MICROWAVE SENSORS AND THE VE2 SYSTEM FOR ON-LINE MOISTURE MEASUREMENT IN THE CERAMIC SECTOR

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ABSTRACT

Production processes are found in different industrial sectors in which the percentage of water has an important influence on end product quality. ***Monitoring moisture*** *volume in real time, independently of colour, with reliable and user-friendly systems has become an objective that will provide great advantages in terms of product quality and economic savings.*

In this study, the general features are considered of the development of an on-line moisture content measuring system for solid substances like ceramic powders. It is to be noted that the system described can also measure density, and water percentage with respect to the liquid: this only requires changing the type of sensor.

1. INTRODUCTION. APPROACHING THE MEASUREMENT: PRELIMINARY STAGES

1.1. CHARACTERIZE THE MATERIALS

It is first necessary to characterize the materials in the microwave range with laboratory tests: "Time domain reflectometry". Determining the dielectric properties [ε' = dielectric constant, ε" = loss factor] of the product means accurately anticipating radiation-material interaction. The definition of moisture is as follows:

$$M\% = \frac{P_{H_2O}}{P_{H_2O} + P_{Dry}}$$

For convenience, we can distinguish three families of dielectric materials in relation to tan δ = ε"/ε' (loss tangent):

Low-loss dielectrics (tan δ << 1)
Medium-loss dielectrics (tan δ = 1)
High-loss dielectrics (tan δ >> 1)

The use of nomograms (ε', tan δ) helps understand the correlation between the dielectric properties of the medium and the electric properties of the electromagnetic field (e.g., for an attenuation of the 10 dB field at a given frequency - knowing tan δ - we can determine the necessary thickness of the product, etc.).

1.2. SENSOR TYPOLOGY

Sensor typology depends on the media (low-loss dielectrics, medium-loss dielectrics, high-loss dielectrics) and on the permittivity behaviour (frequency, temperature, density, formulation): the sensors shall be sensitive to variations of (ε', ε") in the selected working frequency and temperature range. A resonant transmission sensor is indicated for low-loss dielectrics, a resonant reflection sensor is indicated for high-loss dielectrics, antennas (horns) - in free space configuration - are indicated for materials with dielectric losses that are not too small. The VE2 system uses a multiparameter reflection sensor.

1.3. THE FREQUENCY RANGE

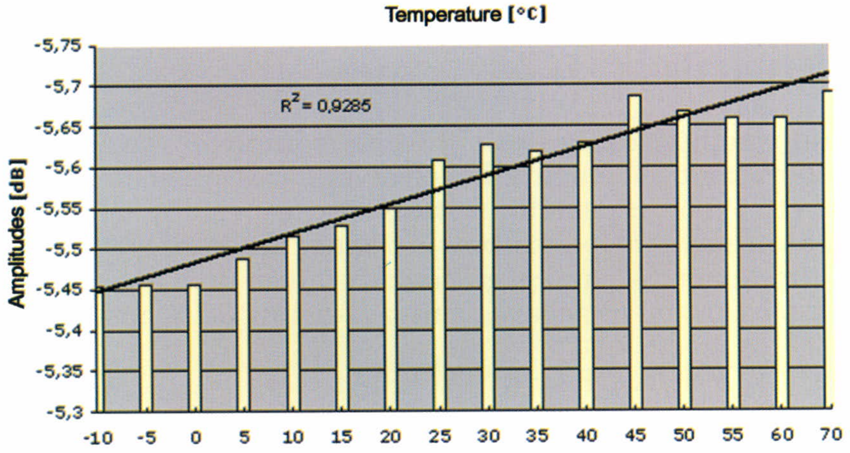
The frequency range we are interested in lies between 600 [MHz] and 13000 [MHz]. In this range, we can measure the moisture content of all the ceramic powders in industrial processes. The selection of the working frequency range depends on the results of the dielectric characterization of the material, the variations of the components that make up the material formulation (metallic compounds, etc.) and also on the type of sensor (resonant structures, radiant systems, etc.).

