ON-LINE SLIP DENSITY AND VISCOSITY MEASUREMENT

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ABSTRACT

In ceramic tile production, milling is a one of the most important processes, as it determines the characteristics of the slip suspension to be spray dried in the spray-drier.

In the last few years, several studies have been presented on how to improve the productivity and controllability of milling, as the slip must have different specific properties depending on the kind of production (porcelain tile, single-fired, etc.) to be implemented. For example, the porcelain tile body must be ground much more finely than the white single-fired tile body.

The increasing need for flexibility has imposed, as a consequence, the requirement for an improvement of on-line control techniques, also because of the high inertia of the phenomena involved in the milling process. This is important in particular for continuous milling, in which the process variables and the slip output characteristics should be kept constantly under control. In the ceramic tile industry, density, viscosity and residue are normally measured manually every 2-3 hours in order to verify the quality of the produced slip.

Therefore, the possibility of measuring these parameters on-line continuously seems to be of great relevance for control purposes.

The aim of this paper is to determine optimum on-line measurement methods for slip density and viscosity. In particular, different measurement methods are considered, outlining advantages, disadvantages and different possible installation configurations for each of them.

Some of these methods have been also analyzed in laboratory tests, in such a way as to perform proper calibration procedures on dedicated test benches. Finally, some on-line tests have also been approached and results are presented.

1. INTRODUCTION

In the last few years the ceramic tile industry has faced a strong increased competition, which has required improving the automation of the production processes with a higher flexibility and a required decrease of production wastes. The quality and durability of the produced tiles depend on various aspects of the adopted production cycles, such as raw materials, regulation of production lines, kind of production process, etc. All these aspects must be finely tuned in order to achieve an optimal compromise between costs, quality and flexibility.

Among the different processes, milling is one of the most important processes, as it determines the characteristics of the slip suspension to be spray dried in the spray-drier. In particular, continuous milling is becoming an ever more widespread technique, as it allows eliminating downtime associated with mill charging and discharging and increasing the efficiency of the grinding process. However, many problems could be connected with the use of continuous mills, such as formation of large conglomerates of clay and milling balls or the requirement of greater process control.

The main parameters which can affect density and viscosity in a process of continuous grinding are mainly linked to the amount and characteristics of the components at the mill inlet [1].

With regard to the weighing and batching of raw materials, water and deflocculant, these operations are usually effected by highly reliable micro-processor controlled systems, which guarantee the constancy of the inlet flows. This leads to good consistency of the slip characteristics.

However, there are a series of factors which are more difficult to control, and which can create fluctuations in the density and viscosity values. Firstly, there is the humidity of the raw materials, which is not easy to monitor in a constant way. Secondly, there are the chemical and physical characteristics of the water, which can have a significant influence on the rheology of the slip. This is relevant in particular when recycled water is used, which has undergone clarification-flocculation treatment and consequently has a high amount of ionic substances, which are not always constant in time and strongly affect the final viscosity. The control of density and viscosity is important for the subsequent processes: viscosity can affect the discharge and sieving of the slip and is also related to the granule size of the spray-dried powder obtained with the spray drying process. Density is directly related to the slip water content, which determines the amount of water to be evaporated by the spray drier, i.e. its operating temperature to achieve a constant value of humidity at the outlet.

Recently several studies (e.g. [2], [3]) have been presented on how to improve productivity and controllability of milling, with a view to achieving optimum characteristics for different types of products (porcelain tile, single-fire, etc.). It has been shown that it is possible to regulate density, viscosity and particle size of the produced slip by adjusting solids content, deflocculant content and milling process parameters.

It is worth noting that the influence of these parameters has quite slow inertias and time scales. Therefore, the increasing need for flexibility has as a result required an improvement of the techniques of on line control. At the moment in the ceramic

industry, density and viscosity are manually measured every 2 hours and possible deviations from the optimal values (derived from experience of years) are manually corrected by the operator.

The present work aims at the development and on-line testing of measurement systems for slip viscosity and density. In the first phase, a laboratory study has been performed to determine static calibrations and accuracy. In addition, a study of the rheological features of the investigated slips has been conducted to obtain useful indications for an optimal installation of the transducers and for optimization of the plant at the exit of the mills. Nevertheless, it has been necessary to solve inherent installation problems (deposit along the measuring line, presence of air in the circuit, etc.) to get accurate and repeatable results. On-line tests were then conducted in the plant of COOPERATIVA CERAMICA D'IMOLA in Borgo Tossignano (Imola), which showed the applicability of such sensors at the exit of a continuous mill and thus the effectiveness of such technique for a subsequent control phase.

2. THE MEASUREMENT SYSTEMS AND THE TEST BENCH

At the beginning of this work, a critical review of all density and viscosity transducers was performed to select the sensors having the following features:

- Facility of on-line installation and maintenance;
- Constructive simplicity;
- Intrinsic insensitivity to on-line interfering inputs such as temperature, vibrations, working frequency of the circuit;
- Measurement range (10-120 s Ford #4 cup for viscosity and 1200-1900 kg/m^3 for density);
- Accuracy of 1% fs;
- Low cost.

The transducers selected have been the following:

- Time of fall viscometer;
- Tuning fork visco-densitometer;
- Tuning tube densitometer.

