AUTOMATIC MEASUREMENT OF SPRAY-DRIED POWDER GRANULOMETRY FOR ON-LINE PROCESS CONTROL

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ABSTRACT

This paper describes the development and application of an automatic on-line measurement system for the determination of the particle size distribution of the spray-dried ceramic powder. The system is based on an optical imaging device suitable for on-line application, quicker than image analysis by microscopy, more accurate than industrial sieving and cheaper with respect to laser diffraction. The basic measurement principle was presented and validated by the author in a previous paper[1]*. After this preliminary work, the measurement performances have been analysed and improved, the system has been industrialised and adapted for operation on the line with an automatic powder sampling device. The device is capable of supplying information on the particle size distribution in a few seconds and can be implemented both at the exit of the atomizer and before the press. Therefore it is suitable for real-time parameter regulation, process control and Quality assurance in several key points of the production chain.*

On-line tests are described, showing significant granulometry variations in short time periods, thus highlighting the importance of such a control procedure. Comparisons with traditional sieve measurements are also presented. Results demonstrated that the influence of the sieving procedure is strongly intrusive on the analysed sample, with relevant consequences on the accuracy and repeatability of the results.

Other important issues related to the statistical significance of the analysed sample are addressed. The problem is in fact that only a few particles at a time could be measured. So the best compromise between measurement precision and statistical reliability of the results is the key concept to the solution of problem. Statistical parameters are used to this aim.

1. INTRODUCTION

In ceramic tile production, the particle size distribution of the atomised powder is a parameter of fundamental importance to have satisfactory structural and dimensional properties of both green and fired ceramic tiles. The particle distribution should be kept into precise limits, in such a way as to have optimal and uniform characteristics in the final product. In particular, the combination of the particle size distribution and of the moisture content of the powder determines the optimal set-up of the pressing parameters. Several are the atomising parameters influencing the granulometry of the powder: density and viscosity of the slip, pump pressure or geometrical characteristics and position of the nozzles, etc. In order to have a more deep and prompt control of the particle size distribution, usually more variables are regulated at the same time. It is clear that different typologies of raw materials will be atomised with different results, therefore for each material a dedicated process set up should be used with different production limits.

Powders are generally characterised by means of a statistical distribution. They consist of particles that differ in terms of size and shape. So to characterise it, it is necessary to know the dimensions and shape of each particle as precisely as possible and at the same time the statistical distribution of these parameters over the entire sample. The knowledge of the shape is important also to reduce uncertainty in converting data counted in diameter frequency distribution (typical of modern digital systems) into weight distribution (typical of state-of-the-art sieving methods) through volume estimation.

The atomized ceramic particles have generally a toroidal shape with an equivalent diameter in the range from 90 to 1000 μm. Their frequency distribution is similar to that of a heterogeneous sample, with the maximum usually disposed around the smallest values. This feature leads to dimensional segregation of the sample, in the sense that the particles will not have a random spatial distribution, but the distribution will be influenced by their dimension. This makes the sampling of the powder from the line difficult: ceramic particles are often fed into containers or on belt conveyors, hence the feeding itself can generate both vertical and horizontal segregation. For these reasons, sampling should be done with precise procedures, as shown in[2,3].

Nowadays, in ceramic industries particle size distribution is usually controlled off-line using manual or electro-mechanical sieving devices[4]. The samples are taken from the line at interval of about 3-4 hours, but this kind of control does not allow a quick feedback on the control of the atomising process. An on-line, non-intrusive measurement technique is thus desirable, since it could give the possibility of continuously regulating the atomising parameters and to keep the produced powder within the desired limits. Therefore accuracy (at least better than 8 - 10 % of the readings in all the diameter ranges, apart those with very small concentration – e.g. 2 - 3 % - where an absolute uncertainty of 0.5 % is enough), velocity and reduced cost are all requirements that a sensor must have at the same time.