1.4. THE TEMPERATURE VARIABLE

Sensors are characterized in terms of temperature, for which a climatic chamber with controlled humidity is used. In addition, it is necessary to characterize the product and the entire measuring system: sensor + product + electronics, as a function of temperature. The moisture measuring system must count on temperature sensors set in some strategically important points.

S21				
T [°C]	frequency [MHz	Q (merit factor)	bw(band width)	loss [dB]
-10	694,28	347,60	1,990	-5,453
-5	694,54	347,60	1,995	-5,456
0	694,75	347,60	1,998	-5,457
5	694,97	348,10	1,998	-5,487
10	695,10	346,60	2,005	-5,515
15	695,19	346,00	2,013	-5,527
20	695,24	344,90	2,019	-5,548
25	695,27	342,40	2,030	-5,608
30	695,20	342,20	2,031	-5,627
35	695,05	341,60	2,034	-5,619
40	695,25	341,20	2,040	-5,630
45	695,04	340,10	2,042	-5,685
50	694,93	340,40	2,040	-5,667
55	694,93	342,80	2,030	-5,659
60	694,96	342,10	2,036	-5,659
70	695,08	343,50	2,020	-5,690

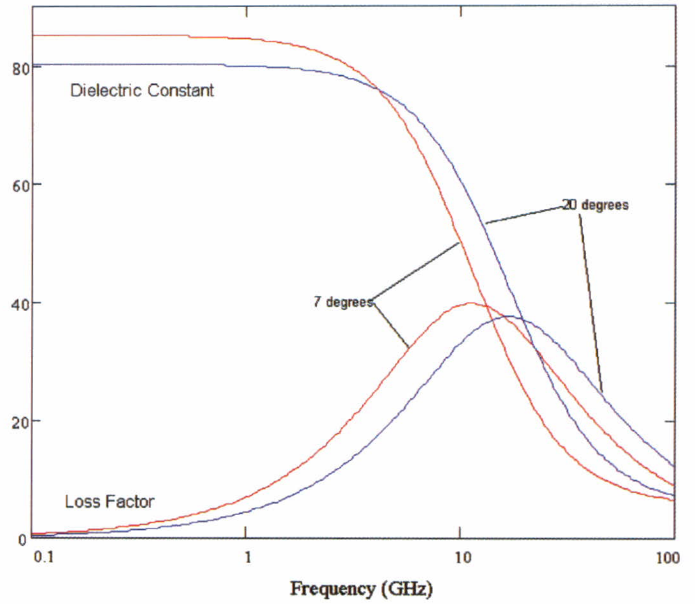
Table 1. Tests in the climatic chamber



Graph 1.

The electric parameters of the sensor and the dielectric characteristics of the powders are influenced by temperature. This requires developing sensors whose electric parameters are relatively insensitive to temperature variations: in certain cases it is advisable to have sensors at 70 - 80°C (by thermostatting).

The figure shows that the dielectric properties of the media depend on temperature: it is necessary to monitor temperature.



Graph 2.

The displacement with temperature is readily compensated with our system.

1.5. ELECTRONIC CONFIGURATION OF THE SYSTEM

The electronics must measure the Sij parameters. The measurement can be scalar or vectorial, reflection or transmission measurements, in an appropriate frequency range, with the appropriate transmission power, always below 10 mW. For ceramic powders, in view of their dielectric characteristics, we have decided to measure the S11 parameter, amplitude and phase at multiple frequencies (vectorial mini network).

1.6. SYSTEM REPEATABILITY TESTS

The vacuum repeatability tests of the complete measurement system deserve particular attention: electronics + waveguides + sensor + process attacks (hopper, ducts, etc.). The repeatability tests and especially product calibration (calculation of the sensor response-powder moisture curves) with temperature variation are "fundamental" and significantly affect the accuracy of the final measurement; a measurement accuracy always below 0.2% absolute.

1.7. CHARACTERIZATION OF THE MATERIALS

The product must be characterized with variation of moisture, density, temperature and formulation. In present measurement systems, it is always advisable to insert the sensor in a process zone where the independent product variables undergo variations that are also controlled by other sensors; it is the objective of every production process to maintain constant working conditions. The VE2 system takes this into account and therefore fully "compensates" all the process variations mentioned above.

