International Journal of Environmental Engineering– IJEE
Volume 2 : Issue 1 *[ISSN : 2374-1724]*
Publication Date: 30 April, 2015

Progress of the IR measurement in the area of the fine particulate material water content

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***Abstract*—The paper compares the results of the test of continuous moisture content measurements of lime hydrate under operating unit production conditions where the infra-red spectroscopy method and the classical method of the mass-decrease of samples in drying chamber were employed. The large differences which two measurement systems show are explained in connection of the changes of the measurement process conditions. The paper shows the taken data of infrared spectral measurement of the powder material moisture content as the function of:**

- **the time of the spectral data that are taken;**
- **the influence of the false IR radiation;**
- **the influence of smoothness of the measured powder material surface;**
- **the distance of the measured material surface of the spectral measurement device focus.**

Keywords—moisture content, fine particulate material, infrared spectra

I. Introduction

The moisture content of powdered materials is usually measured by evaluating the mass-decrease of samples in drying chambers or using special, nowadays mostly semiautomatic laboratory balances under standardized conditions, usually at temperatures between 110-140 °C. This method, however, involves some noticeable drawbacks due to the necessity of sampling the material periodically. They are especially the following ones:

- The production control obtains the results periodically at the usual maximum frequency of 1–3 samples per hour.
- The results lag behind the production by a space of time spent in sampling, transporting the sample to the laboratory, and the evaluation itself.
- The procedure requires a laboratory technician who is responsible for the evaluation and sampling.

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A progressive method of moisture control starts to gain ground in the field of particulate production worldwide, namely the moisture control based on evaluating the radiation spectrum of powdered products in the infrared zone.

In cooperation with the companies Nicolet CZ Ltd, Carmeuse Czech Republic Ltd Mokrá, Mettler Toledo and Polz Instruments Ltd, a number of testing spectral measurements were taken in a lime hydrate production plant. During the tests, the moisture content values in lime hydrate which were measured in the conventional manner used by the Carmeuse company, i.e., by measuring the mass decrease on aSartorius automatic balance, were compared with those measured by a Mettler moisture analyser and the spectrum devices of the companies Nicolet and Polz Instruments, Ltd.

The results of our measurement have shown that this prospective technology, apart from its indisputable advantages, also involves some failings and drawbacks.

II. Moisture content measurement

A. *NIR spectroscopy*

The principle of Near Infrared Spectrometry (NIR) consists of measurement and evaluation of radiation coming from the wavelength zone of 700–2500 nm, or wave numbers 12900–4000 cm^{-1}, which is reflected by or goes through the sample. A part of the radiation energy is absorbed by diatomic bonds -CH -OH, -SH, and -NH occurring in functional groups of the substances measured and brings about a change of rotation-vibration state of these bonds. The NIR spectra consist of strips of higher harmonic vibrations (overtones) and their combination strips. It is possible to define zones in which the respective strips predominate: the strip of combinational transitions and the first one up the third overtones. The typical spectrum changes caused by water presence are shown in Fig. 1 where the red line reflects the state with lower water content and the blue one with higher water content.

NIR spectroscopy is a nondestructive method, the sample is neither decomposed nor consumed during the analysis and do not need to be adapted. The measurement itself takes several tens of seconds, the entire process inclusive of an evaluation adequate to present-day computer and measurement technology takes less than 2 minutes. NIR spectroscopy is primarily usable in qualitative analysis. The interpretation of NIR spectra applies mathematical and statistical methods used for data processing in chemometry. Professional literature specifies the parameters obtained by means of NIR spectroscopy in a great number of substances subject to measurement [1], [2], [3] and [4]. Undoubtedly, however, it is MOISTURE content, i.e., water-content in the substance, that constitutes the basic parameter measured by this method.

which utilizes intensive absorption of the OH bond in a water molecule. Theoretically, the specification of moisture content can be performed in all water-containing substances from solid and particulates to pasty, suspension and liquid substances, see [5] and [6].