Automatic measurement methods suitable for ceramic powder are those based on Light Scattering or Diffraction. A light-scattering photometer measures the angular distribution of the intensity of polarized light scattered by particle sample[5]. For large particles, down to approximately 4 μm, the scattered light intensity is proportional to the square of their diameter for most scattering angles. The light scattering system defines a rectangular measurement volume by imaging rectangular apertures with the sending and receiving optics. A particle passes through the illuminated measurement volume and scatters light that is received by the detector optics and focused onto a photo detector. Each optical signal is converted into an electrical pulse that is processed electronically. The signal processor counts these pulses and builds up a particles size histogram. These sensors present the advantages of having high accuracy (in the order of 1-2 % of the reading) and real-time response, but they are expensive and quite complicated to be installed on the line, thus limiting the utilisation mostly to laboratories.

Recently also acoustic spectroscopy has been investigated. Sound waves interact with objects in a similar manner to light, but have the advantage of being able to travel through concentrated suspension and emulsion,[6]. It uses ultrasound frequencies, typically from 1 to 200 MHz. The attenuation of those waves is then accurately measured. The measured attenuation of sound wave as a function of frequency is known as the spectral data. There is a direct correspondence between the particle size distribution/concentration and the associated spectrum. However, at present, the acoustic spectroscopy sensors are at a development stage, which is suitable only for laboratory applications.

In this paper the development and application of an automatic on-line measurement system for the determination of the particle size distribution of the atomized ceramic powder is described. The system is based on an optical imaging device suitable for on-line application. The basic measurement principle was presented and validated by the author in a previous paper[1]. After this preliminary work, the system has been further developed, improved, industrialised in co-operation with the company SACMI and adapted for operation on the line with a patented automatic powder sampling device [7]. The device is capable of supplying information on the particle size distribution in a few seconds and can be implemented both at the exit of the atomizer and before the press, where the granulometry distribution can vary because of the storage period in the silos. Therefore it is suitable for real-time parameter regulation, process control and Quality assurance in several key points of the production chain.

2. THE PROPOSED MEASUREMENT METHOD

The proposed measurement technique is based on Computer Aided Image Analysis (CAIA) and simply measures the area of the particles present in a recorded image. The image is acquired using a high resolution CCD camera with a macro objective (Figure 1). This method measures a frequency distribution in counts, as it examines the particles one by one. The details of the basic measurement method are described in[1], however hereafter the main steps are discussed.

The measuring method consists of four main steps:

1. automatic collection of a significant sample of powder from the spray dryer exit,
2. uniform distribution of the powder on the measuring surface,
3. image acquisition,
4. image processing.

The powder is automatically collected from the exit of the spray drier using a mechanical arm pneumatically controlled (for more details see Par. 4), with a procedure studied to optimise the accuracy of the sampling step. Then, the sample is gently disposed on a plate and homogeneously dispersed for measurement on the plate surface by induced light vibration. This is necessary in order to reduce particle overlapping.

In this work a progressive scan interline CCD camera (B&W, 1280 x 1024, pixel pitch 9 x 9 μm) has been used with a 60 mm macro objective. The camera was connected via USB to an industrial PC. The illumination has been carefully optimised in such a way as to reduce the shadow for each particle and to have optimal resolution. To this aim it is important to have the lamps symmetrically disposed with respect to the scene of interest. Once the viewing angle of the camera and the illumination condition are fixed, the resolution depends only on the distance between camera and scene. The numerical value of the resolution was found through a dedicated calibration, performed at the maximum allowable resolution, i.e. with the maximum lens magnification (1:1) and at the minimum distance. As reference a caliber with known dimensions was employed. The resolution achieved in this work is in the order of 10 μm/pixel. The resulting investigated area for each image is in the order of 130 mm^2.