1.8. ON-LINE MEASUREMENTS

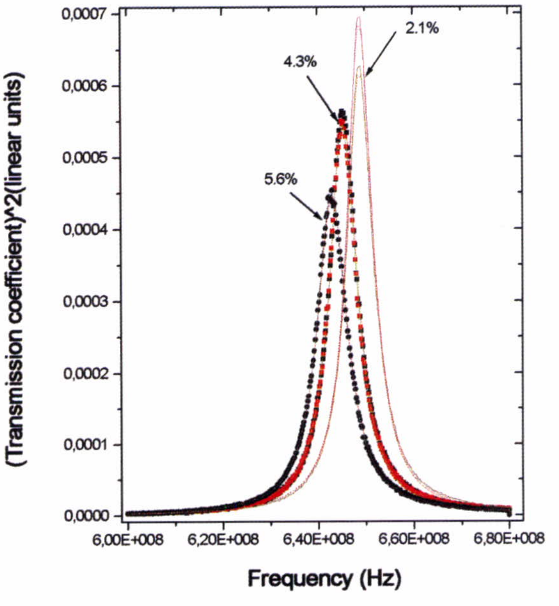
The VE2 system has a product presence-absence sensor, minimum and maximum moisture thresholds with alarms, 4-20 mA outputs for full integration and feedback in the production process: actuator steering, etc.

1.9. PROCESS ATTACK

The attack on the process must be designed to satisfy the needs of the Client. Sensors of different types are foreseen for each need.

2. RESONANCE FREQUENCY MEASUREMENTS WITH MOISTURE CONTENT VARIATION

Graph 3 depicts 3 pairs of measurements conducted on powder masses with different moisture contents by means of the HP875a network analyzer, in an appropriate measurement range.



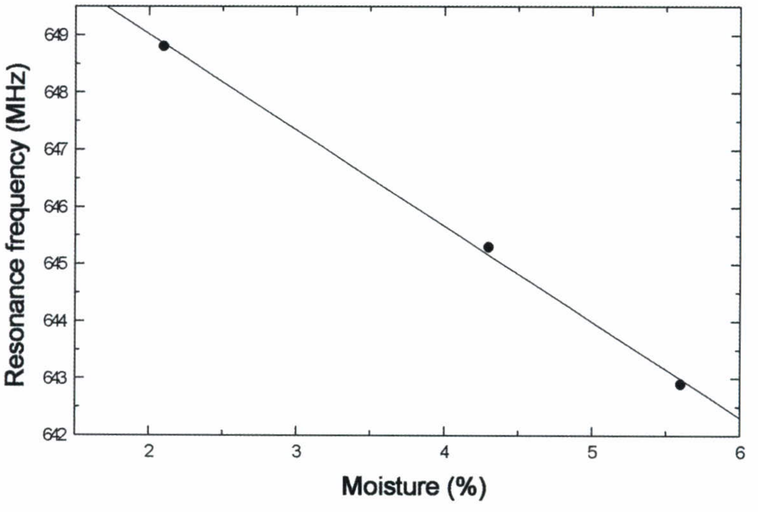
Graph 3. Resonator transmission coefficient with the variation of ceramic powder moisture content.

Graph 3 shows the different attenuation and displacement of the sensor frequency. For a precise evaluation of the response to frequency, the resonance curves have been interpolated by means of the Lorentz functions:

$$y(f) = y_0 + \frac{2A}{\pi} \cdot \frac{w}{4(x - x_0)^2 + w^2}$$

where X_0 is maximum frequency, A total curve area, w fwhm (half amplitude width), y_0 the base line.

- The measurements conducted on samples of white spray-dried powder for porous single-fired wall tile manufacture, with different moisture contents, yielded the response curve displayed in Graph 4.

