CALIBRATION OF AN INFRA-RED HUMIDITY SENSOR
FOR ON-LINE APPLICATION IN CERAMIC TILE INDUSTRY

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Abstract. In the ceramic tile industry the humidity content in both the atomised powder and the green bodies is one of the most influential variables and therefore it should be controlled in real-time. The humidity of the atomised powder is usually kept within the range 4.5-6.5 % by control of the atomiser parameters. Normally, the humidity control is made off-line by manual thermal balance systems with sample taken just after the atomiser every hour. The aim of this work is to evaluate the possibility of using a Near-Infrared (NIR) sensor (1-3 µm), for on line application on the ceramic powder, so that closed loop automatic feedback control can be implemented. A laboratory calibration is performed, in order to estimate repeatability, sensitivity and the effects of possible interfering inputs. Then the sensor was installed on-line and the calibration was performed with a reduced number of samples, in order to assess the efficiency of a quick procedure to use for industrial applications. An accuracy of about ± 0.60 % of humidity was found. At end, the sensor was employed, in conjunction with a slip flow rate sensor, to create a complete real-time control on the atomiser. Results show the effectiveness of the proposed technique.

1. INTRODUCTION

In recent years the industrial research has spent many efforts in the field of process automation, where satisfactory results have been achieved also thanks to the development of innovative measurement techniques. The possibility of accurately and non-intrusively measuring important quantities stimulates the investigation on the industrial processes, with the fundamental aim of implementing on-line control systems based on real-time feedback information supplied by the sensors. Such control was not feasible in the past, when measurements were mostly based on contacting techniques.

The ceramic industry is one of those sectors where the degree of automation of the production processes is high, but the on-line control strategies and techniques need to be

completely developed and applied yet. The humidity content in both the atomised powder and the green bodies is one of the most influential variables and therefore it should be controlled in real-time. High or low powder moisture content at the exit of the atomiser may generate problems in the subsequent processes: for example, the green bodies can have low mechanical resistance and thus some failures, as cracks or delaminations, may appear before the tiles go into the kiln. A variety of non-destructive evaluation methods, NDE, for on-line control have been developed and presented in literature. General NDE techniques applicable for structural faults in ceramics and in their composites are based on optical (Cielo, 1992, De Andrade et al., 1998, De Andrade et al., 1999), radiography and ultrasonic (Vary et al., 1993; Bhardwaj, 1997) methods. However, discovering problems "after-the-fact" can be a costly situation in terms of reject.

The humidity of the atomised powder is usually kept within the range 4.5-6.5 % by means of the atomiser parameter, as flow rate and pressure of the slip and air temperature, Negres et al (1994). Normally, the measurement of humidity is made off-line by manual thermal balance systems with sample taken, every hour, immediately after the atomiser. This long period of time between two measures makes the control of the atomiser difficult and might cause humidity irregularity or scarce humidity in the powder. For this reason on-line control is desirable and it should be performed immediately after the atomising process, as shown in Figure 1.
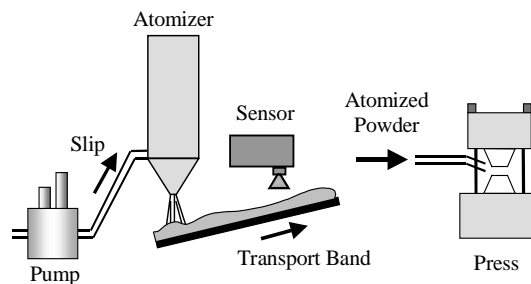
Figure 1. Measurement of the ceramic powder moisture content: scheme of the on-line installation.

Several measurement techniques for on-line analysis of moisture content in solids have been presented in literature (Dartnell and Berry, 1974): these are mostly based on micro-wave attenuation, nuclear radiation, infrared absorption, electrical resistivity and capacitance determination. The choice of a technique for a specific application must take into consideration the effects of interfering inputs (such as temperature variations, dissolved salts, particle size variations or bulk density) and the purpose for which the measurement is being made.

