NEW TECHNIQUE FOR CONTINUOUS PARTICLE SIZE MEASUREMENT IN THE CERAMIC SUSPENSION MILLING OPERATION

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ABSTRACT

The measuring equipment and method developed in this study enable automatic sampling of concentrated solids suspensions exiting the continuous industrial mills customarily used in the ceramic process, obtaining the major characteristic cut-offs in particle size distribution in industrial practice, as well as evaluation of the state of the sieves arranged at the milled stream output. For this, the concentrated sample is run through a cell irradiated by an X-ray tube while images are taken using a high-resolution matrix sensor. The images obtained are processed using specially developed software that analyses and displays the results during production, thus opening the door to automatic monitoring of milling operations, in line with current digitalisation and automation trends.

1. INTRODUCTION

Milling is a basic operation whose aim is to produce a homogeneous suspension of solids in water (slurry) with a suitable particle size distribution (PSD) for the following manufacturing process, compatible with a high solids content, and of appropriate viscosity to ensure optimal spray drying. In addition, the particle size distribution of the solid contained in the suspension conditions how the ceramic tile will behave during processing (compaction, diffusion, etc.), and defines a large number of the finished tile's characteristics (final size, porosity, etc.). Measuring particle size distribution is relatively complex and expensive, which is why the method traditionally used at industrial level is based on the close relationship between PSD and the amount of coarse particles of solid in a certain material and type of mill. In fact, wet milling mostly reduces coarse particle size, narrowing PSD, so that measuring the residue or "reject" (as the oversize on a screen is colloquially called) means the milling stage can be monitored by simple manual testing. Such a way of operating was sufficient for as long as discontinuous milling technologies were in use, as it was relatively easy to adjust milling time to obtain a certain residue in the end slurry. However, with the widespread implementation of continuous milling, the need has been evidenced for faster and more automatic techniques to continuously determine the residue.

The main difficulty with automatic milling control lies in achieving reliable, continuous measurement of density, viscosity and residue. Nowadays, the continuous industrial metering of suspension density has been successfully solved by using Coriolis effect density meters. On the other hand, measuring viscosity and especially residue still requires applied research to assess the viability of its automation, since today, it continues to be done by manual screening, even though different methods to evaluate particle size are now available [1] [2].

Particle size analysis techniques can be split into two groups, depending on whether they are direct or indirect [3]. On the one hand, indirect methods are based on recording how particles respond to a given physical phenomenon. Such phenomena include gravitational or centrifugal sedimentation, laser light diffraction, particle counting by impedance variations, laser reflectance probes, or sonic spectroscopy, among others [4] [5]. On the other hand, direct methods are based on directly determining particle size, the most widespread techniques being optical and electronic microscopy, image analysis probes, high sensitivity cameras, or X-ray tomography [6]. All the above measurement methods have certain limitations in terms of robustly estimating the size of particles in ceramic slurries in industrial environments, thus rendering them difficult to implement.

The main limitations are the degree of intervention and skill required of the operator to analyse the measurement, the cost of the equipment, length of testing time, complexity of automation, and the influence on estimated particle size played by variables that are naturally altered during the milling process, such as true density, coefficient of absorption, the medium's refractive index, temperature, concentration, viscosity and/or mineralogical composition. Ceramic slurries, specifically, have a high solids concentration, so that a number of current methods require the sample to be diluted considerably, which leads to a false depiction of actual milling behaviour due to sub-sampling [7][8][9][10][11].

In short, no system or technique exists at present that allows automatic and continuous measurement of particle size in concentrated suspensions exiting an industrial ceramic mill. For that reason, this paper describes a new measurement technique capable of overcoming the limitations noted above. The proposed technique is based on industrial X-ray imaging, which, given the supreme capability of X-rays to penetrate into materials, enables relatively large amounts of undiluted samples to be analysed, thus making the samples more accurate and reliable [12]. On the basis of this study and by switching from the discrete, manual measurement currently used in milling control to real-time particle size measurement, it is hoped that closed loops can be implemented to monitor this critical process variable as part of a control management system, in consonance with today's Industry 4.0 trends.