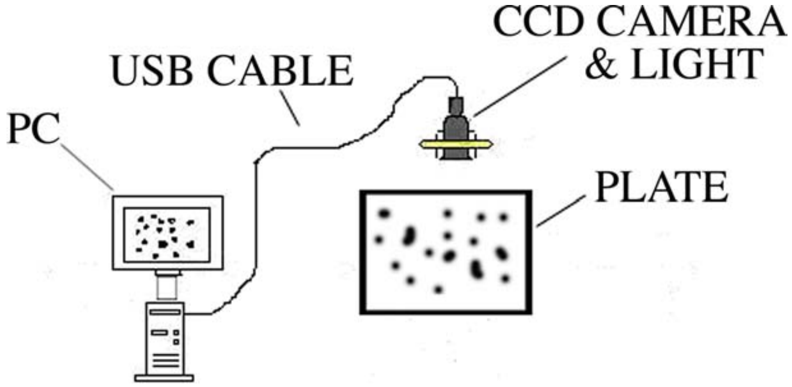


Figure 1. Scheme of the basic measurement chain.

The digital image processing procedure aims at measuring the diameter from each particle and then at extracting the statistical distribution of granulometry. This will allow extracting from the scene the information of interest and to compress the amount of data[8]. In the present application, the relevant parameters are the particle area, perimeter and number of particles in the analysed scene.

The acquired image must be conveniently processed before any measurement is done, because the particles must be highlighted with respect to the background. The acquired image is first filtered (by de-interlacing and Laplacian filters), thresholded (threshold value is automatically chosen using the Clustering Technique[9], which analyses the image histogram and calculates the major centres of gravity of the histogram regions: each grey level is then assigned to its nearest centre). The hole for each toroidal particle is filled by a filter based on connectivity and erosion is applied to eliminate spurious and noisy pixels. Then particles are separated in the image (the final image differs from the original one only for the elimination of the smallest connections) and finally it is possible to proceed to count and measure the particles. Figure 2 shows a typical acquired image, while Figure 3 illustrates the effects of the different applied filters.

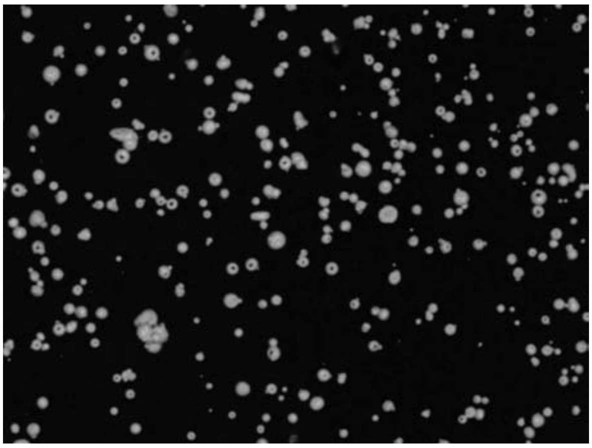


Figure 2. Typical acquired image.

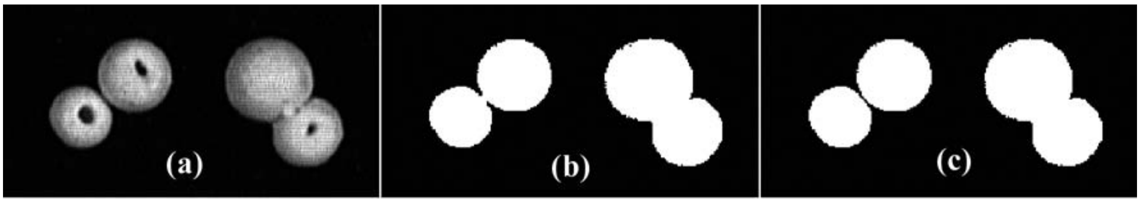


Figure 3. Sequence of applied filters: (a) de-interlacing, (b) hole filling (c) separation.

The measurement algorithm takes a pre-processed image in input and the number of pixel belonging to each particle is simply counted. Given a resolution value, the software measures the area in pixel or in μm2 and the perimeter in pixel or μm. From the value of the area, the Heywood diameter can be computed, which is defined as diameter of the circle having the same projected-surface area of the particle. This diameter is statistically acceptable and it is preferred to other available parameters (e.g. Feret or Martin diameters), because it takes into account the geometrical shape (i.e. both dimensions) of the object.