The aim of this work (carried out within the research of the BRITE-EURAM Project No.96.3632 *IMPACT*, Integrated Manufacturing and Production Automation for the Ceramic Tile industry) is to evaluate the possibility of using a Near-Infrared (NIR) sensor for the specific application on the ceramic powder. After a brief review on the state-of-art of on-line measurement systems for moisture content, the attention is focused on the calibration procedure for the NIR sensor. The calibration is firstly performed in the controlled environment of the laboratory, in order to estimate repeatability, sensitivity and the effects of possible interfering inputs, as the powder colour. Once the calibration procedure was established, the sensor was installed on-line in the plant of LEONARDO 1502 CERAMICA SpA at Imola (Italy). The calibration was performed once again, but with a reduced number of samples, in order to assess the efficiency of a quick procedure to use for industrial applications. Also the "speckle" effect due to the movement of the powder on the belt under

the measurement light spot has been quantified and considered in the calibration results.

At end, the sensor was employed, in conjunction with a slip flow rate sensor, to create a complete real-time control on the atomiser. Results show the effectiveness of the proposed technique.

A similar approach was presented by Negre et al (1992), which have found a good correlation between measured and reference data. The same authors utilized the sensor also for the atomiser control using an empirical relationship between air temperature and moisture content in the powder, Negres et al (1994). The present work confirms their results and extends the uncertainty analysis for the NIR sensor in terms of both statistics and interfering inputs. Furthermore, the time constant of the atomiser control is here evaluated.

2. STATE OF ART FOR ON-LINE MEASUREMENT OF MOISTURE CONTENT OF SOLIDS IN TRANSIT

Humidity is a quantity depending on the presence of water and its vapour, in solid, liquid and gas. Specific humidity (SH) is defined as the ratio between the water mass quantity in a substance and the total mass quantity of the substance.

For on-line measurement in ceramic powder the following practical requirements must to be satisfied by the sensor, (Brunelli A, 1992):

- high sensitivity over a wide humidity range (3 - 8 % SH);
- faster response than off-line measurement;
- robustness and reliability in harsh environment (temperature, contaminants, vibrations, etc.);
- good repeatability and no hysteresis;
- simple structure and low cost.

Several measurement techniques for on-line analysis of moisture content in solids have been studied (Dartnell and Berry, 1974, Pyper, 1985): these are mostly based on micro-wave attenuation, nuclear radiation, infrared absorption, electrical resistivity and capacitance determination. In general, the choice of a technique for a specific application must take into consideration the form in which the moisture is present (e.g. total moisture, surface moisture or free moisture content), the effects of interfering inputs (such as temperature variations, dissolved salts, particle size variations or bulk density) and the purpose for which the measurement is being made.

Micro-wave attenuation technique is based on property of water having an absorption at certain frequencies of electromagnetic radiation in the microwave range (1 GHz $\div$ 300 GHz). Absorption is proportional to water concentration of sample substance, but it also depends on water conductivity. More reliable results can be achieved at frequency greater than 100 GHz, because at such frequencies effects due to conductivity are minimized. Sensor based on microwaves could give problems for the on-line application because of its inherent sensitivity to several interfering inputs. For example, the sensor output is sensitive to the presence of metallic parts in the measurand and this is a quite frequent situation in ceramic industry, since the raw materials are usually transported by trucks, where metallic products can be carried during other deliveries. In some on-line tests performed (not shown here), this sensor presented strong non-linearity in the range between 3 % and 8 % SH, which is the range of interest for the application on the ceramic powder. Such instruments can be found applied more over in fields such as paper, cardboard, plastic or textile. Micro-waves principle has a theoretical measurement range from 0 % to 100 % SH and typical accuracy 1 %.

Nuclear radiation principle is based on water absorption of nuclear radiation. The instrument uses radioactive sources emitting α, β, γ or X radiation. Radiation, passing through

humid substance, is attenuated and absorbed by hydrogen atoms of water molecules. These instruments are able to measure very little concentration of water in solid substances. The most important drawback of radiation principle is due to human exposition to radiation, which imposes costly and complicated installations. Instruments, which use radioactive sources, must be in conformity with the straight regulations and frequent inspection. Also, special personal is required to manipulate the instrument. Nuclear radiation principle measurement range is from 0 % to 100 % SH and typical accuracy is 1 %.