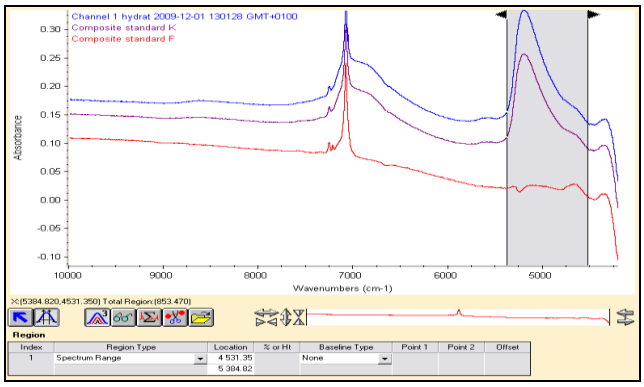

Figure 1. Comparison of NIR spectras of lime hydrate as the function of moisture contents (taken fromthe NIR device screen)

B. *Color spectroscopy*

For describing the color, three parameters can be applied and defined as a point in a color space. In practice, the rectangular system of coordinates CIELAB L, a, b (Fig. 2) is used as color space most frequently. In the scale, L measures brightness and moves from 100 for perfect white to zero for black in a range adequate to human eye's perception. The dimensions of coordinates a and b measure the green-red and blue-yellow transfers, respectively.

In evaluating the samples in which the perception of white color prevails, the degree of whiteness by CIE – WCIE is an important measured parameter. Whiteness is associated with the zone of color space in which the nuances are identified as white. The degree of whiteness is measured by the degree of difference in color from perfect white.

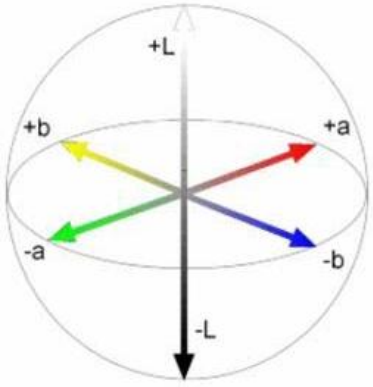

Figure 2. Rectangular system of coordinates CIELAB L, a, b

This type of specifying the whiteness as well as the entire CIELAB system of coordinates is the most adequate to human sight. Its value is defined by the following relationship:

$$W_{CIE} = Y + 800(x_0 - x_a) + 1700(y_0 - y_a) \quad (1)$$

where Y is the so-called tristimulous value or luminosity factor, x_a and y_a are trichromatic coordinates of the sample, x_0 and y_0 are trichromatic coordinates of ideal white for the applied standard CIE lighting and source.

III. Experimental part

In the present paper the data of the current moisture content of lime hydrate produced in the continuously operating production plant of Carmeuse Czech Republic, Ltd, Mokrá-Horákov were compared; the data were obtained by means of four measuring methods:

- a HR 83 Mettler Toledo commercial batch moisture analyser;
- a MA 30 Sartorius commercial batch moisture analyser;
- an on-line moisture-analysis using an Antaris II FT-NIR analyser with a Series 400 diffuse reflex probe (the Nicolet company);
- comparative online analysis using a HK6-POLZ compact microwave device (producer Harrer Kassen);
- a MiniScan EZ spectrometer (the Hunter Associates Laboratory, Inc.).

A. *Results of measurement on the full scale process*

The results of the measurement performed in the spectral manner and, on the other hand, the results obtained by "conventional" checking the loss of mass using thermobalance are compared in Fig. 3, which shows the variability of moisture content in lime hydrate being produced during a part of the day.

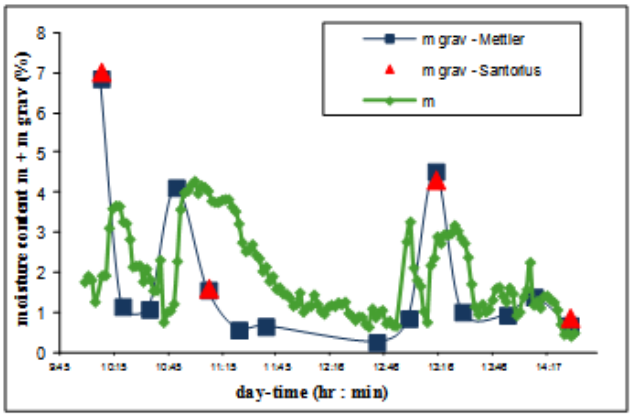

Figure 3. Comparison of moisture-Content measurement by the "conventional" and the NIR spectral methods.