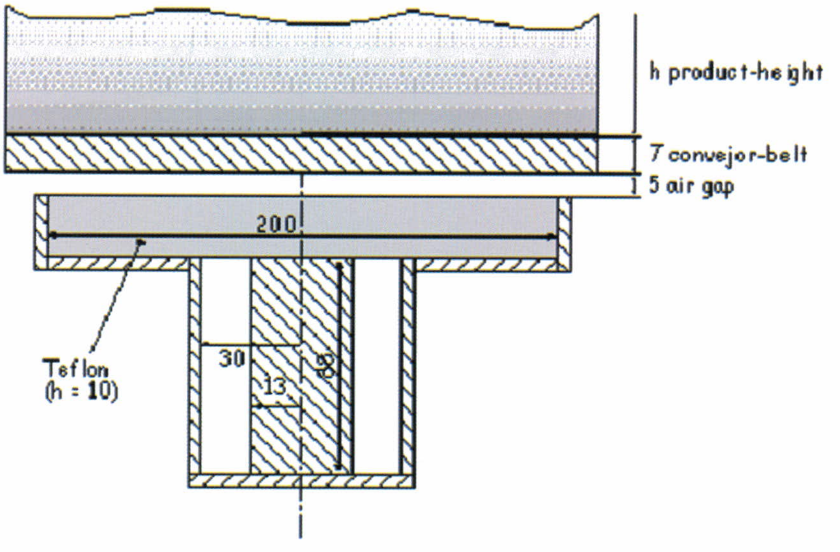


Graph 4. Resonance frequency with the variation of powder moisture content and regression straight line.

3. THE SIMULATION

3.1. MOISTURE EVALUATION BASED ON SENSOR ELECTROMAGNETIC PARAMETERS

Graph 5 shows the geometry used by the HFSS code.



Graph 5. Dimensions in millimetres

The HFSS simulations indicate that the variation of the vacuum resonance frequency (Δf_0) depends on ε_r' and is independent of ε_r'' in the range $\varepsilon_r' = 1 \div 5$. The dependence can be schematically represented by the following polynomial:

$$\varepsilon_r'(\Delta f_0) = 0.00073 \cdot (\Delta f_0)^3 - 0.00497 \cdot (\Delta f_0)^2 + 0.19104 \cdot \Delta f_0 + 1$$

The merit factor Q of the product charged sensor is dependent on ε_r' and ε_r''.

In the range $\varepsilon_r'' = 0.150 \div 0.350$, the dependence can be schematically represented by the following polynomial:

$$\varepsilon_r''(\varepsilon_r', Q) = (-0.0014523 \cdot \varepsilon_r' + 0.0012288) \cdot Q + 0.20734 \cdot \varepsilon_r' + 0.18402$$

Defining parameter $A = \varepsilon_r'' / (\varepsilon_r' - 1)$, independently of the variations of product density, yields the relation that links this parameter to moisture variation.

Evaluation of parameter A:

$$A = \frac{(-0.0014523 \cdot B + 0.0012288) \cdot Q + (0.20734 \cdot B + 0.18402)}{B - 1}$$

with:

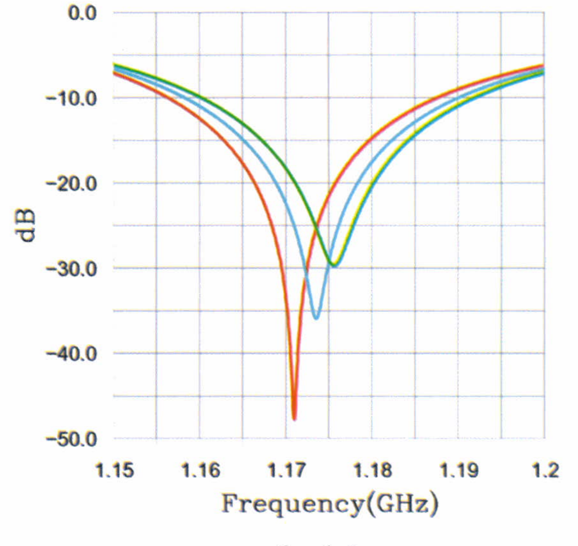
$$B \equiv \varepsilon_r' = 0.00073 \cdot \Delta f_0^3 - 0.00497 \cdot \Delta f_0^2 + 0.19104 \cdot \Delta f_0 + 1$$