The tuning fork visco-densitometer [4] (shown in figure 1) is based on the principle (shown in figure 2) that the resonant frequency of an element depends on the density of the fluid in which it is immersed. This is presumably because the fluid is dragged with the vibrating element changing the effective mass and thus the resonance frequency. The viscosity of the fluid applies a damping force to the system. The Q (quality factor) of the resonance therefore decreases with increasing viscosity. A tuning fork design is used because it is immune to external vibration sources. The tuning fork is excited by a driving piezoelectric element and the resulting motion of the tuning fork is sensed by a pick-up element. The voltage applied to the driving piezo is proportional to the stress applied to the tuning fork. The electronic circuit applies a phase shift to the signal from the pick up piezo and amplifies it to a constant peak to peak level. This voltage is then applied to the driver piezo in such a way that

only further deviations will be observed. Depending on the phase shift applied it is possible to vibrate the tuning fork on resonating or to either side of the resonance.

The working equations are:

$$Q = \frac{1}{\left[\left(\frac{\tau_A}{\tau_B}\right)^{0.5} - \left(\frac{\tau_B}{\tau_A}\right)^{0.5}\right]} \qquad \mu = V * \left(V_0 + \frac{V_1}{Q^2} + \frac{V_2}{Q^4}\right) \qquad \rho = K_0 + K_1 * \tau_B + K_2 * \tau_B{}^2$$

where Q is the quality factor, τ is the time period, v_i and k_i are factory calibration coefficients. The tines of the tuning fork are prismatic with segments of a circle as cross-section.

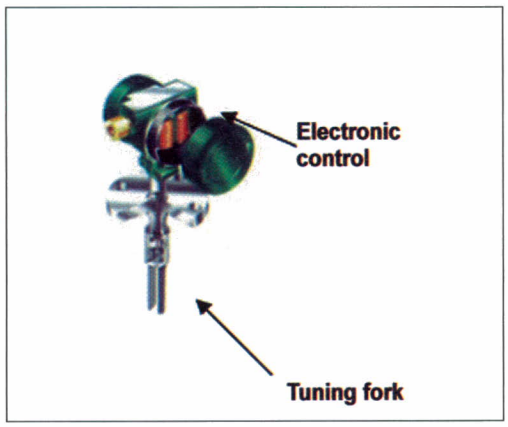

Figure 1. Tuning fork visco-densitometer

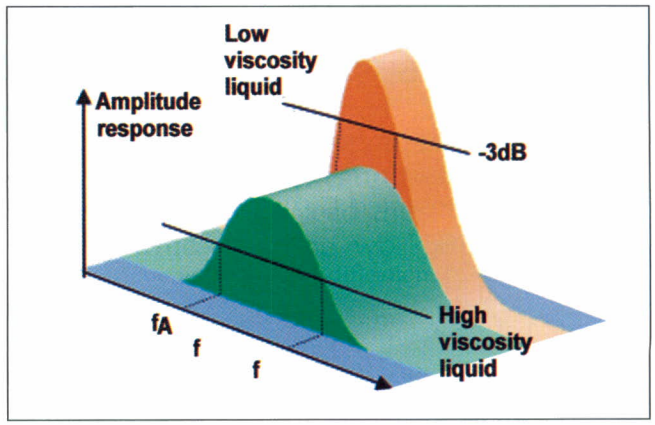

Figure 2. Working principle

The steel base on which they are mounted has a diameter of 17.5 mm and the proposed housing would have an inner diameter of 26 mm.

The working principle of the tube (shown in figure 3) densitometer is the same as that of the tuning fork, but in this case the vibrating element is a straight tube. The tube has a length of 1 m and an inner diameter of 2.54 cm. Also in this case the element is maintained at its natural frequency and every variation of density causes a frequency shift of resonance peak.

The basic working principle of the piston viscometer is based on the linear relation between the time of fall of a piston immersed in the investigated fluid and the viscosity of the same fluid. The transducer is composed of a measuring chamber, automatically filled with the fluid, in which the piston is mounted.

At the time instant T1, the pneumatic control system releases the piston to fall; then the proximity sensor, mounted at the end point of the piston run, saves the instant T2 and electronic processing system determines the time of fall T.O.F as T2-T1. Using different piston sizes it is possible to cover viscosity ranges from 0 to 2000 cP.

To test the three transducers a workbench (shown in figure 4) has been designed to optimize the installation of the sensors and to obtain the best accuracies.