2. OBJECTIVE

The main aim of this study was to develop and validate a new particle size measurement technique based on X-ray imaging that would enable residue monitoring to be automated during the continuous milling of ceramic slurries. To that end, the measurement technique was tested in laboratory conditions inside a radiography bunker, and with the knowledge acquired from those experiments, a working industrial prototype was built.

3. EXPERIMENTAL PROCEDURE

3.1 MATERIALS

To assess the proposed measurement technique and validate the new prototype's operating capabilities, an industrial slurry was used that is currently employed to manufacture a spray-dried powder for porcelain stoneware with 6.4% residue at 45 microns and 2.0% at 63 microns. In addition, slurries with different degrees of milling were prepared in the laboratory from the same raw materials as those used to prepare the suspensions used industrially. To do so, they were milled in a ball mill for various milling times and/or changing the types of balls to produce slurries with different residues. Table 1 shows the residues measured from the 5 prepared slurries on 45 and 63 µm sieves.

Reference	Milling time (min)	Loading	$R_{63\mu m}$ (%)	$R_{45\mu m}$ (%)
1	20	Energetic	0.3	1.7
2	15	Energetic	0.7	3.6
3	10	Energetic	2.9	9.2
4	20	Regular	5.1	13.8
5	10	Regular	10.7	22.5

Table 1. *Residues at 45 and 63 µm from lab-prepared slurries*

3. 2 EXPERIMENTAL APPARATUS

Figure 1 shows the experimental apparatus arranged inside a radiography bunker to assess operation of the proposed measurement technique. The assembly consists of a microfocus X-ray transmission tube (1), a measuring cell which the sample flows through (2), and an X-ray sensitive matrix detector, responsible for generating the image to be processed and analysed (3). For simple assessment of the measuring chain, the slurry was fed through the cell by two syringes connected to its inlet and outlet. The measuring procedure consisted of pumping a certain amount of slurry from the inlet to the outlet syringe and then stopping the flow of slurry before turning on the X-ray tube to generate a high-focus, collimated beam of radiation that passed through the sample in the cell and was detected by the matrix sensor. In the resulting X-ray, the thicker, coarsest solid particles in the sample were detected as darker areas, because they attenuate a greater amount of radiation, while the rest of the slurry was seen as lighter areas.

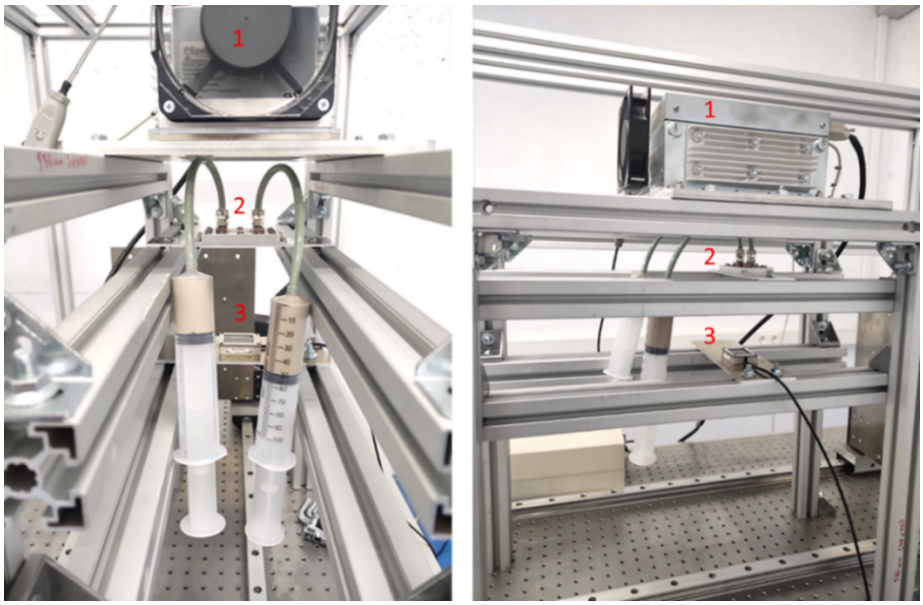

Figure 1. *Front and side view of the experimental X-ray imaging set-up with aluminium profiles inside the radiography bunker. (1) Micro-focus X-ray tube, (2) measuring cell, (3) Radiation-sensitive matrix sensor.*