Δf_0 : variation of vacuum resonance frequency
Q : merit factor of the product charged sensor

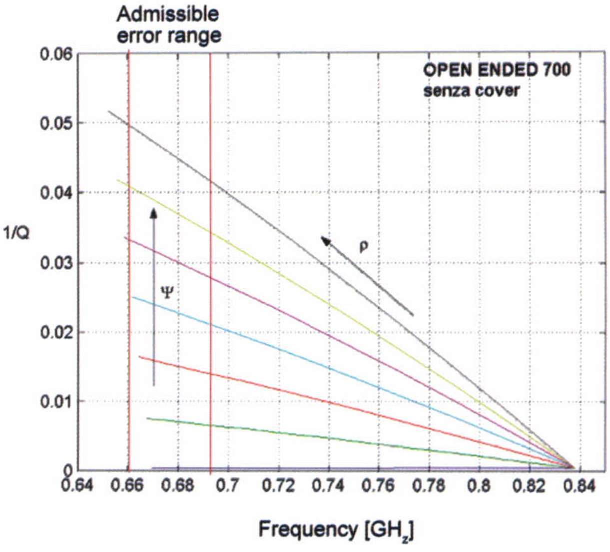
In the following simulations, we have determined the relation that links the variation of ε_r'' to the variation of $|S_{21}|_{max}$, an important relation for the correct evaluation of sensor sensitivity.

4. RESPONSES WITH FREQUENCY, MOISTURE M AND DENSITY VARIATION.

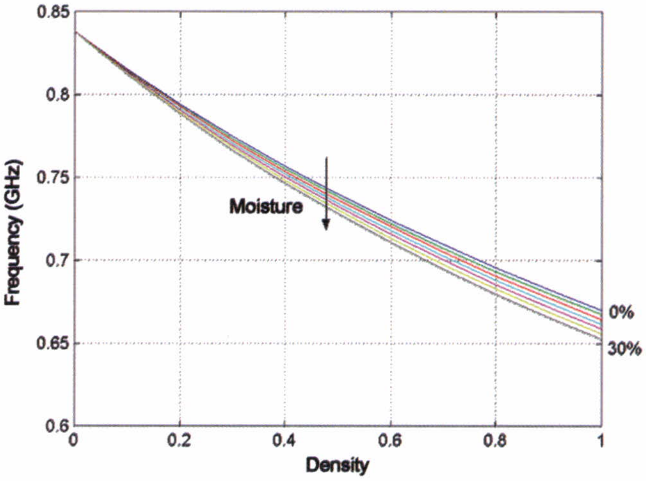
The following figure displays the response with frequency of a reflection sensor. The S11 parameter is measured. The characteristics of this sensor make it particularly appropriate for measuring materials with high water content.



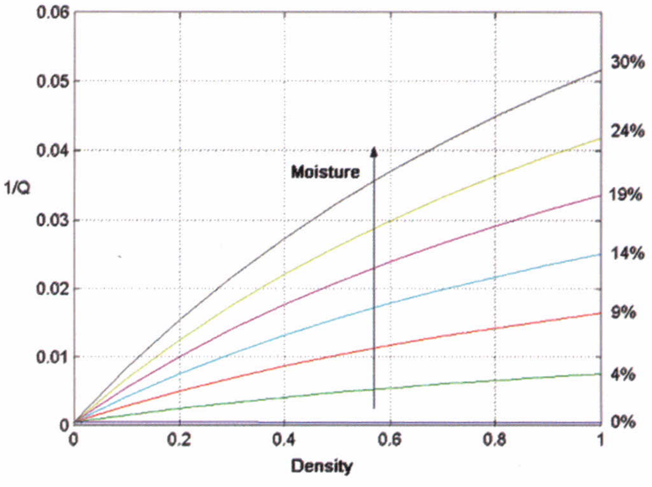
Graph 6 .