In order to have a representative sample of the population of the powder produced in the control interval, a sample of about 10-20 g is taken from the line, several consecutive images are taken on different fractions of this sample (see also Par. 4) and a final average value is extracted. For the m-th acquired image, once known the total number of particle and the Heywood diameter for each one, the particle size distribution can be computed as:

$$F_{m,i} = \frac{n_{m,i}}{\sum_{i=1}^{I} n_{m,i}} \tag{1}$$

where:

$F_{m,i}$: partial fraction of particles with Heywood diameter in the *i*-th interval of the distribution;
$n_{m,i}$: number of particles, measured in the *m*-th acquired image, with Heywood diameter in the *i*-th interval of the distribution;
I: total considered intervals in the particle size distribution.

If N consecutive measurements are performed on the considered sample, the final average value of the partial fraction in the *i*-th interval of the distribution can be computed as:

$$\overline{F}_{N,i} = \frac{1}{N} \cdot \sum_{m=1}^{N} F_{m,i} \qquad (2)$$

The number N of images necessary to have a significant representation of the population is determined using statistical parameters. In particular two of them can be used which are strictly connected:

- the estimate of the uncertainty of the average value[10] expressed as the experimental standard deviation of the average:

$$s(\overline{F}_{N,i}) = \frac{1}{\sqrt{N}} \cdot \sqrt{\frac{1}{N-1} \sum_{m=1}^{N} (\overline{F}_{N,i} - F_{m,i})^2} \qquad (3)$$

- the % difference between two consecutive values of $\overline{F}_{N,i}$ (i.e. the estimate of the quantitative influence of the m+1-th image on the estimated average value):

$$v(\overline{F}_{N+1,i}) = \left| \frac{\overline{F}_{N+1,i} - \overline{F}_{N,i}}{\overline{F}_{N,i}} \right| \qquad (4)$$

The measure is stopped, and thus N fixed, when these values go below specified thresholds for all the considered intervals. The threshold is chosen depending on the desired compromise between accuracy and measurement time.

3. UNCERTAINTY ANALYSIS IN THE OPTICAL MEASUREMENT

The uncertainty analysis of the optical device identifies 3 main elements of uncertainty: 1) diffraction effects in the objective creates a difference between real diameter and image diameter[11]; 2) Optical aberrations; 3) Uncertainty due to image processing.

In[1] the quantitative evaluation of these sources were approached. It was shown that, for the present application, the first two factors can be considered as negligible (always below 1 %), while the contribution from image processing plays a major role. Spatial sampling of the image performed by the digital acquisition system limits spatial resolution. This fact, coupled to the use of a threshold in order to have a binary image needed for particle morphological analysis, causes uncertainty. Such an effect appears on the pixels at the particle edge. Particle diameter is measured, according to Heywood definition, through an area measurement, which is measured in number of pixels; then uncertainty on area affects measured diameter. Particle area has a minimum and a maximum value which derives from the computation of area taking into account border pixels or not; border pixels have an equal probability to be part of the particle or not. Therefore it can be said that the real area will lie between limits that are set by the number of border pixels. As a consequence, for the area it is possible to write:

$$A = A_{px} N_A \pm u(A) = A_{px} \left[N_A \pm \frac{N_P}{2} \right] \qquad (5)$$

where:

A = effective area of the particle;
A_{px} = effective area associated to each pixel (about 100 µm^2 in this work);
N_A = measured number of pixels in the area of the particle;
$u(A)$ = maximum uncertainty interval for the particle area;
N_P = measured number of pixels on the perimeter of the particle.

The Heywood diameter D can be computed as:

$$D = \sqrt{\frac{4A}{\pi}} \tag{6}$$

and the uncertainty on the Heywood diameter $u(D)$ can be thus evaluated by combined standard uncertainty:

$$u(D) = \frac{\delta D}{\delta A} u(A) \tag{7}$$

Using Equation (5), (6) and (7):

