AVANTES
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APPLICATION NOTE

MEASURE MOISTURE CONTENT IN GRANULATES WITH NIR SPECTROSCOPY

EXECUTIVE SUMMARY

Near-infrared (NIR) spectroscopy is a highly valuable and widely utilized technique for real-time moisture content measurement in various substances. Its non-destructive, rapid, and precise results have made it indispensable across diverse industries such as Pharmaceutics, Food, Chemistry, Agriculture, and Construction.

It is important to emphasize that the calibration model for moisture measurements using NIR spectroscopy is material-specific and critical to ensure accuracy and reliability of moisture predictions. Therefore, distinct calibration models are required for different types of granulates or sample matrices. For instance, the measurement of moisture content in silica granulates used in the rubber industry serves as an illustrative example in this application note.

For cases where moisture bands are clearly present and above a few percent, the recommended setup includes the Avaspec-NIR256-1.7-EVO in combination with the Avalight-Hal-s-Mini2 and FCR-7UVIR400-2-BX fiber. On the other hand, when dealing with low concentrations of moisture, challenging sample discrimination, or the presence of multiple components, the suggested setup involves using the Avaspec-NIR256-2.5-HSC-EVO with the same accessories.

Once a reliable chemometric model is established and the actual moisture is determined, it becomes feasible to correlate different moisture levels with water bands. By leveraging the Avantes NIR spectrometer alongside the chemometric model, an efficient, non-destructive, and accurate moisture detection system can be achieved.

INTRODUCTION

Near-infrared (NIR) spectroscopy is a well-recognized technique used for real-time in-line measurement of moisture content in various substances including silica particles. The technique is effective due to its ability to perform non-destructive measurements, capture extensive data and operate in real time, providing swift and accurate results[1]. It is used in many industrial applications:

Pharmaceutical Industry: NIR spectroscopy is widely employed to determine the moisture content of granulates used in tablet manufacturing. By analyzing the NIR absorption spectrum of the granulates, the moisture content can be quantified accurately. This information is crucial to ensure a safe and consistent production of medicines.

Food Industry: In the food processing industry, NIR spectroscopy is used for moisture analysis of granulated food products such as grains, cereals, and powdered ingredients. By measuring the absorption of NIR light by the granulates, the moisture content can be determined rapidly and non-destructively. This helps in quality control and optimizing the production process.

Chemical Industry: NIR spectroscopy is also utilized in the chemical industry to monitor and control the moisture content of granular materials. It allows for real-time measurement of moisture levels during the manufacturing process, ensuring consistent product quality, preventing issues caused by excessive moisture, and the safety of employees during a chemical process.

Agricultural Applications: NIR spectroscopy finds applications in agriculture for monitoring the moisture content of granular materials like fertilizers, soil samples, and animal feed. By analyzing the NIR spectra, farmers and researchers can assess the quality and effectiveness of these granulates, making informed decisions about their usage. It is possible to determine the moisture concentration during the harvest of crops. This, in combination with other parameters, determines the quality of the product before the crops are transported off the field.

Construction Industry: NIR spectroscopy is employed in the construction industry to measure the moisture content of granular materials like sand, gravel, and concrete aggregates, but also the moisture in walls, paint and wood. This information is crucial for achieving the desired strength and durability of construction materials and ensuring compliance with quality standards.

To measure moisture content in granulates using NIR spectroscopy, the following steps are typically followed:

1. Calibration: A calibration model is developed by collecting a set of reference samples with known moisture content. These samples should cover a wide range of moisture levels to establish a reliable calibration curve. The reference samples are analyzed using a laboratory method, such as Karl Fischer titration or loss on drying, to determine their actual moisture content.

2. NIR Measurement: The NIR spectrometer is used to measure the near-infrared reflectance, transmittance, and/or absorbance of the samples. The halogen light source emits a beam of near-infrared light onto the sample, and the reflected or transmitted light is collected and analyzed. The NIR spectra obtained contain information about the molecular vibrations and characteristics of the sample, including moisture content.

3. Data Analysis: The NIR spectra obtained are preprocessed using the first derivative in conjunction with center scaling to remove baseline drift and other effects, such as particle size and temperature. Additionally, the data are processed using chemometric techniques, such as multivariate analysis or regression algorithms. The calibration model developed in the first step is applied to the measured NIR spectra to predict the moisture content of the samples.

4. Validation: To ensure the accuracy and reliability of the moisture predictions, the calibration model needs to be validated using independent samples with known moisture content. These validation samples should be separate from the calibration set and cover a similar range of moisture levels. The predicted moisture values obtained from the NIR measurements are compared to the actual moisture values determined by a reference method.

5. Prediction: Once the calibration model is validated, it can be used to predict the moisture content of unknown samples. The NIR spectrometer measures the NIR spectra of the samples, and the calibration model is applied to calculate the moisture content based on the spectral information.