The set-up was used to assess performance of up to three different matrix sensors. According to their specifications, all of them initially provided a resolution of 20µm/pixel, which, considering the magnification of the detector produced by the tube apparatus, gave an actual resolution of 10 µm/pixel. Two of the sensors evaluated, which are commonly used in dental radiography, had a black plastic casing, while the third was a matrix sensor used in industrial X-ray inspection in a metal casing (Figure 2). The latter and one of the dental X-ray detectors had a gadolinium oxysulphide (GOS or GADOX) scintillator, while the other one had a caesium iodide (CsI) scintillator. The scintillator is the material that transforms the X-rays received into visible light that can be quantified by the photodiode and amplified to produce a scaled electrical signal with respect to the radiation received.

Figure 2. *Plastic-packed matrix sensor (left) and the matrix sensor with aluminium casing and a carbon fibre window (right).*

Figure 3 shows details of the measuring cell through which the slurry to be characterised flows. The chassis is made of two pieces of aluminium that fit one inside the other. The cell windows are made from transparent dental acrylic to facilitate visual inspection and prevent the build-up of dirt. This material is also highly transparent to X-radiation, which is of great importance to ensure a strong signal in the detector. The top acrylic window has threaded lugs where the connectors that guide the slurry from the intake to the return piping are fitted. The same figure also shows details of the industrial matrix sensor anchored to the experimental assembly.

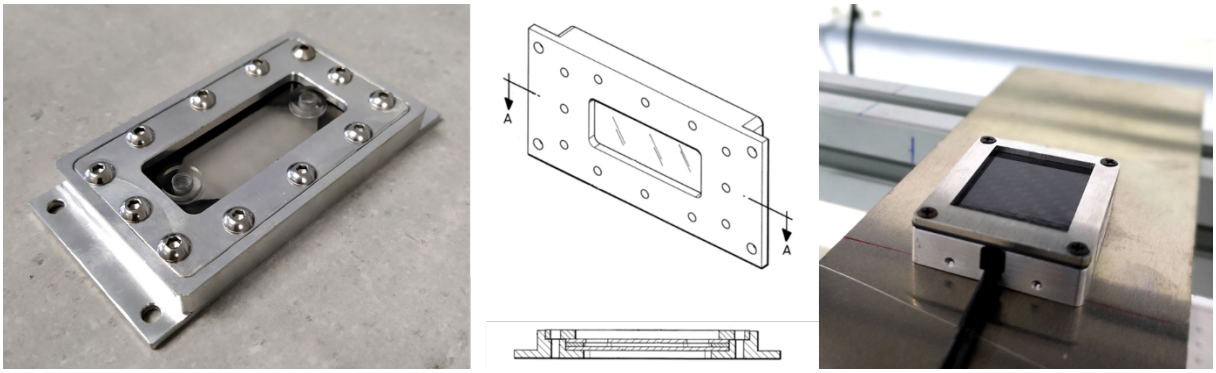

Figure 3. *Slurry measuring cell (left and centre). Matrix sensor (right).*

3.3 IMAGE PROCESSING AND ANALYSIS

It should be noted that the X-rays obtained with the proposed technique are images of which simple viewing provides hardly any information about the properties of the characterised sample. It was therefore necessary to develop an image analysis algorithm to enable quantitative information to be extracted from the X-rays after they had been binarized. Consequently, once the X-ray has been taken, the newly developed software performs a multi-stage process of image analysis to obtain numerical values (number of particles), which it then converts into a percentage of residue with the aid of prior calibration. The process followed by the software is described below and is also outlined in the example in Figure 4:

- Normalisation: Since the image obtained may have light and dark areas and/or gradients that may hinder automatic binarization, it first has to be normalised. To do so, a copy of the highly blurred image is used and by "dividing" it from the original, the

light and dark areas and/or gradients are eliminated, while the ceramic particles to be analysed are preserved.