The technique based on the measurement of the electrical resistivity has been widely used on a range of materials from cereals to moulding sands and iron ores. It measures the surface humidity and is not applicable to materials which in themselves are electrical conductors or in which water soluble salts may be present. In standard condition, it has an accuracy of about 0.5 %.

The capacitance method is based upon water having a dielectric loss approximately 10 times that of most substances in which it is absorbed. It is not suitable for use on materials which are electrical conductors and its accuracy is of about 1.5 %. It measures the total moisture content.

Both electrical methods have the drawback of requiring a dedicated calibration for each particular kind of installation and measurand, since they are sensitive to many interfering inputs (e.g. particle size). Furthermore, as with most contact devices on moving belts mechanical wear is a problem and attention must be paid to wear of materials changing the geometry of the measuring head.

3. NEAR-INFRARED SENSOR

Near-Infrared principle is based on water vapour absorption of electromagnetic radiation in the near infrared range (1 − 3 µm). The absorption is dependent on the chemical composition including the humidity contents. Figure 2 shows the infrared reflection spectra (ρ) for 1 % and 5 % SH content. The spectrum shows that at a wavelength of 1.70 µm, the infra-red reflection is independent of humidity, whilst at 1.94 µm the infra-red reflection is reduced by increased humidity. In order to utilize the change in reflectance as an indirect SH measure, it is necessary to eliminate all changes in reflectivity due to extraneous effects (as the distance between the source and the measurand and environment temperature). This is usually achieved by using the reflectance at 1.70 µm as reference. The logarithm of the ratio between the reference beam and the measurement beam energies is directly proportional to the specific humidity content of the sample substance (Lambert-Beer's lay). The humidity is therefore determined by the ratio of the signal emitted at reference frequencies and the humidity material reflection frequencies. Infrared principle has a theoretical measurement range from 0 % to 100 % SH and an accuracy of about 0.5-1 %.
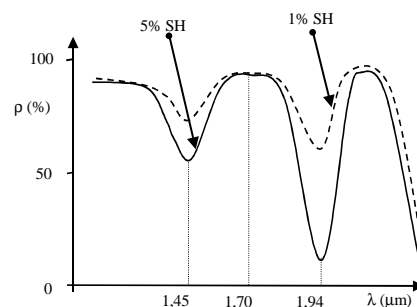

Figure 2 - Infra-red reflection spectra for 1 % and 5 % SH content.

For the application on solids, the infrared ray analysers are composed essentially of an infrared light source, a wheel with filters and a round mirror that converges towards a detector both the reference beam and the measurement beam, Figure 3. Accurate filters mounted on the wheel select the two different wavelengths. The motorised wheel splits, through its filters, the light beam into the two different wavelengths and sends them alternatively to the detector: the reference beam directly, while the measurement beam is reflected from the sample substance, which however does not entirely reflect the incident ray because of absorption from its water content. Moreover there are often two additional rays in the analyser. They come from the same light source and they arrive to the same detector, but they are not projected on to the sample substance; these two rays are used to compensate any light source intensity change.
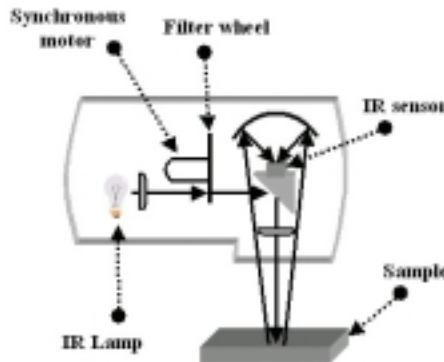
Figure 3 - Infrared measurement device.

4. CALIBRATION PROCEDURE FOR THE NIR HUMIDITY SENSOR

Since the NIR sensor is based on an indirect measurement, it must be calibrated by comparison with a reference method (usually a thermal balance). During the calibration, the slope (sensitivity) and the zero (offset) of the calibration line are determined, together with the random uncertainty.