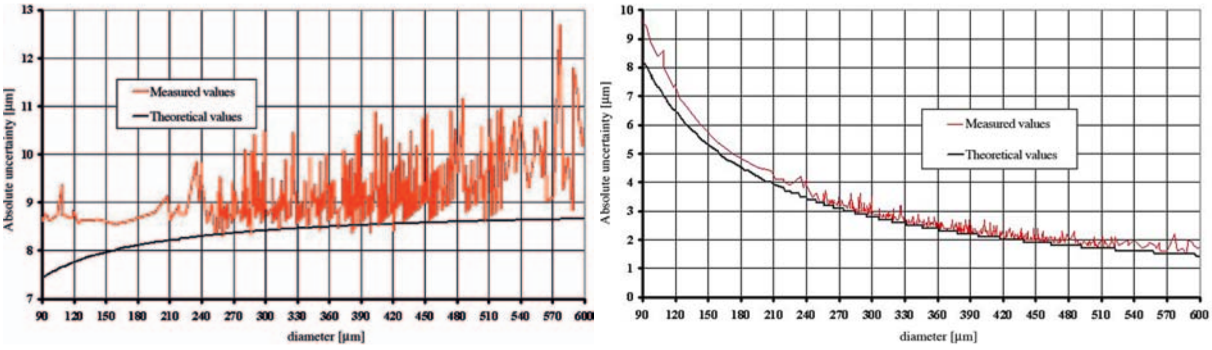
$$u(D) = \frac{A_{px} N_P}{2\sqrt{\pi A_{px} N_A}} = \frac{1}{2}\sqrt{\frac{A_{px}}{\pi}}\,\frac{N_P}{\sqrt{N_A}} \qquad 8)$$

which clearly shows how uncertainty on the diameter is related to resolution.

Relative uncertainty can therefore be computed as:

$$i_D = \frac{u(D)}{|D|} = \frac{N_P}{4N_A} \tag{9}$$

Based on the above equations, absolute and relative uncertainty υ(D) and iD can been computed for each particle diameter. Figure 4 shows theoretical and experimental values computed for a typical image.



(a) (b)

Figure 4. Absolute (a) and relative (b) uncertainty vs. particle diameter (theory and experiments).

The following comments can be drawn from figure 4:

- Relative uncertainty decreases with D;

- Absolute uncertainty increases slightly with D, with an asymptotic trend;
- Theoretical curves are the lower limit to uncertainty, because they are computed under the assumption of spherical particles, which have the minimum perimeter for a given area; in reality the shape is not spherical, therefore their image is not a circle and experimental estimates of uncertainty provide larger values with scattered results.

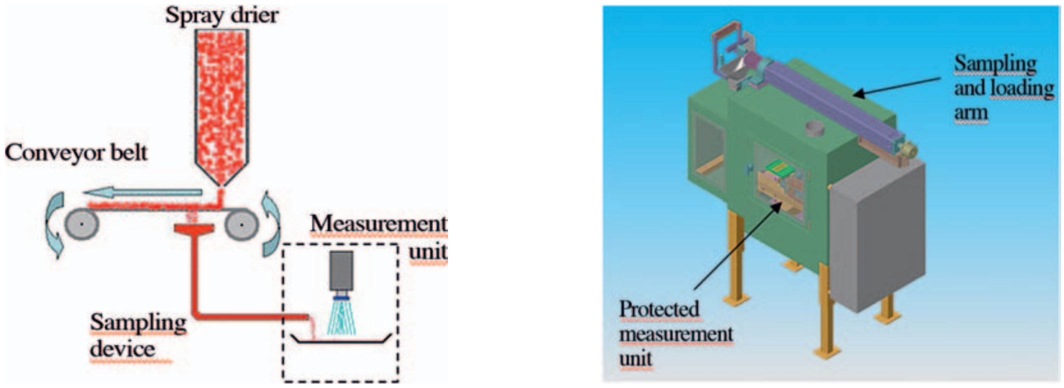
As a general result, uncertainty due to image digitisations and processing is maximum 10 % (9 μm) for the smallest particles and about 2 % (11μm) for the larger ones.