- Equalization: This enables the pixels in the image to acquire non-integer values by turning it into a 16-bit image within the same range. This allows all images to be compared in identical conditions, regardless of the way they were taken.

- Binarization: Using advanced binarization methods, the 16-bit image is turned into a black and white image, where each of the particles can be recognised and analysed fully automatically. This is the most important step in the entire process and must be performed under objective, robust and systematic criteria, because otherwise the resulting variability would make this new method inconsistent.

- Analysis: In the analysis process, all particles are identified, and those that are below the residue value to be estimated are eliminated. At the same time, particles that form clusters or are of a size that may be distorted and possible bubbles are removed. The value obtained by analysing the image is the number of particles over the considered size (45, 63, 125 µm...). This number of particles is converted into a percentage residue by calibration.

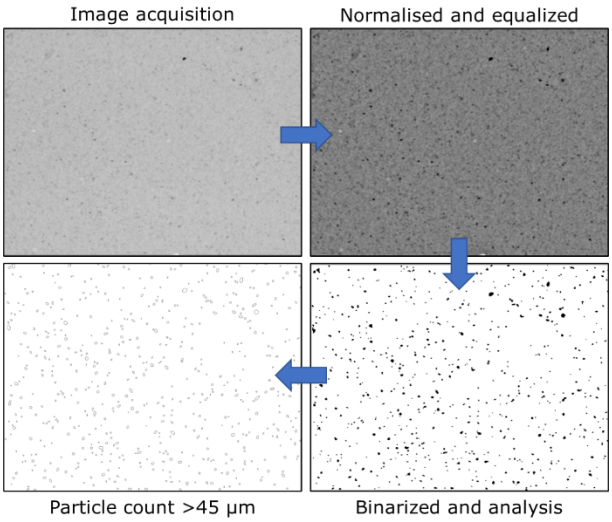

***Figure 4**. Example of how the X-ray image analysis and adjustment algorithm works for particles over 45 µm*

4. RESULTS AND DISCUSSION

4.1 TECNIQUE VALIDATION

Using the slurry with the largest residue (reference 5), a series of tests were performed to select the optimal slurry layer thickness in the measuring cell. On the one hand, optimal thickness offers an image with a sufficient number of particles to ensure it has adequate sensitivity without them overlapping, and on the other hand, it allows the X-ray tube to be used with low-quality radiation, which lengthens its service life and simplifies shielding requirements in hypothetical industrial equipment. From this series of tests, it was concluded that 1500 µm was an appropriate layer thickness. In addition, the optimal tube emission parameters (voltage, amperage and exposure time) were also set to ensure images with a good signal-to-noise ratio.

Subsequently, the way the three matrix sensors operated in the experimental set-up described above with the 5 lab-prepared slurries was compared. Figure 5 the resulting mean number of particles over 45 µm detected by each of the three sensors versus the residue measured with a 45 µm sieve.

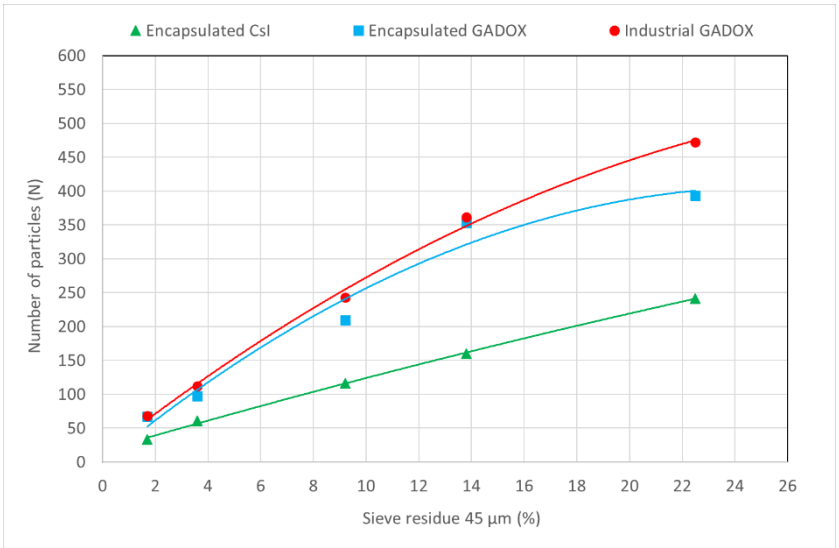

Figure 5. *Number of particles over 45 µm detected vs. residue with 45 µm sieve.*