NIR calibration accuracy can be no better than the sampling and the reference methods used. The quality of sampling techniques, the laboratory references methods utilised, and the range of samples available are critical to gain the best possible accuracy in a NIR application. The sampling technique must insure that the laboratory sample is representative of the sample measured by the sensor on-line. Furthermore, the calibration samples should cover at least the entire range of variation ever produced by the process.

Errors in the reference and sampling methods are frequently the limiting factors on the NIR measurement accuracy. Thus, a dedicated study was performed to design a correct calibration procedure. This procedure has been divided into five steps.

1) Preparation of the samples: a certain quantity of powder (base colour), withdrawn at the exit of the atomiser, has been artificially humidified by a spray of water and then put in a pond container. After shaking it, we have waited a time sufficient in order for the moisture to be distributed in a homogeneous way within the powder. The humidified powder has been divided in N different portions. Each portion has been desiccated for a different time, in such a way as to have representative samples within the complete range of interest (3% to 8% of SH), since it has been seen that the desired value is about 5%.

2) Measurement of the reference specific humidity, H_R. The humidified powder from each portion has been weighed by a precision balance, W_h. Then it was completely desiccated and the dried powder has been weighed, W_d. The reference specific humidity H_R has been

calculated as follows:

$$H_R = \frac{W_h - W_d}{W_h} \tag{1}$$

This procedure is particularly long, and it destroys the portion of sample used as reference, which therefore is available only once.

3) Laboratory calibration of the infrared sensor. The NIR sensor was calibrated in "ideal" conditions, i.e. without the interfering inputs typical of the on-line installation (like movement of the powder on the belt, temperature variations, vibrations, etc.). The acquisition time was set at 10 s, in such a way as to have an average on 10 readings. Once the instrument has measured the samples, the electronic control unit calculates a calibration straight line, which will be used for the conversion of the measured intensity into humidity values.

The calibration accuracy was then checked: the same samples was measured by the sensor and the interpolating calibration line, Equation 2, was estimated by a last-squares criterion (Doebelin, 1990). The dispersion of the data in output $SD(H_M)$, Equations 5, and then the standard deviation in the input variable $SD(H_R)$, Equation 6, have been statistically evaluated too.

$$H_M = mH_R + b \tag{2}$$

where H_M is the SH measured by the NIR sensor, m is the slope of the straight line and b the interception on the vertical axis, calculated as follows:

$$m = \frac{N\sum(H_R H_M) - \sum H_M \sum H_R}{N\sum(H_R)^2 - \sum(H_R)^2} \tag{3}$$

$$b = \frac{\sum H_M \sum(H_R)^2 - \sum(H_R H_M)\sum H_M}{N\sum(H_R)^2 - \sum(H_R)^2} \tag{4}$$

$$SD(H_M) = \sqrt{\frac{\sum_1^N (mH_R + b - H_M)^2}{N}} \tag{5}$$

$$SD(H_R) = \frac{SD(H_M)}{m} \tag{6}$$

This step can be repeated several times, in order to iteratively increase the calibration accuracy, but some contrivances must be taken. The results of three consecutive calibrations are reported in table 1 in terms of standard deviations in input and output and of slope and intercept of the least-squares interpolation line:

Table 1. Results of three consecutive laboratory calibrations

Calibration	m	b	SD(H_M)	SD(H_R)
a	0.83	0.95	0.15	0.18
b	0.98	0.22	0.20	0.20
c	0.99	0.16	0.35	0.37

The Calibration a) is performed by using $N = 15$ samples, Calibration b) uses the same reference samples two times, Calibration c) uses them three times. So, the calibration is performed by increasing the number of reference samples. This improves the results, m tends to 1 and b to 0, but dispersion $SD(H_M)$ increases. This is due to the deterioration of reference samples, whose humidity changes during the long time needed for an accurate calibration: therefore, it should be necessary to re-measure the reference samples at each calibration. Calibration time is a serious concern for industrial application, and should be kept as short as possible. Calibration b) (Figure 4) represents the best result here achieved, with an accuracy of about $\pm$ 0.60 % of humidity ($\pm 3\ SD(H_R)$) .