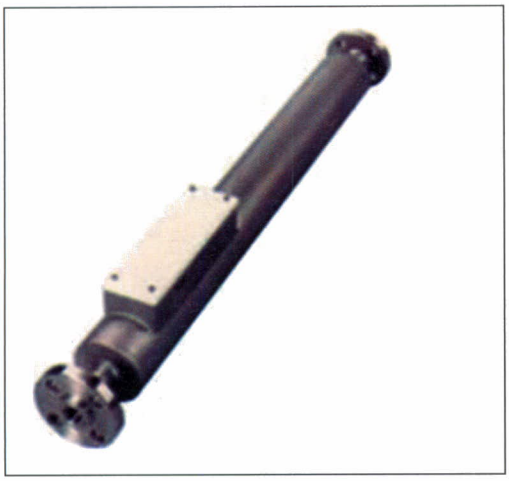

Figure 3. Tube densitometer

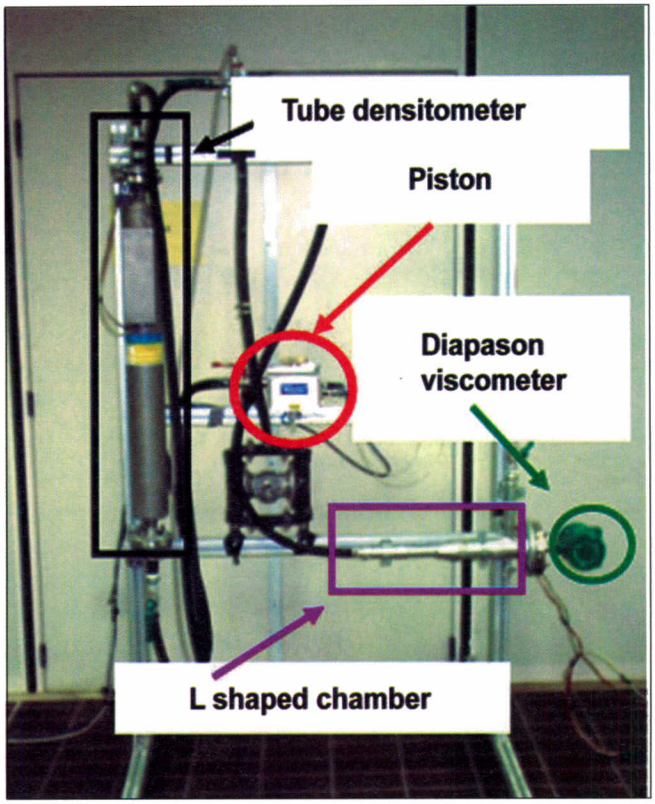

Figure 4. Test workbench

The workbench has been designed with the main aim of having a structure suitable for both laboratory calibration and on-line installation. A membrane pneumatic pump was installed to obtain good circulation of the slip within the pipeline of the bench.

It is important to stress that for these transducers the installation mode is crucial to achieve the best performance.

The tuning fork has been installed on an L-shaped measuring chamber; this working mode allows creating an optimal measurement volume with no influence on the sensing element and measuring density and viscosity also for fluids with not-Newtonian features.

The laboratory tests showed that the tuning fork is very sensitive to the wave pressure due to the membrane pump, so the tuning fork has been mounted with the tines parallel to the flow and in this position it is possible to reduce the influence of the pump. The dimension of the L-shaped chamber was specifically designed to create a sufficient flow rate to allow an optimal slip recycle and a good dynamic response, not depending on the installation conditions but depending only on the sensitivity of the instrument to viscosity and density variations. In order to avoid the presence of air and obtain a perfect filling of the measuring chambers, the transducers were mounted in the pipeline with a hydrostatic head. In particular both the tube densitometer and the L-shaped chamber of the tuning fork were fed with the slip from the bottom up to the top. This allows improving accuracies, as the presence of air in the measuring volumes causes a great dispersion of the measured values.

Another important problem is connected to the slip accumulations inside the instruments. A valve was therefore installed on the lower part of the L-shaped chambers to monitor the presence of deposit and to eliminate it, while the tube has been mounted vertically.

Concerning data acquisition (figure 5), all the transducers are clearly able to generate standard digital outputs. The piston viscometer is controlled by an electronic

digital converter that allows acquiring the data by RS-485 serial protocol. The tube densitometer and the diapason visco-densitometer are controlled by the same analog-digital converter capable of acquiring data both by RS-232 serial protocol and RS-485.

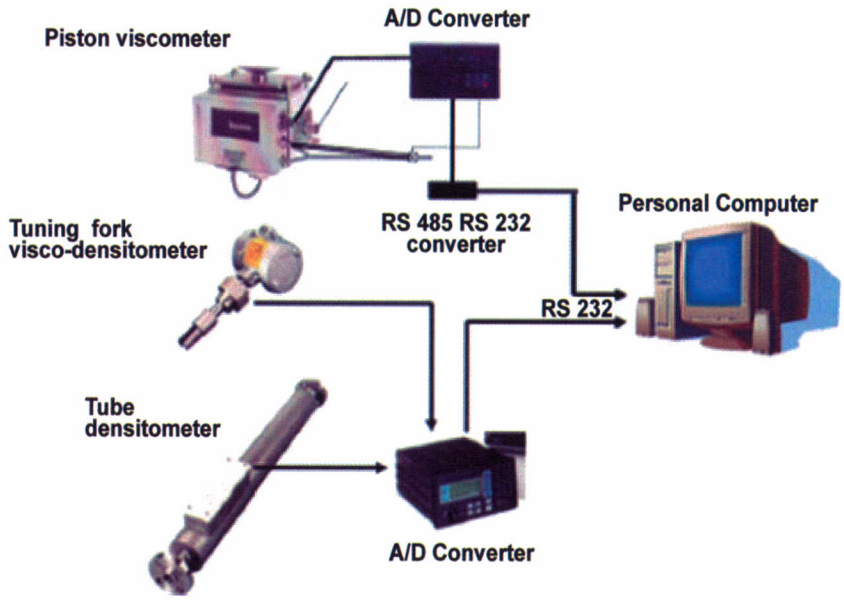

Figure 5. Network data acquisition

3. THE LABORATORY TESTS

The on-line tests on the continuous mill have been preceded by a preliminary laboratory study, which has furnished useful indications on the performances of the transducers, on the characteristics of the fluid and on the optimal conditions for stable and repeatable measurements.