Several conclusions can be drawn from the above graph:

- The correlation between the number of particles detected and the reside is good with all 3 sensors.
- The sensor with the highest particle detection rate is the industrial one, followed by the dental sensor using GADOX.
- Although it detected a smaller number of particles, the sensor in plastic casing with CsI scintillator has better linearity and correlation coefficient in the fit.

Since both the CsI plastic-cased matrix sensor and the industrial GADOX sensor had yielded good results, in order to select one of them to use in the slurry measuring prototype, the outer wall of an aluminium container was X-rayed with the different

sensors (Fig. 6). The wall, with a total thickness of 50 µm, in fact comprises two outer plastic layers that form a sandwich with a middle layer of aluminium. Although all three sensors are specified to have the same resolution, the GADOX industrial sensor offers the best definition and contrast. That was the reason why the industrial detector was selected to be fitted on the industrial prototype.

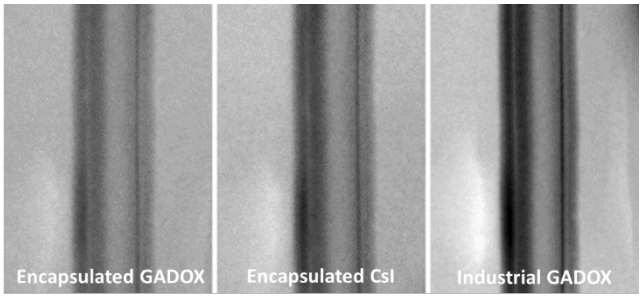

Figure 6. *X-rays by the three sensors of a three-layer aluminium receptacle with a total thickness of 50 µm.*

4.2 INDUSTRIAL PROTOTYPE

With the knowledge acquired from the experimental set-up, an industrial prototype shown in Figure 7 was designed and built. The unit combines the X-ray measuring chain with an automatic slurry transfer and cleaning system. The equipment is controlled by a central computer and interacts with the user through a touch screen fitted on the outside, from where the developed control, analysis and visualisation software is run.

Figure 7. *Front view of the GrindSizer® industrial prototype with the door closed (left), door open (centre), and shielded area open (right).*

Figure 8 shows detail of the top compartment on the unit where the X-ray tube is fitted, the cell through which the slurry under study flows, and the matrix sensor that generates the images to be processed. The measuring cell sits on a guided trolley that is connected to an electric linear actuator, which enables the cell to be moved out of the field in order to obtain target references in the absence of a sample. The bottom of the equipment houses the electrical panel with control components and the slurry drive system with automatic cleaning that ensures the equipment remains in service for long periods of time without operator intervention.