It is worth noting another factor, which is related to the fact that the thermal balance measures the total humidity, while the NIR sensor the superficial humidity. This difference may generate both systematic and random uncertainties in the results. However, the accuracy level here achieved results satisfactory for the control of the atomiser. This problem must be considered for applications where a lower uncertainty is necessary. However, it is important to underline that, being the research developed for an industrial application, all the steps in the calibration procedure were designed in such a way as to be as simple as possible, trying to achieve an optimal compromising between accuracy and complexity.

High uncertainty was found for the low humidity level (below 3 %), as the SNR significantly decreases. Thus, it could be better not include samples within this range in the calibration set for the application on ceramic powder (where the range of interest is 4.5 – 6.5 %), since these can introduce relevant deviations in the calibration line.

$$H_M = 0{,}98\ H_R + 0{,}17$$
$$R^2 = 0{,}98$$
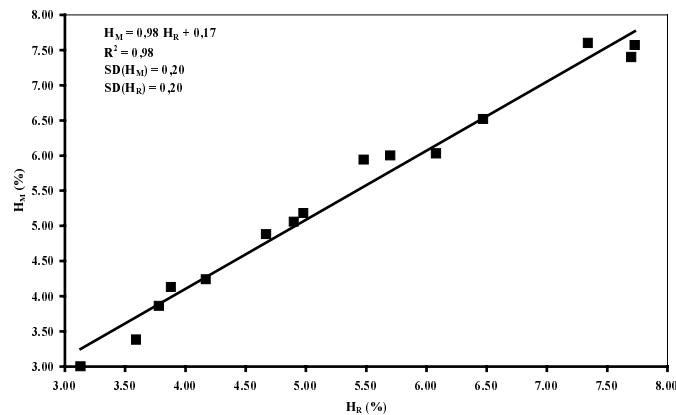$$SD(H_M) = 0{,}20$$
$$SD(H_R) = 0{,}20$$

Figure 4 - Infrared laboratory calibration results.

4) *Adjustment in the range of interest.* The infrared sensor was also calibrated by using very reliable samples (whose reference values H_R have been obtained by averaging on more measurements performed by the thermal balance on different portions of the same samples), all having values of SH, which are close to each other and near to the mean value of the range of interest ($\cong$ 5,5 %). In this way the slope of the calibration line is unaffected and only the intercept b is corrected; the systematic uncertainty is further reduced.

5) *On-line calibration: evaluating the influence of possible interfering inputs.* The infrared sensor has been tested also on-line, in order to evaluate the influence of possible interfering inputs due to the on-line installation (e.g. vibrations, light, etc.). The same procedure of step 4) was repeated with the sensor installed on-line: the only difference with the real functioning conditions is that the powder is at rest under the measurement spot light, and not moving transported on the belt. Furthermore, the calibration was performed with a reduced number of samples, in order to asses the efficiency of a quick procedure to use for industrial applications. It was found that an acceptable uncertainty ($m = 0.96$, $b = 0.17$,

$SD(H_M) = 0.26$, $SD(H_R) = 0.26$, Figure 5) could be achieved using at least 7 samples. If a lower number of samples are utilised, the slope of the line could be significantly shifted.
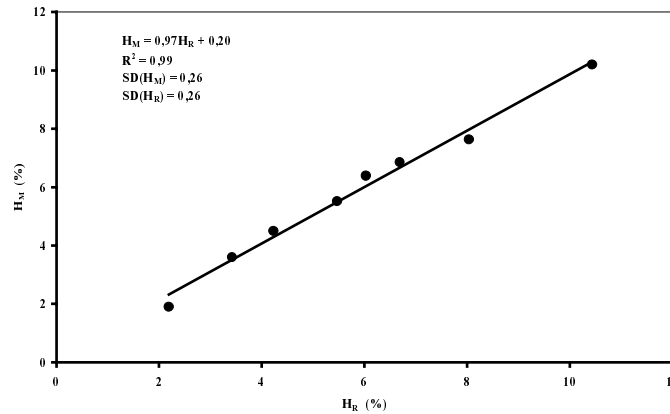

Figure 5 - Infrared on-line calibration results.