Particularly tests have been conducted on:

- Rheological features of the slip;
- Determination of the accuracies of the transducers in the used bench;
- Interfering and modifying inputs for the investigated transducers and eventually present in the production line.

The slip is evidently a non-Newtonian fluid, whose viscosity is strongly influenced by working shear rate. In addition the shear rate-viscosity rheological curves could easily vary changing components concentration or other parameters in the mill.

On the other hand, the shear rate to which the fluid is subject at the exit of the mill and in the outlet line varies on passing from small inlets to pipes of great diameter. This variation of viscosity along the line is reflected in the efficiency of the plant. For instance, a slip with strongly dilatant behaviour (viscosity increases with shear rate) would create great problems in passing into a small inlet, causing occlusion. Otherwise a slip with pseudo-plastic behaviour would easy create crusts in zones subject to low flows.

Naturally it is important to also consider the thixotropic feature of the slip, which becomes evident if this is left for several minutes in a zone not subject to flow. An increase of the viscosity can be immediately noticed (partly due to the evaporation of the most volatile part) up to the complete solidification.

The rheological laboratory tests have been conducted using a rotational viscometer to define the shear rate-viscosity curves for different percentages of dilution. An example of the result is shown in figure 6, where the curves for one type of slip ("white slip") are reported.

It is possible to note a pseudo-plastic behaviour of the slip. The increase of dilution generates a dispersion of the solid particles and the curves tend to flatten assuming an almost Newtonian behaviour. Naturally the objective of the wet grinding process is to increase the solid content in the slip, in order to thus reduce the energy required to evaporate the water in the spry drying. In addition the growing dilution increases the possibility of disaggregation of solid parts within the solution (with high viscosity) from the diluent (water), which is characterized by a lower viscosity, with the consequent risk of deposit in the different zones of the plant.

These considerations, even though reported only for a type of slip (which is the one later tested in the line), underline the importance of the rheological aspects on the production of the slip in continuous mills, both for optimization of the plant and to obtain a better configuration of the installation for a possible viscosity and density measurement system. The tested sensors work at a high shear-rate (over 10^4 s^{-1}), very close to the one of the Ford cup #4 employed at the moment in industry for manual control. It is therefore important to note that the viscosity variation observed at different dilution levels could be significantly smaller than those actually achieved at low shear rates, which is of interest for example for slow mill discharging by large diameter pipes. This information must be taken into account for control purposes and can be easily recovered by similar characterisations.

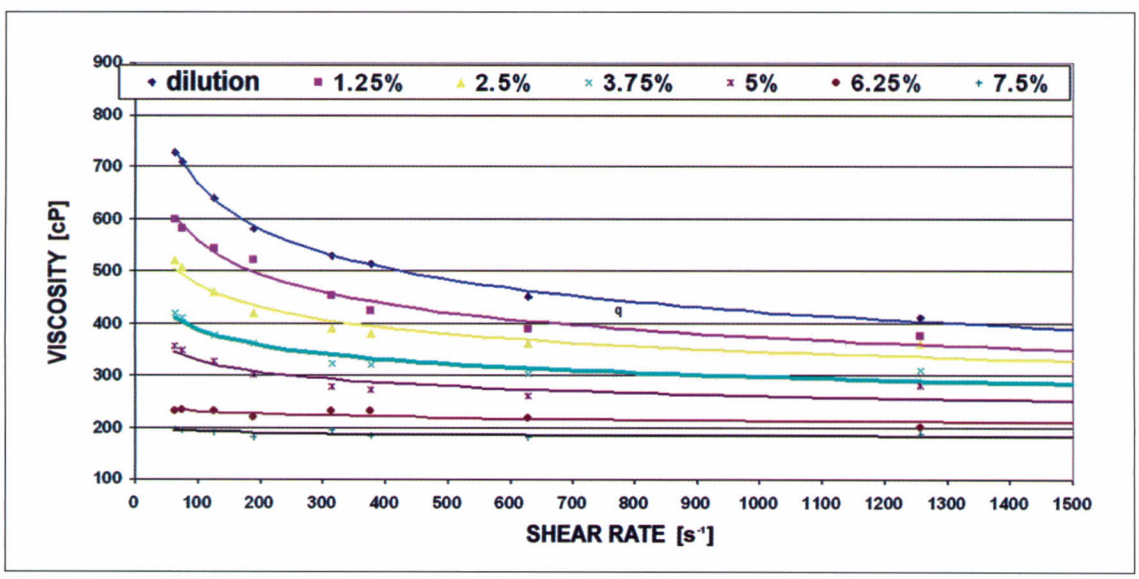

Figure 6. Rheological behaviour of "white slip"

The sensor calibrations have been conducted under conditions very similar to those present in the production line, in such a way as to create calibration data to

correct the results of the on-line tests. Calibration is a necessary step in the use of these instruments, as they perform an indirect measurement based on the observation of different phenomena (vibrations, time of fall, etc.). Therefore the raw data always have to be post-processed for correction by applying the calibration coefficients. The calibrations were performed by comparison with the mostly used laboratory instruments for viscosity and density, i.e. the Ford Cup #4 and the weight-based pycnometer respectively.