As the given values indicate, the moisture deviations absolute momentaneous values but markedly also in the trends often differ diametrically from each other not only in of these values according to which of the methods – conventional or spectral - were applied.

B. *Results of comparative measurements*

To explain the disproportions in the foregoing measurement, a whole series of subsequent measurements were performed with a view to mapping out possible influences which may have caused the shift of values.

This block of measurement contains mainly:

- The effect of the distance between the sensor and the surface of the material under moisture-test (see Table 1 and Fig. 4), the effect is very strong.
- The effect of "non-smoothness" of the surface of the material on the scanned spectra (see Table 2 and Fig. 5), where "smooth" means the surface smoothed.

TABLE I. EFFECT OF DISTANCE D (BETWEEN THE SENSOR AND THE MEASURED LAYER OF MATERIAL) ON THE M (MOISTURE CONTENT MEASURED BY NIR METHOD)

distance *d* [cm]	**sample 17** *thermogravimetric moisture content* m_{grav} = 0,78 %	**sample 21** *thermogravimetric moisture content* m_{grav} = 0,10 %
20	0.80	0.10
19	0.90	0.40
18	1.10	0.50
17	1.30	0.70
16	1.50	0.50
15	1.80	0.20
14	3.80	0.60
13	-	0.40

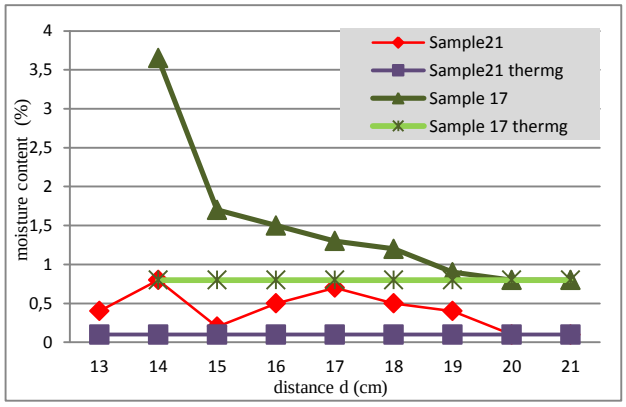

Figure 3. Effect of distance between the sensor and the measured layer of material (where the line Sample 21 is of data taken from IR measurement and Sample 21 thermg of the „classical" thermogravimetric measurement)

TABLE II. EFFECT OF SMOOTHNESS OF SURFACE ON VALUES OF THE SCANNED MATERIAL MOISTURE CONTENT

sample No.	**NIR moisture content measured *m* [%]** *quality of surface*			**thermogravimetric moisture content measured m_{grav} [%]**
	smooth	*not treated*	*defined*	
16	0.5	0.5	0.2	0.79
17	0.8	0.8	0.1	0.78
18	0.8	0.8	0.7	0.59
19	1.0	0.9	0.8	0.78
20	1.1	1.1	0.9	0.77

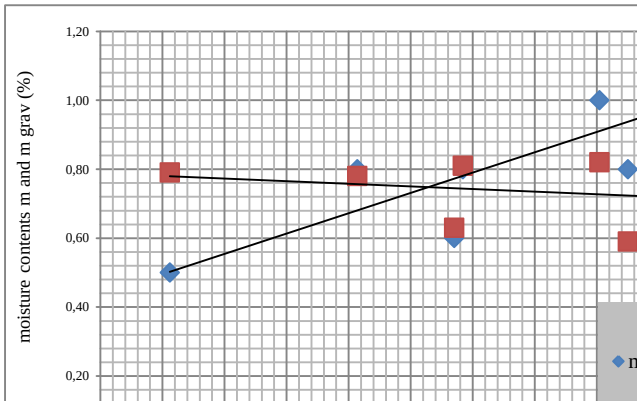

Figure 4. Effect of the changing of colors parameter L of the material surface on the scanned moisture content (where „NIR" means data obtained by IR measurement, „grav" by the classical thermogravimetric measurement)

- By the smooth plate pressing, "not smoothed surface" means the free pured layer surface, "defined" means the surface with 4mm scratches made by the plastering comb. The effect is very strong as well.
- The effect of even very slightly changing colors of the material surface (see Table 3 and Fig. 5), the effect was strong.
- Any effect of the scanning times used has not been found in the area of units to hundreds seconds.
- The effects of hampering lights and mobiles function in the room where the spectra were measured were found to be very strong.