It was experimentally verified that the influence of the light external is notable, to such an extent that the sensor goes in error if directly illuminated. For this reason we have thought of protecting the sensor in line from the light using a box.

At the end also the interfering inputs due to the movement of the powder on the conveyor belt was considered. In fact, it generates a typical *speckle* phenomenon (Beckman and Spizzichino, 1963) on the measurement light spot, which introduces optical noise on the detector and thus further measurement uncertainty. Dedicated experimental tests (where reference samples were measured while moved on the belt) have shown that the effect of speckle is systematic and a correction of about 0.3 ÷ 0.4 % SH is required in order to compensate it.

6. CALIBRATION LINES FOR DIFFERENT COLOURS

The pigments used in the body of porcelain stoneware can adsorb light in the same wavelength of work of the IR humidity sensor, so that sensor performance is affected. This has been experimentally verified by effecting trials of reading on samples of powder having the same content of humidity but a different colour, i.e. added with different pigments or combination of pigments. In many cases the reading differs significantly (even 3-4 % SH).

For this reason it was necessary to perform different calibrations according to the powder colour. For the calibration we have used the same procedure already described and we have obtained the same values in terms of linearity and random uncertainty.

In Figure 6 the signal intensity (normalised between 0 and 1) with respect to the humidity content is shown for the different colours, while Figure 7 reports the calibration results in the range 4.5-6.5 % obtained for the same colours. As expected, the colours black and brown give a lower signal level with respect to the others. Besides, once the sensor was calibrated, it presents calibration lines nearly coincident for all the colours in the range of work in the spray drying process, or the differences are of the same order of magnitude of the calculated uncertainty in the reading.

7. CLOSED LOOP PROCESS CONTROL FOR SPRAY DRYING

The spray drying process consists in the drying of the ceramic slip by spraying it in a hot

air stream, by means of which the water undergoes rapid evaporation. The product obtained is made up of round granules with controlled particle size and humidity.

The principal process parameters are indicated in Figure 8. The variables are divided in:
- input variables: process parameter that can modified;
- output variables: variables which should be controlled or monitored;
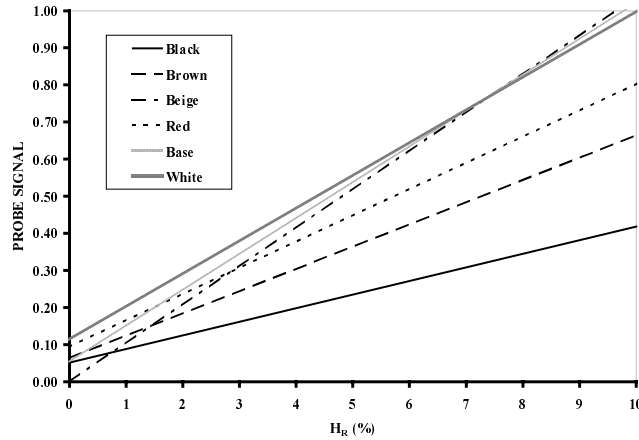- disturbances: variables which affect the process output but are not modifiable during the process.

Figure 6 – Probe signal intensity (normalised between 0 and 1) with respect to the humidity content for the different colours.
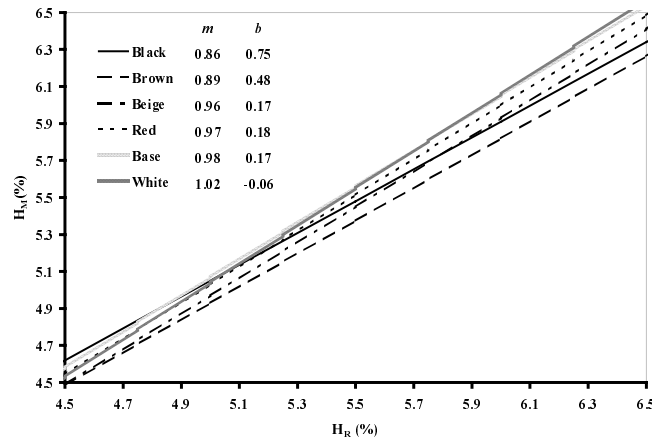