It is important to note that the calibration model for moisture measurement using NIR spectroscopy is specific to the material being analyzed. Therefore, a separate calibration model is required for different types of granulates or different sample matrices. Additionally, it is crucial to establish and validate robust calibration models to ensure accurate and reliable moisture predictions using NIR spectroscopy.

EXAMPLE ON SILICA GRANULATES FOR THE RUBBER INDUSTRY

To highlight the feasibility of NIR spectroscopy to measure moisture content, different silica powders were analyzed.

Silica is commonly used as a reinforcing filler in rubber compounding due to its unique properties. When added to rubber compounds, silica can enhance mechanical properties, such as tensile strength, tear resistance, abrasion resistance, and stiffness. It also improves the dynamic properties, such as the rebound resilience and hysteresis of the rubber. This is achieved via a silanization reaction which creates a strong linkage between the silica granulates and the rubber network.

Silica is a hygroscopic material, meaning it has a strong affinity for moisture absorption from the environment. Since the silanization reaction is strongly influenced by the amount of water in the silica granulates, variations in the moisture level can significantly affect its performance as a reinforcing filler in rubber compounding.

Too little moisture can result in a slow or incomplete reaction. In such cases, the silane molecules may not hydrolyze efficiently, leading to limited availability of reactive silanol groups. Consequently, the attachment of silane molecules to the silica surface will be hindered, resulting in a lower degree of functionalization or bonding[2].

On the other hand, excessive moisture can also have detrimental effects.
High moisture levels can lead to uncontrolled hydrolysis of silane molecules, causing premature condensation reactions. This premature condensation can result in the formation of siloxane bonds between silane molecules before they have a chance to attach to the silica surface. As a result, the availability of silane molecules for bonding to the silica surface decreases, leading to reduced silanization efficiency[2].

Therefore, controlling the moisture level is crucial for optimizing the silanization reaction.
The ideal moisture level depends on the specific silane and silica system, as well as the desired degree of functionalization. It is often necessary to carefully balance the moisture content to ensure sufficient hydrolysis and reactivity while avoiding excessive condensation reactions.

MATERIALS USED

Spectrometer 1:

Specification	Description
AvaSpec	NIR256-1.7-EVO
AvaBench	USB3 - EVO-RS - 50mm
Grating	NIR200 - 1.5 (A) - 200 lines/mm
Range	900 - 1700 nm

Spectrometer 2:

Specification	Description
AvaSpec	NIR256-2.5-HSC-EVO
AvaBench	USB3 - EVO-RS - 100mm
Grating	4F - 75 lines/mm
Range	1000 - 2500 nm

Light source:

Light source type	Light source name
Halogen	HAL-S-Mini 2

Software:

Software type	Version number
AvaSoft	8.16.1

Optical fiber:

Fiber type	Diameter (µm)	Lenght (m)	Shielding
FCR-7UVIR	400	2	BX

Reference:

Reference type	Reference name
White tile reference	WS-2

Samples:

Silica type	BET surface area (m2/g)	Loss on heating (2h @ 105 °C) (%)
1	165 - 185	4 - 7
2	150 - 170	6 - 8
3	150 - 170	4.5 - 7.0

METHOD USED

Measurement settings:

Parameter	Value
Slit (µm)	100
Integration time (ms)	5.33
Averaging	100
Smoothing	2

Measuring in AvaSoft:

1. Set the integration time using the Autoconfigure button using the White tile as Reference
2. Set the smooting to 2
3. Close the shutter switch off the light source
4. Record a Dark measurement
5. Switch on the Light source
6. Record a Reference using the White tile
7. Switch the screen to Absorbance mode
8. Record a spectrum of the Sample

RESULTS

Moisture, i.e. water, will appear as two distinct peaks within NIR spectra, namely around 1400 nm and around 1900 nm. Therefore, it was chosen to investigate the samples using both a NIR1.7 and a NIR 2.5.

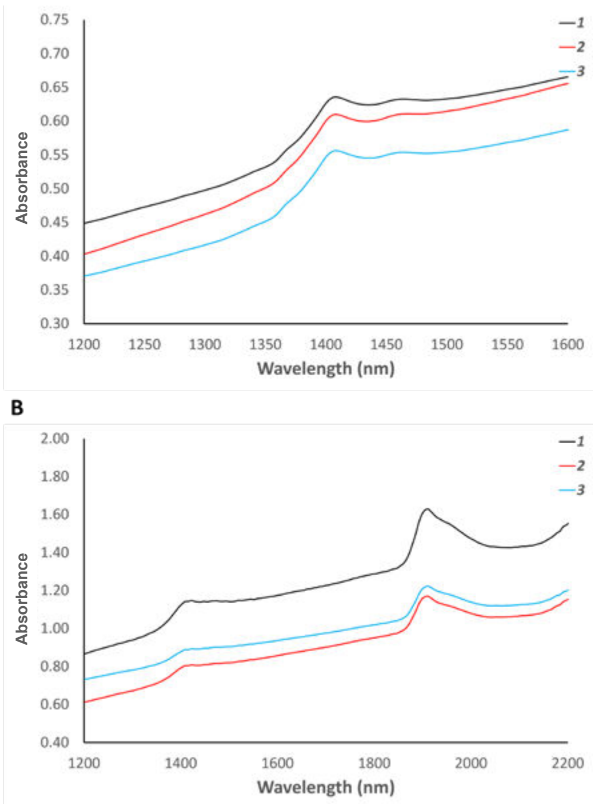

FIGURE 1: Spectra of Silica granulates (sample 1, 2, 3) using a NIR1.7 (A) and a NIR2.5 (B), respectively