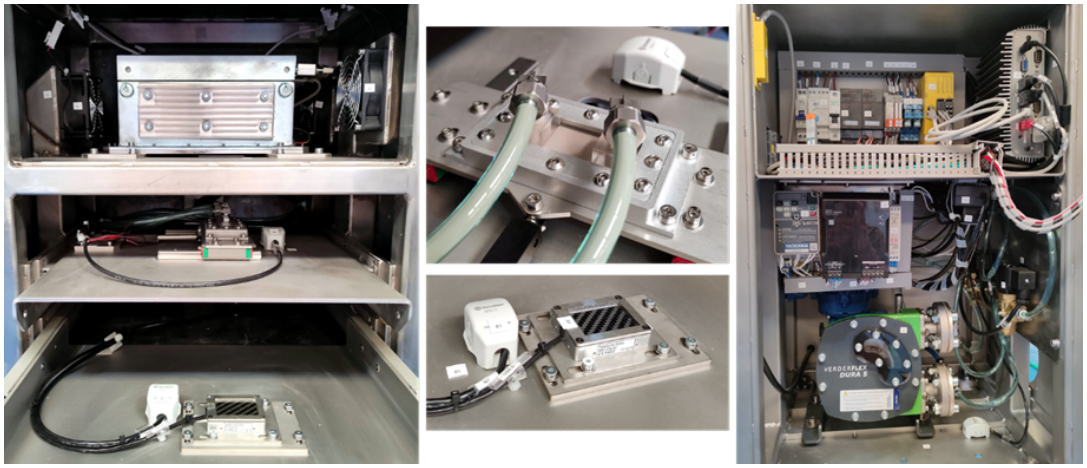

Figure 8. *Upper shielded compartment of the prototype open (left). Detail of the measuring cell filled with slurry (top centre), and matrix sensor (lower centre). Detail of the electrical panel and hydraulic transfer and automatic cleaning system (right).*

Figure 9 illustrates one of the screenshots from the new software after measuring a sample of industrial slurry. The plot of the number of particles detected versus the calibration made with laboratory slurries of the same composition and solids concentration shows that the measurement fits the calibration perfectly, with a residue of 6.4% at 45 µm.

Apart from acquiring high-resolution X-rays of the slurry in the cell, the software implemented in the industrial prototype automatically controls the pumping processes from the mill and carries out the analysis required to estimate the residue. In addition, residue values, as well as other variables, are historicised and plotted on a graph that enables how this critical variable has evolved over time to be visualised.

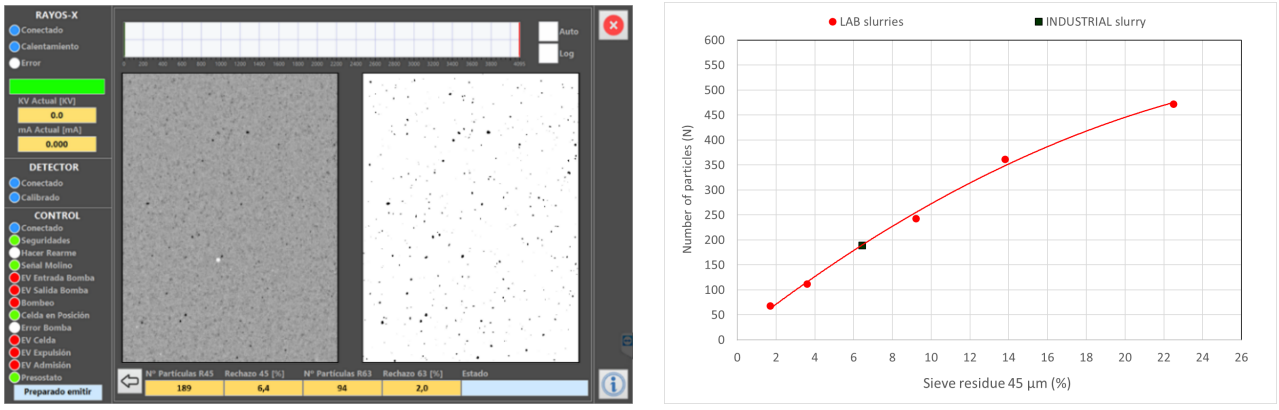

Figure 9. *GrindSizer® software display after performing industrial slurry measuring*

5. CONCLUSIONS AND FUTURE WORK

The following conclusions can be drawn from this study:

- A new particle size measurement technique based on X-ray imaging has been developed for concentrated ceramic slurries, capable of estimating the amount of residue at various cut-off points of interest to our industry (45, 63 and 125 µm).
- This new measurement technique has been used to construct an industrial prototype capable of sampling and analysing industrial slurry automatically and continuously in a closed cycle.p0
- The industrial prototype designed and built here has demonstrated its ability to adequately estimate the residue in industrial slurries using X-ray imaging techniques. The prototype has been validated in a radiography bunker but not in industry, since the use of an X-ray tube means it cannot be utilised outside a strictly controlled operating environment until the equipment is certified as exempt by the Nuclear Safety Council (NSC). Validation of this prototype will continue within a controlled environment to assess its measuring reliability under simulated industrial conditions.

6. REFERENCES

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