Graph 7.



Graph 8.



Graph 9.

5. SOME CONSIDERATIONS IN CONCLUSION

Since water molecules stick to those of the dry material that exhibit lower rotating mobility, the dielectric property of the materials mixed with water differs significantly from what we might expect, taking into account the added liquid water fraction. It is useful to plot the curves of ε'(M) and ε"(M) of the material to verify the different behaviour caused by the water bonds and free water molecules.

The changes in the electromagnetic field in the interaction with the material are proportional to the water concentration (mass/volume), while they vary very little with the variations of dry material density. For correct moisture measurement it is therefore necessary either to measure the density of the material + H_2O, or to seek a function which is - within certain variations - independent of density itself. In addition, it is necessary to monitor product temperature and to divide the product in families or formulations.

In this fashion we can minimize the moisture measurement error caused by swings in density and other process variables.

If we measure the amplitude and electromagnetic field phase at a certain frequency, the search for a function independent of density leads to two functions of the type:

$$A = k1 + k2 \cdot \rho_{H_2O} + k3 \cdot \rho_{dry}$$

$$\phi = h1 + h2\rho_{H_2O} + h3 \cdot \rho_{dry}$$

where k1, k2, k3, h1, h2 and h3 are constants.

The foregoing functions enable deriving moisture M and density ρ in the form:

$$M = \frac{a \cdot \phi + b \cdot A + c}{\phi + d \cdot A + e}$$

$$\rho = \frac{f \cdot \phi + g \cdot A + h}{i}$$

The resolution of this problem is of fundamental importance and must be evaluated for different families of substances.

Our system takes into account and solves all the foregoing observations.

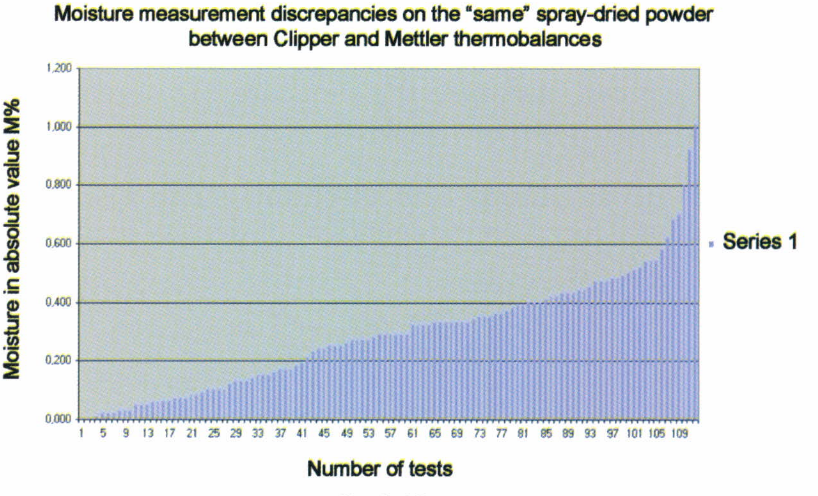
6. REPORT OF THE MEASUREMENTS CONDUCTED IN THE FIELD WITH THE VE2 SYSTEM AT THE RI.WAL GROUP PLANT

As a result of the experience accumulated during the on-line measurement sessions in the month of March, we have made a hopper that allows the spray-dried powder to stop inside it for some seconds. In the month of April, 120 powder samples were taken from spray dryer ATM65. The H_2O percentage was measured with two thermobalances: Clipper and Mettler.

Graph 10 displays the differences (deviations) between the moisture readout on the same sample of carefully mixed powder, by the two thermobalances, expressed as slowly increasing percentages of water.