A very important variable kept under control was the slip temperature, which can significantly vary at the exit of the mills. In our test the temperature oscillated between 56 and 58 °C. It was therefore sufficient to calibrate the transducers to an average temperature of the liquid of 57°C, variations in a temperature interval of 2°C not being relevant for the industrial process. However, the temperature variations at the mill exit could be higher, and in such cases it would be necessary to define different temperature intervals in which the calibration would have to be performed. In most of the cases it will be enough to apply constant corrective coefficients.

The tests were performed in a narrow range of viscosity and density around the values of interest. In fact it was noticed that for strongly non-Newtonian fluids, the accuracies of the transducers depended highly on the measurement range, worsening with its increase (with significant dispersions in the readings). Calibration performed in larger ranges may lead to the exhibition of non-linear behaviours with higher resulting uncertainties.

The calibration lines for the tested viscosity and density sensors are shown in figures 7, 8, 9 and 10. The achieved accuracies, expressed as three times the standard deviation with respect to the calibration lines, are the following:

- TUBE DENSITOMETER: ± 2.19 kg/m^3;
- DIAPASON FOR DENSITY MEASUREMENT: ± 4.01 kg/m^3;
- DIAPASON FOR VISCOSITY MEASUREMENT: ± 0.15 °E;
- PISTON VISCOMETER: ± 0.1 °E;

