedback from the agricultural and mobile phone market first steps have been implemented for two new MEMS based approaches. The required specifications of below approaches are the result of discussions with agricultural machinery manufacturers and suppliers in the US and Europe.

a) The next generation MEMS spectrometer is aiming at the handheld market. The outer dimensions should not exceed 50 x 50 x 30 mm^3.

Table 8.2: Requirements for NIR spectroscopy within the food value chain.

Requirements for NIR system	Style	Size [cm³]	Sensitivity [a.u.]	Environmental conditions	Estimated Cost [€]
Field (soil, water, fertilizer, manure)	tractor mounted	9000	4.5-5.0	harsh (rough handling, vibration, temperature, dirt)	5000
Plant/crop	multiple (e.g. UAV)	75	3.5	light weight	100
Harvest	tractor mounted	800	4.0	harsh	500
Animal	handheld or wearable	75	3.5	corrosive, dirt	100
Processing	in-line	800	4.0	sterilization compatible	500
Storage	handheld or in-line	75	3.5	low temperature	500
Distribution	handheld and integrated (e.g. scales)	75	3.5	moderate	50
Consumer	mobile phone, refrigerator, kitchenware (e.g. mixer)	< 2	3.0	low cost	5

The target performance will be similar to the commercial available SGS 1900 system (, i.e. λ = 950 .. 1900 nm, 10 nm spectral resolution and 3.5 a.u. information depth) but with reduced outer dimensions.

b) An extremely miniaturized MEMS spectrometer for the potential use in mobile phones could have a very flat layout with 15 x 10 x 6 mm^3, possibly even slightly thinner. If necessary and affordable, the spectral range can be 1000–1900 nm with 10 nm resolution and at least 3 a.u. performance. However, reduced parameters will possibly be sufficient and less demanding.

3.4 Other options

Besides MEMS based spectrometers, there are other options for NIR spectral analysis. Diode array spectrometers with a fixed grating were the first step to miniaturization. Here, expensive detector materials, e.g. extended InGaAs, have limited the cost reduction. Assuming 10 nm spectral resolution and a minimum of 3 detector elements for this wavelength interval, the 900 – 1000 nm range corresponds to 256 or 512 ele-

Table 8.3: Requirements and development goals for different market tiers.

Market tier	Requirement / Development
High-end for harsh environment	Full 16 bit performance needed corresponding to 4.5 … 5 a.u., tough shock survival, size less critical Currently: diode array spectrometers (e.g. Zeiss Corona or Foss) Estimated cost reduction from 15-30 k€ to 1 k€ in 5-8 years
In-line process control device	14 bit performance corresponding to 4 a.u., Currently: either diode array spectrometer (see above & e.g. Avantes, OceanOptics) or MEMS based system (Hiperscan) Size reduction is preferred, but cost has to reach level below 500 € within the next 3-5 years
Handheld / integrated PoS System	12 bit performance, i.e. 3.5 a.u. Currently: for example "Phazir" handheld from Thermofisher Size target is below 75 cm^3, price target below 100 € in 3-5 years with estimated further decrease to 50 € region
Consumer	New development Price below 5 €, size smaller 2 cm^3, thickness requirement for mobile phone is below 6 mm Performance and time to market are to be defined Huge investments necessary in technology development, integration, …

ments in total. Today a typical width of 50 μm is used for each element to grant good SNR properties. This leads to a width of 12 – 25 mm of the sensitive area, which must be granted by the systems optical design. Lower requirements and performance may only demand 5 nm interval per pixel, 25 μm element width and 128 elements for 600 nm free spectral range. Thus the sensitive area could be shrunk to 3.2 mm. Still the question of prices for high volume production remains a challenge.

Cost may be reduced by applying single detector and a digital light processor (DLP). Although a DLP might be too large for mobile phone applications, the approach is suitable for handheld and in-line devices.

Interferometer based systems such as Fabry-Perot filters have been presented (e.g. Spectral Engines, Hamamatsu). These have a small form factor and reasonable price but still insufficient free spectral range and possibly limitations to vibration and shock in mobile and handheld systems. Filter based solutions with individual filters on each element of a detector array share the same cost issue like fixed grating spectrometers.

3.5 Recent developments

The scanning grating technology implemented at Fraunhofer IPMS is facing new developments heading for compact and ultra-small optical setups. The key to a cost efficient assembly is the alignment and reduction of the optical components cost. The actual hybrid mounted "stacked component" spectrometer SCS prototype requires complex off axis mirrors for the folded optical path. As of today, these mirrors have been fabricated by ultra-precision technologies from aluminum. Plastic or glass molding technologies do not meet the optical requirements today. Here, a joint development of optical layout and mirror technology might lead to a cost efficient way to build this device in high volume.

4 Summary and Outlook

Food safety and food quality are important topics for the individual consumer and to our society in general. Thus analytical methods for food analysis are of growing importance driven both by environmental and legislative requirements. In this context fast and non-invasive techniques are in favour. These requirements are fulfilled by optical methods. Vibrational spectroscopy is a strong tool for analysis of organic materials and especially near-infrared (NIR) spectroscopy is a suitable and promising candidate. It has been shown that miniaturized NIR spectrometers can be realized using MEMS technology. Several systems designs are presented and optimization required to match with size and cost requirements has been discussed. To reach deployment in challenging environmental conditions and in larger volume there is still development investment necessary. Ultimately, for a broad implementation of food analyzing systems mobile phones offer a very promising platform. However, the application software must display the relevant data in a way that is applicable for the non-scientific user. A joined effort of hardware, software and database providers may open the door for widespread usage of MEMS-based NIR food analyzers and will lead to new market opportunities.

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