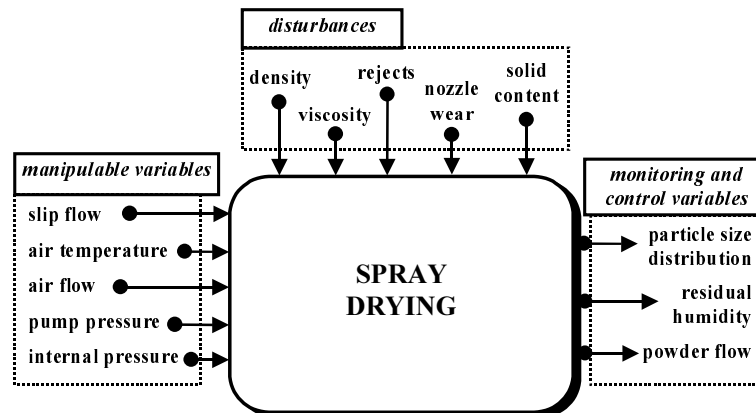Figure 7 - Calibration results for different colours.

Figure 8 – Spray drying process diagram

The two most important output variables are grain size distribution and humidity, because they greatly influence the compacting behaviour of the powder in the following phase of the producing cycle: the pressing phase. They are commonly controlled by the operator off-line. The input variable by which the humidity is controlled is the temperature of the hot air stream (Negres et al 1994). The installation of a sensor able to monitor continuously the humidity allows to implement a closed loop control system, that is schematised in Figure 9. The manipulated variable is also in this case the hot air temperature, T, which is regulated in function of the difference $\varepsilon = H_M - H_{SP}$ between desired and measured humidity.

This type of control has been implemented at Leonardo 1502 with good results, by the use of a PI controller. Figure 10 shows the time history of the air temperature and of the consequent powder humidity during the automatic control. The time delay between temperature regulation and humidity variation is about 3 min, which is a measure of the inertia of the spray drying process.

Figure 11 reports the value of humidity (measured by the NIR sensor) in function of time in the case (a) of manual control (effected by the operator at intervals of about one hour) and in the case (b) of closed loop control. The differences from the set point are in the second case decidedly lower, showing thus the effectiveness of the proposed technique.
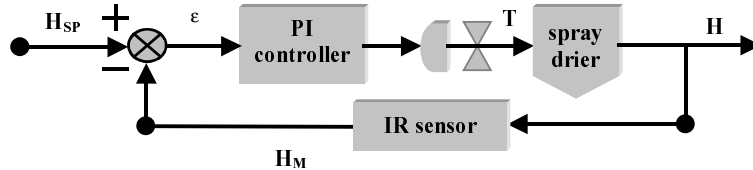

Figure 9 - Closed loop control of humidity in the spray drying process
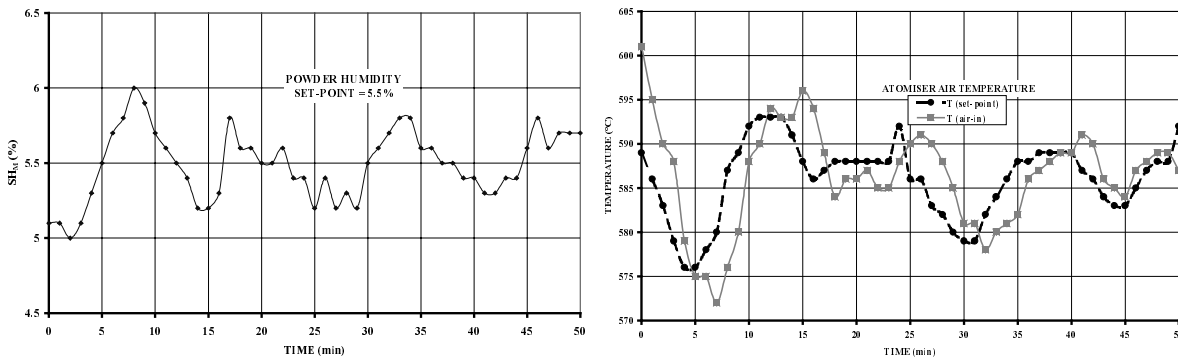