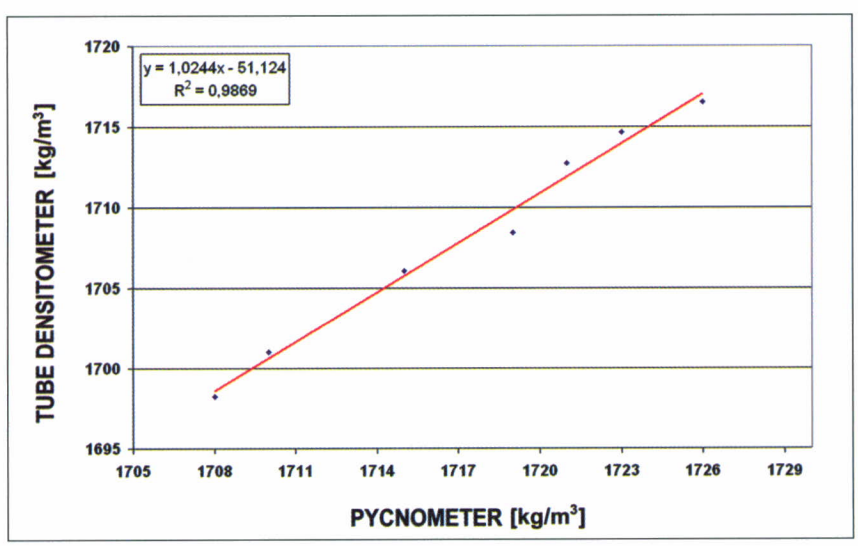

Figure 7. Static calibration of the tube densitometer

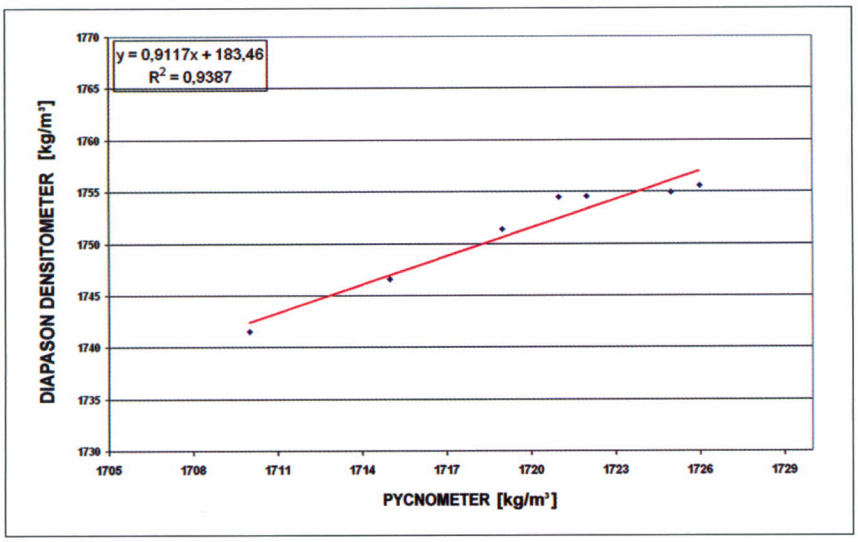

Figure 8. Static calibration of the diapason densitometer

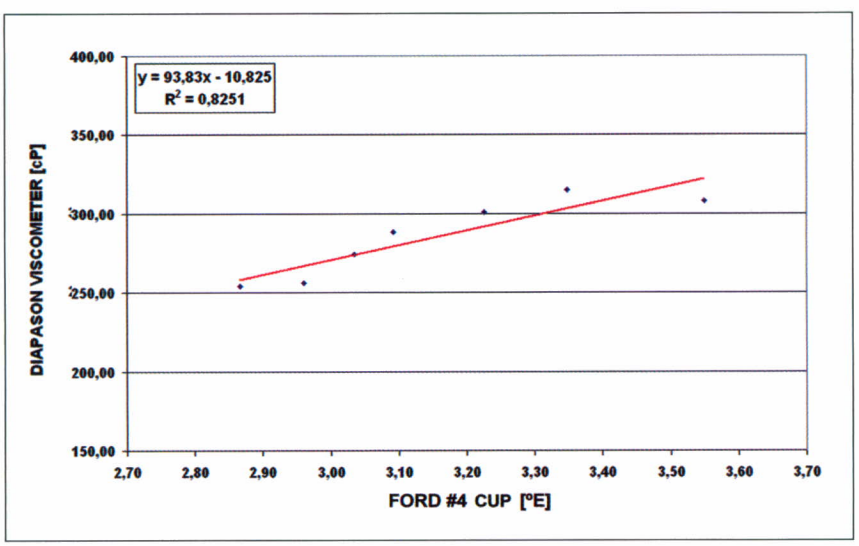

Figure 9. Static calibration of the diapason viscometer

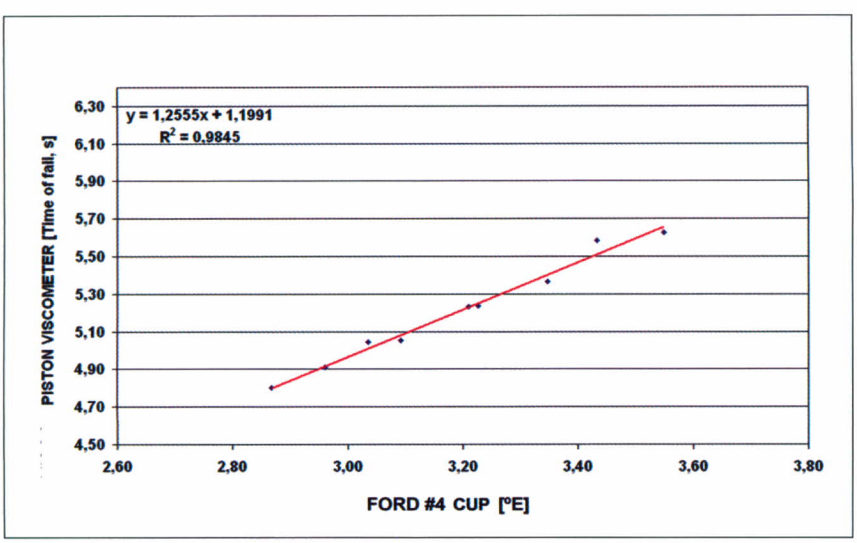

Figure 10. Static calibration of the piston viscometer

The dynamic performances of the instruments in the tested installation were also evaluated in the laboratory experiments. In particular, a step input was generated by a sudden addition of water in the slip and the outputs were measured. It was shown that, while the response of the tube and of the diapason were almost immediate (less than 1 s of delay), the piston viscometer showed a first order behaviour with a time constant of about 40 s in the tested installation. This is due to the fact that the recycle in the chamber is not complete at every measurement; however this value is completely satisfactory for the application at the exit of the mill, where the phenomena have a longer time scale.

4. ON-LINE TESTS

The main aim of this work has consisted in testing the effective use of the selected transducers in the milling process. The possibility of applying the transducers to on-line production depends mainly on these factors:

1) sufficient accuracy and repeatability in measuring every viscosity and density variation from the set-point;

2) fast dynamic response allowing following implementation of a control system.

For the previous reasons it was decided to test the transducer in a continuous mill in order to verify the applicability of these sensors. The production cycle of the tested continuous mill lasted about 20-22 hours; during this period the mill feeds a tank where the slip suspension is stored.

In order to quickly test the transducers with no interferences on the production cycle, the measuring workbench was installed close to the exit of the tube carrying the slip suspension from the mill to the tank (figure 11).

The slip flow rate passing into the tube was not constant, so, in order to avoid having air in the measuring pipelines, the inlet tube of the membrane pump was put in a bucket near a cascade generated by the overflow of slip suspension coming from the alimentation tube. The inlet tube of the membrane pump has been positioned just under the slip surface to avoid suction at a stagnation point of the bucket. This particular set-up has resulted indispensable in order to obtain better accuracies and dynamic response, depending only by the performances of the transducers and not on the installation.

Further to density and viscosity, the slip temperature was also measured, as during the milling process it slowly varied from 56 to 58 °C. The measured values of density and viscosity were periodically compared with results manually measured by a pycnometer for density and by a Ford cup #4 for viscosity. Results are reported in figures 12 and 13. The data was acquired for a total time of 530 minutes (about 9 hours) to monitor all the variation of density, viscosity and temperature during the whole milling process. For the tube and the visco-densitometer the data was acquired sampling every two seconds, while for the piston viscometer, measurement frequency was 20 s and an average value was acquired every minute. The lower sampling frequency of the piston viscometer did not generate losses of information because, as shown in the results, viscosity just as density oscillated with a time period of about 80-90 minutes.