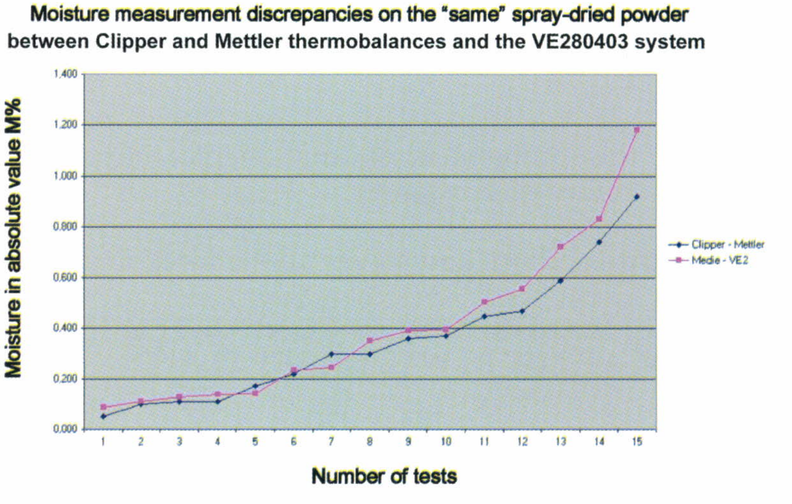
Examination of the graph shows that:

36 measurements display a reciprocal divergence below 0.2%;
39 measurements display a reciprocal divergence between 0.2% and 0.4%;
27 measurements display a reciprocal divergence between 0.4% and 0.6%;
5 measurements display a reciprocal divergence between 0.6% and 0.8%;
2 measurements display a reciprocal divergence between 0.8% and 1%;
1 measurement displays a reciprocal divergence > 1%



Graph 10.

Graph 11 shows the number of moisture measurements, each on the same sample of powder, with the increasing deviations - expressed as a percentage of water - between the mean values of the two thermobalances ((M%_Clipper + M%_Mettler)/2) and the VE2 system.



Graph 11.

The graph shows that the behaviour of the deviations faithfully follows the behaviour in graph 10 of the deviations between the two thermobalances.

The test on the good operation of the VE2 system is in line with the discrepancies: the two dissimilar measurements of 0.92% and 1.18% respectively between the two thermobalances (Clipper-Mettler: error of the thermobalances) and between the Mean Values and the VE2 system (Mean-VE2) also lie within the maximum deviation found.

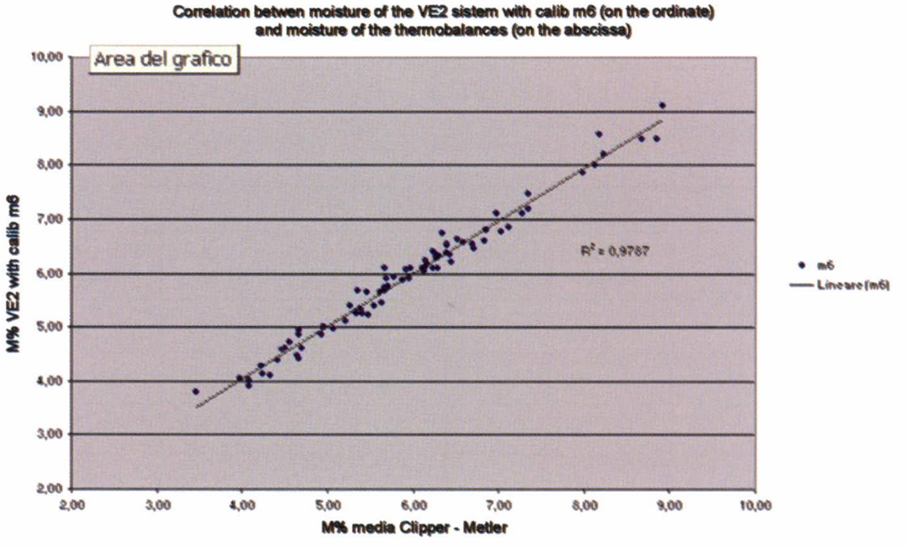
On-line measurements "GOOD VE OPERATION" of 28 April 2003						
M% Clipper	**M% Mettler**	**Clipper_Mettler measurements**	**M% VE2**	**time**	**Clipper - Mettler**	**Mean - VE2**
4,720	4,610	4,665	4,803	12.02.07	0,110	0,138
6,050	5,130	5,590	5,450	12.22.37	0,920	0,140
6,130	5,660	5,895	6,138	12.56.13	0,470	0,243
4,880	4,580	4,730	5,080	14.39.06	0,300	0,350
6,050	6,000	6,025	6,260	14.53.49	0,050	0,235
6,650	6,200	6,425	7,606	15.03.25	0,450	1,181
6,900	6,800	6,850	6,978	15.08.14	0,100	0,128
4,970	5,080	5,025	5,110	15.12.22	0,110	0,085
5,660	5,490	5,575	5,970	15.22.00	0,170	0,395
5,690	5,100	5,395	5,900	15.32.00	0,590	0,505
7,000	7,220	7,110	7,000	15.47.00	0,220	0,110
6,990	6,250	6,620	7,450	15.55.00	0,740	0,830
5,900	5,600	5,750	5,360	16.17.37	0,300	0,390
5,580	5,220	5,400	6,120	16.23.02	0,360	0,720
4,380	4,010	4,195	4,750	16.34.00	0,370	0,555