Figure 10 – Air temperature and control of humidity in the spray drying process
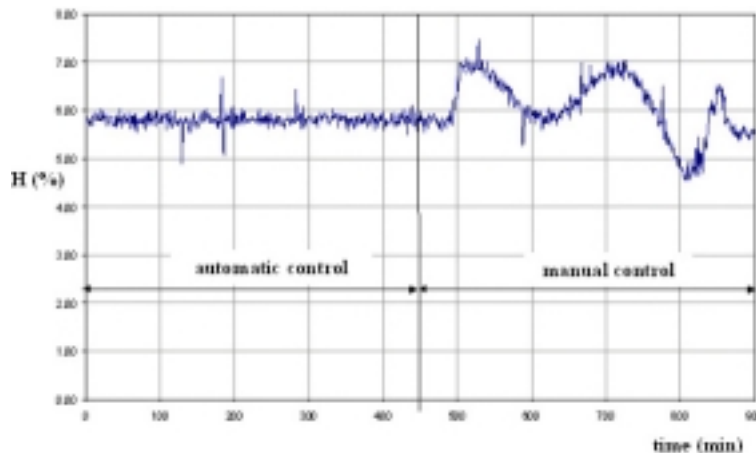

Figure 11 – Humidity on-line control on the atomised powder

7. CONCLUSIONS

Near-Infrared (NIR) sensor is a suitable instrument for on-line monitoring of moisture content in many different materials, but its correct calibration is particularly difficult and crucial in order to have reliable results. NIR calibration accuracy can be no better than the sampling and the reference methods used to characterise the reference samples. The quality of sampling techniques, the laboratory references methods utilized, and the range of samples available are critical to gain the best possible accuracy in a NIR application.

In this work a calibration procedure of a NIR sensor for on-line humidity measurement on ceramic powder was established and tested. It was shown that the calibration accuracy improves if performed by increasing the number of reference samples, but dispersion increases. This is due to the deterioration of reference samples, whose humidity changes during the long time needed for an accurate calibration: therefore, it should be necessary to re-measure the reference samples during the calibration or to keep the calibration time as short as possible. This allows to keep the samples reliability high in time. The best result here achieved is a calibration with an accuracy of about ± 0.60 % of humidity (± 3 $SD(H_R)$) .

The real problem of the calibration procedure is in the preparation and accurate definition of the reference samples. In fact, as it is very difficult to homogeneously humidify the powder, the moisture content of the samples used for the reference measurement is often different from that of the samples used for the calibration. In addition, the infrared sensor measures the value on the sample surface, while the balance, used as reference, determines the moisture of the whole sample. At the end, as previously stated, it is not easy to maintain their humidity content constant in time.

The incidence of interfering inputs typical of the on-line installation was evaluated by repeating the calibration procedure on-line. Furthermore, the calibration was performed with a reduced number of samples, in order to assess the efficiency of a quick procedure to use for industrial applications. It was found that an acceptable uncertainty ($m = 0.96$, $b = 0.17$, $SD(H_M) = 0.26$ %, $SD(H_R) = 0.26$ %, Figure 5) could be achieved using at least 7 samples. If a lower number of samples are utilised, the slope of the line could be significantly shifted. Also the correction required for the application on different powder colours or for the interfering inputs due to the movement of the powder on the conveyor belt was considered.

At the end, the sensor was applied to close a complete real-time control loop on the atomiser. Results show that the implemented procedure is capable of regulating the input air temperature, in such a way as to significantly reduce the variations of the moisture content with respect to the desired set point.

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