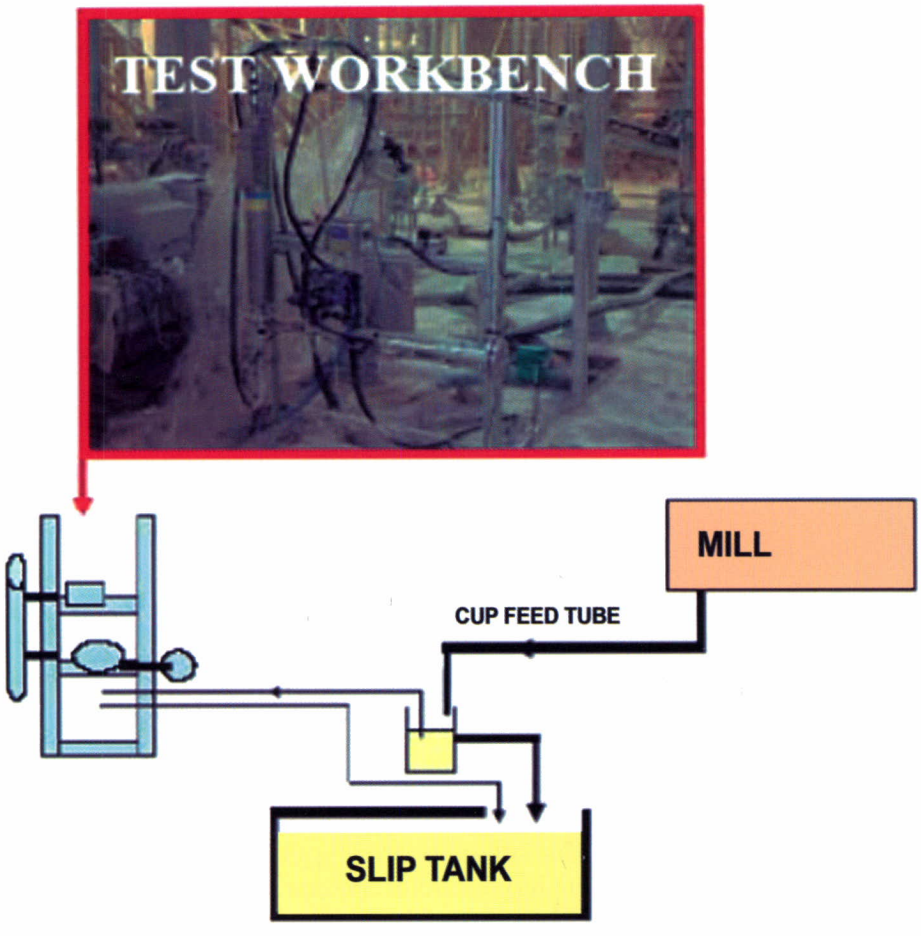

Figure 11. Test workbench installed in the Cooperativa Ceramica d'Imola plant

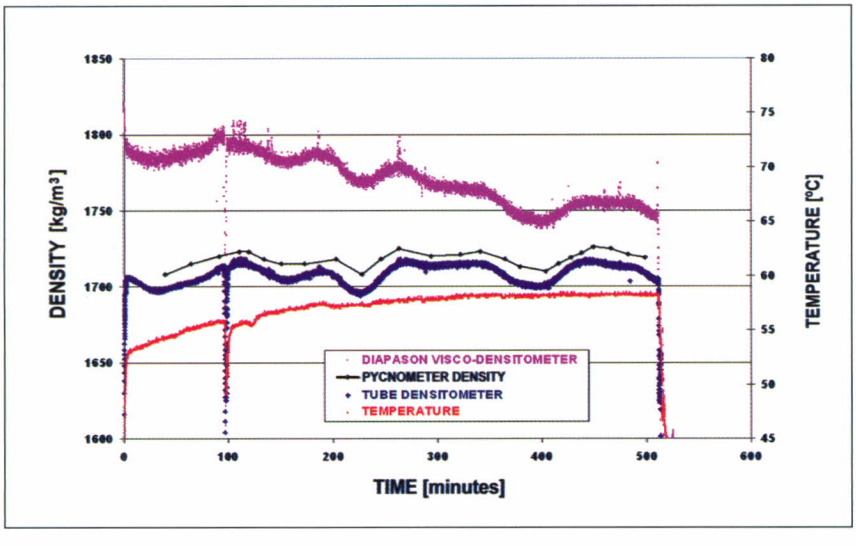

Figure 12. On-line density acquisition (diapason and tube densitometer)

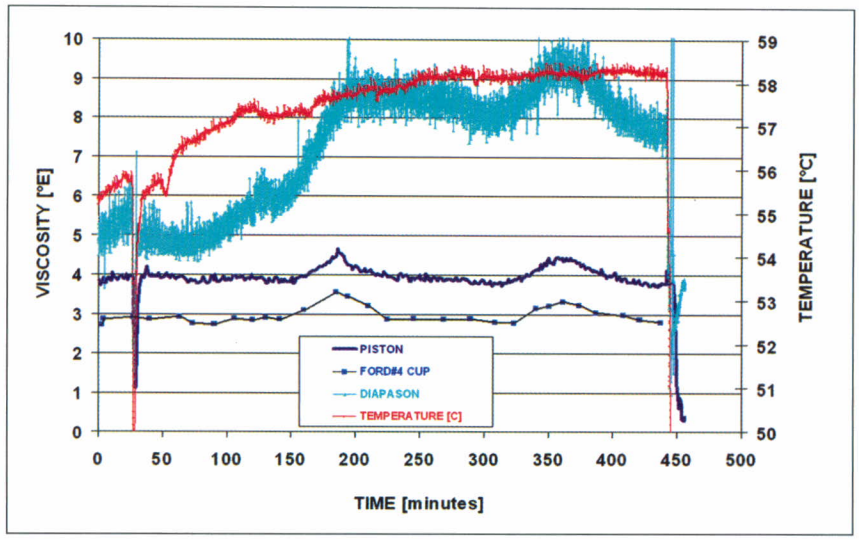

Figure 13. On-line viscosity acquisition (diapason and piston viscometer)

The measured slip parameters varied with a slow oscillation around an average value of 1718 kg/m^3 for density and 3.12°E for viscosity.