The calibration curve was then calculated of the VE2 microwave system for moisture by "linear multiple regression". The graph in Figure 3 displays the correlation between the moisture measurements of the VE2 system and those of the two thermobalances. The R^2 coefficient (0.9787) indicates the degree of identity of the two measurements: a value of 1 would indicate the same measurement values.

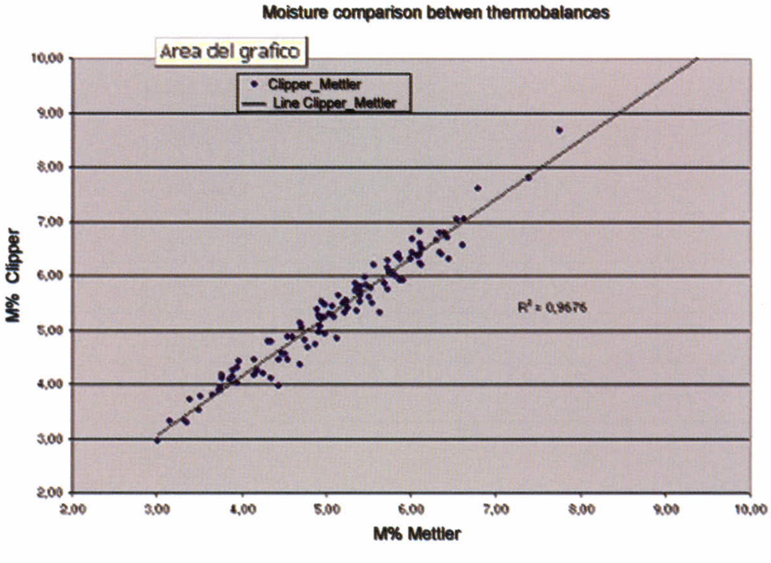
The graph in 13 displays the correlation between the Clipper and the Mettler moisture measurements. It can be observed that the correlation of the graph 12 in is better than in graph 13 (R^2=0.9676): this means that <u>if the real value of the moisture is selected as the mean value of the two thermobalances</u>, the VE2 approaches this value with greater accuracy than the two thermobalances between each other, despite them having the same measuring method (gravimetric difference).

The result observed in graphs 12 and 13 - optimum in itself - is in any case in line with that of the VE2 measuring method, a method that enables reducing the measurement error by increasing the number of points introduced in the calibration.

By still further increasing the number of points, a moisture measurement would be obtained whose error would be below the error of the thermobalances (0.25%).



Graph 12.



Graph 13.

Accurate automatic control of moisture content and density is required in real time in many industrial processes, which provides benefits in terms of product quality and economic savings.

In many cases, the use of microwave technology is the only possible solution; it is necessary, however, to fine tune innovative sensors, which are steerable by simple and reliable electronics.

The VE2 system is equipped with a completely automatic system for powder charge/discharge and sensor-hopper cleaning.

Companies that are able to take advantage of microwave technology will see their operating costs drop rapidly, as a result of the wide use that RF technology will have in the immediate future.