RESULTS

The spectra depicted in Figure 1 reveal that Silica granulates contain a notable quantity of moisture within their structure. When employing NIR1.7, which offers a shorter wavelength range but higher resolution compared to NIR2.5, the first derivative shows multiple sharp changes around 1400nm, where the water peak is located.

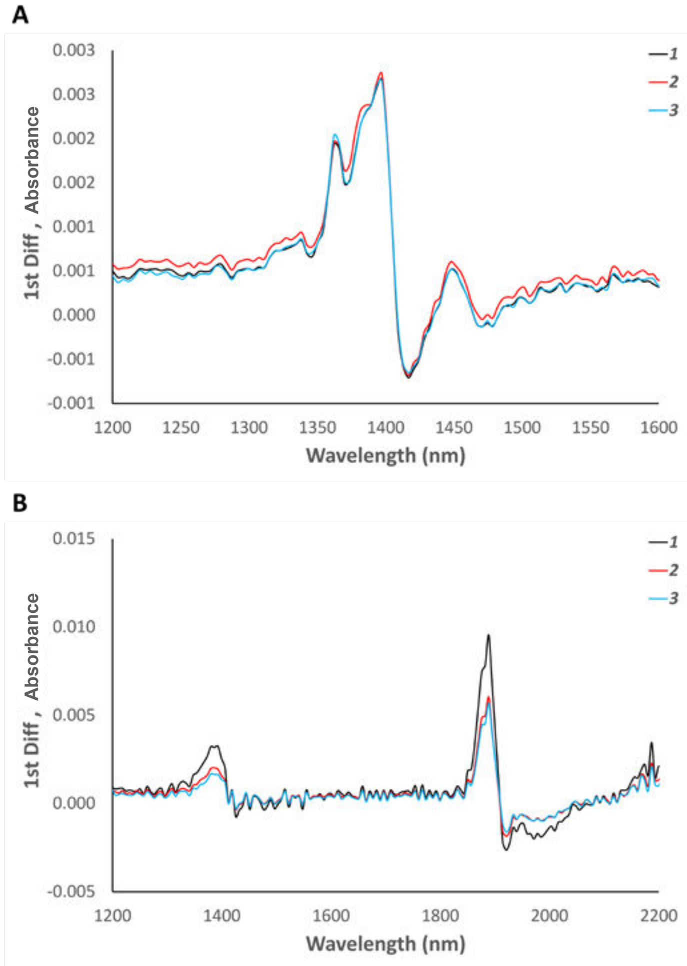

FIGURE 2: First derivative of the spectra (sample 1, 2, 3) measured using a NIR1.7 (A) and a NIR2.5 (B), respectively

After calculating the first derivative of the spectra (Figure 2) to eliminate baseline drift and other influences like particle size and temperature, a slight distinction is noticed among the various types of Silica granulates. However, without knowing the specific moisture content, it is impossible to draw any definitive conclusions.

To establish a meaningful chemometric model, it is necessary to determine the actual moisture level and establish a correlation with the peak areas in the measured spectra. By conducting this analysis for different realistic moisture levels, a reliable chemometric model can be developed.

CONCLUSION

The investigation of moisture content in Silica granulates using NIR spectroscopy highlights its significant analytical value. The NIR spectra reveal distinct moisture peaks at approximately 1400 nm and 1900 nm. Utilizing the higher resolution of NIR1.7 enables precise identification of absorption variations at the water bands' positions, facilitating subtle distinctions among different types of Silica granulates.

Based on specific parameters to be observed, selecting the appropriate spectrometer becomes feasible. For cases with clearly present moisture bands above a few percentage points, the optimal setup involves employing the Avaspec-NIR256-1.7-EVO in combination with the Avalight-Hal-s-Mini2 and FCR-7UVIR400-2-BX fiber.

However, when dealing with low concentrations of moisture, challenging sample discrimination, or the presence of multiple components, the recommended setup comprises using the Avaspec-NIR256-2.5-HSC-EVO with the same accessories.

The establishment of a reliable chemometric model and accurate determination of moisture content enables measurements at different levels and their correlation with water bands.

Applying the Avantes NIR spectrometer alongside the chemometric model yields a fast, non-destructive, and accurate moisture detection system. This approach holds great promise in enhancing analytical capabilities in moisture assessment and offers significant practical benefits in numerous applications.

REFERENCES

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CONTACT

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