TABLE III. EFFECT OF CHANGING COLORS OF THE MATERIAL SURFACE ON THE SCANNED MATERIAL MOISTURE CONTENT

sample	***L***	***a***	***b***	W_{cie}	**moisture content**	
					m_{grav} [%]	***m*** [%]
16	89.56	-0.17	5.1	51.23	0.79	0.5
17	91.07	-0.06	4.27	58.97	0.78	0.8
18	93.25	-0.58	4.09	64.62	0.59	0.8
19	93.02	-0.55	3.63	66.2	0.82	1
20	93.9	-0.69	3.35	68.92	0.77	1.1
22	91.85	-0.51	3.88	62.21	0.63	0.6
23	91.92	-0.45	3.76	62.96	0.81	0.8

IV. Conclusion

In cooperation with the companies Nicolet CZ Ltd, Carmeuse Czech Republic Ltd Mokrá, Mettler Toledo, and Polz Instruments Ltd, testing spectral measurement in limehydrate production was carried out.

The main purpose of the work was to examine the usability of present-day FT-NIR spectrometry in moisture content measurement of fine-grained powdered materials produced continuously on full production scale. During the measurement under production scale conditions, critical process situations had been generated, which had an essential effect on changes in moisture content of the product; this moisture content was monitored by the conventional gravimetric method using the Sartorius automatic balance and, on the other hand, with the Mettler moisture content analyser and the Nicolet spectral device. The measurement has shown that spectral measurement enables the production technologies of fine-grained particulate substances to check the moisture content of the product continuously with minimal delay. Differences in the recorded moisture content values obtained by spectral measurement and those obtained by gravimetric "conventional" methods have raised a number of issues concerning especially the reliability of maintaining a regular flow of particulate material along the spectral sensor, and also the possible influence of the kinetics of the flow of the measured material on the spectra scanned. Some of the large quantity issues which are connected to the regular flow and its kinetics maintaining were examined by additional measurements. The measurements proved that the moisture content values are exposed to an essential effect of the distance between the sensor and the layer of the material measured, an effect of the constantly adapted surface of the material layer, an effect of even slightly changing colors of the material surface and an effect of hampering lights and switched-on mobiles phones in the room where the spectra were measured. The effect of the duration of the spectrum scanning has not been found. Generally speaking, the measurement proved that the method of spectral moisture content measurement in the production and processing of fine grained particulate substances is still a novelty which requires a longer verifying period. Apparently, however, after getting over the unavoidable time of teething troubles, the method of spectral moisture content measurement, which is able to monitor the quality of continuous production processes permanently, will certainly be preferred in the near future.

Acknowledgment

This work is an output of cooperation between the project No. CZ 1.07/2.3.00/20.0188, HEATEAM – Multidisciplinary Team for Research and Development of Heat Processes and by the Support to the WP EFCE Activity, LG13062, INGO II of Czech Ministry MSMT and Development of Heat Processes and NETME Centre, regional R&D centre built with the financial support from the Operational Programme Research and Development for Innovations within the project NETME Centre (New Technologies for Mechanical Engineering), Reg. No. CZ.1.05/2.1.00/01.0002 and, in the follow-up sustainability stage, supported through NETME CENTRE PLUS (LO1202) by financial means from the Ministry of Education, Youth and Sports under the „National Sustainability Programme I.

References

[1] Rouquero,l J.: Absorption by Powders and Porous Solids, Elsevier, N.Y.1999)

[2] Hillel, D.: Introduction to Environmental Soil Physics, Elsevier, N.Y. 2003

[3] Nicolet devices handbook, FTIR spectrometer, Thermo Electron Corporation, Prague 2007

[4] Uomoto, T.: Non-Destructive Testing in Civil Engineering 2000, Elsev., London 2000.

[5] Míka,V. at all.: Spektroskopie v blízké infračervené oblasti (Czech) (Spectroscopy in NIR), Praha, 2008.

[6] Sverak, T., Sikorova, K., Stevulova, N., Estokova, A., Hollein, M.: Moisture Content of Powdered Materials Continuous Measurement, 7th European Congress of Chemical Engineering, Prague, Czech Republic, Prague 2010