The data reported are not corrected with the calibration coefficients achieved during the laboratory experiments. For the tube densitometer and the piston viscometer this fact generates the offset with respect to the values measured by the Ford cup and the pycnometer, which seems to be constant with time and temperature.

Figures 14 and 15 report the results corrected with the calibration coefficients, showing a satisfactory agreement with the reference measurements performed off the line.

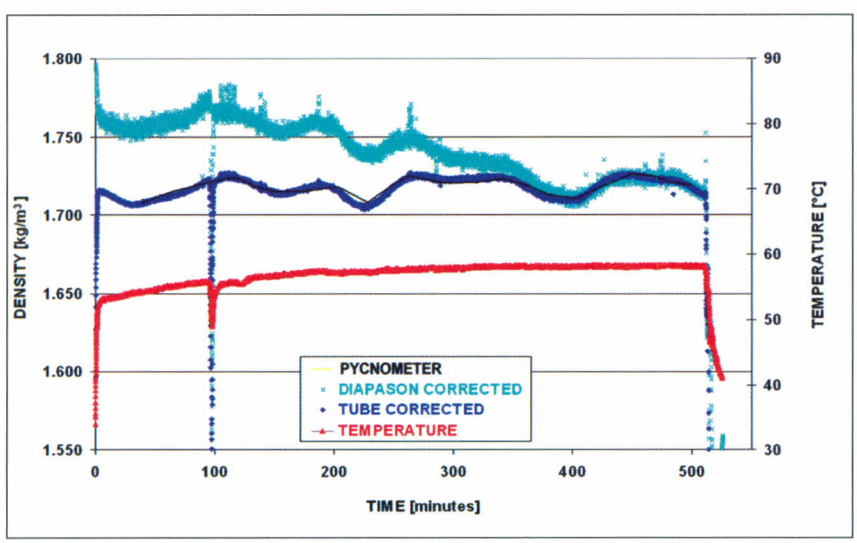

Figure 14. Density corrected with the static calibration coefficients (tube and diapason densitometer)

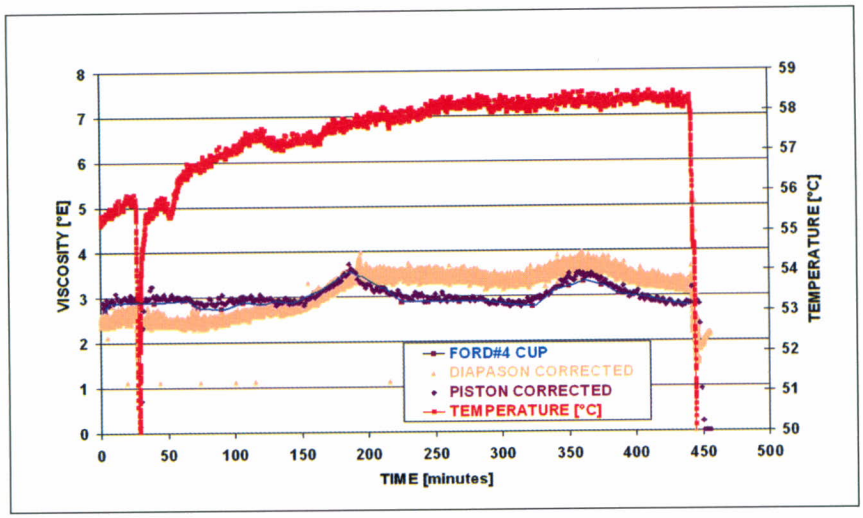

Figure 15. Viscosity corrected with the static calibration coefficients(diapason and piston viscometers)

It is evident how the tube densitometer and the piston viscometer follow every variation of density and viscosity during the process with the same accuracy achieved during the laboratory calibration. This is confirmed by the fast transition recorded at about 100 minutes, which was due to a cleaning operation of the mill. The cleaning of the mill was obtained with an important addition of water that caused a fast decrease in temperature and density, correctly measured by the implemented transducers.

The diapason visco-densitometer also showed a good dynamic response (all the variations are correctly followed), but also showed a drift in time, decreasing for density and increasing for viscosity. This drift could be due to a very high sensitivity to temperature (which could work as an interfering input) or to slip deposit inside the diapason chamber with an amount increasing in time. Therefore, since the possibility of using a single compact sensor for density and viscosity could be very interesting, it seems that the installation should be further improved.

5. DISCUSSION AND CONCLUSIONS

The research presented in this paper shows that it is possible to accurately measure density and viscosity on-line at the exit of the mill. In particular, three different transducers have been calibrated and tested both in the laboratory and in the line with very satisfactory results both in terms of accuracy and dynamic response. The fluctuations which have been recorded during the experiments both for density and viscosity are inside the ranges that are considered acceptable in ceramic tile production. Nevertheless, in some cases it can be important to monitor such variations. For example, in the production of porcelain stoneware, continuous mills are used to produce a colourless slip which is then added with concentrated coloured stains in order to obtain a coloured body. The control of slip density, together with the reliability of the batching systems, is fundamental to achieving a final slip with an accurate ceramic body / pigment ratio.

6. ACKNOWLEDGMENTS

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continuous mill and to the companies producing the instrumentation for supplying the transducers for the whole duration of the research.

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