Sub-pixel interpolation can reduce such values. This is done by interpolating image edges with a two-dimensional function in the image intensity domain. Any pair of pixels along x or y is interpolated with a linear function, which is continuous. The image is then re-sampled with smaller pixels, so that a higher resolution image is obtained. Sub-pixel interpolation provides advantages up to a zoom factor equal to 3x. In this case uncertainty is maximum about 3.5 % (it lies between 2 μm ÷ 4 μm). If zoom factor is increased, then diffraction effects dominate, keeping uncertainty in the order of 1%. Taking into account that sub-pixel interpolation is relatively time consuming, a zoom equal to 3x is an acceptable compromise for on-line application.

4. DEVELOPMENT OF AN INDUSTRIAL PROTOTYPE

The basic measurement chain was then developed into an industrial prototype for on-line application. This was a very critical step, as the on-line sampling procedure must respect precise requirements in order not to introduce additional uncertainty in the results. Furthermore, several noise sources (e.g. dust, vibration, etc.) must be isolated from the harsh on-line environment. The result of this study was a patented system[7] for automatic sampling and measurement of the powder at the exit of the spray drier, suitable to give fast feedback information for the atomizing process control. The measurement system delivers high performance, comparable to that of a diffraction laser system, at significantly lower cost.

In Figure 5, the general scheme of the sampling and measurement procedure is shown, together with the 3D design of the developed industrial prototype.



(a) *(b)*

Figure 5. General scheme of sampling and measurement procedures (a); 3D design of the industrial prototype (b).

The system is composed of a piston (sampling and loading arm) which at regular intervals (e.g. 5 or 10 cycles/h) collects a small amount (e.g. 10-20 g) of powder using a sampling cup. The sampling procedure has been studied in order to be sure to take all the different powder fractions and avoid segregation problems. To this aim, the sample is taken by the sampling cup while the arm is moving horizontally along the whole length of the powder fall from a conveyor belt. Several different loading procedures were tested and compared and this one showed the best performances. Performances have been evaluated by comparison with measurements performed by accurate manual sampling and loading procedure from a homogenised and mixed powder sample. Results showed that discrepancies are in the same order of magnitude of the repeatability of the whole chain (both with manual and automatic operation, see Par. 5, Figure 6), thus demonstrating that the automatic sampling and loading procedure is not adding further dispersion and uncertainty and is collecting all the different powder fractions.

Once collected, the sample is gently disposed on a plate and homogeneously dispersed for measurement on the plate surface by a controlled short and light vibration. This is necessary in order to reduce particle overlapping in the digital image. Then, the high-resolution digital camera combined with an optical magnification system acquires the image. Finally, a device for powder aspiration is used to clean the plate and a new measurement procedure is started.

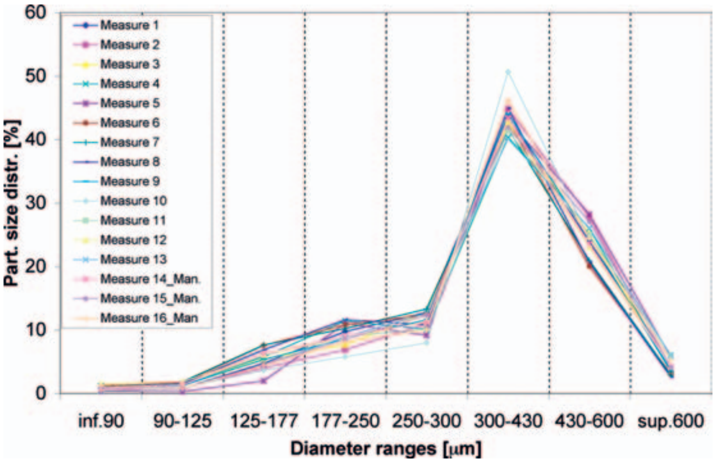
All the components of the measurement unit are protected against the dust, using isolating boxes for optical components and compressed air.

An industrial PC is used to control the measurement sequence and to process the images. To this aim, the algorithms previously described have been implemented in a dedicated software. Parameters, such as average diameter of the distribution or historical production data, can be easily analyzed in real-time. In addition, it is possible to analyze the granulometry curve in any arbitrary number of intervals, depending on the desired resolution.

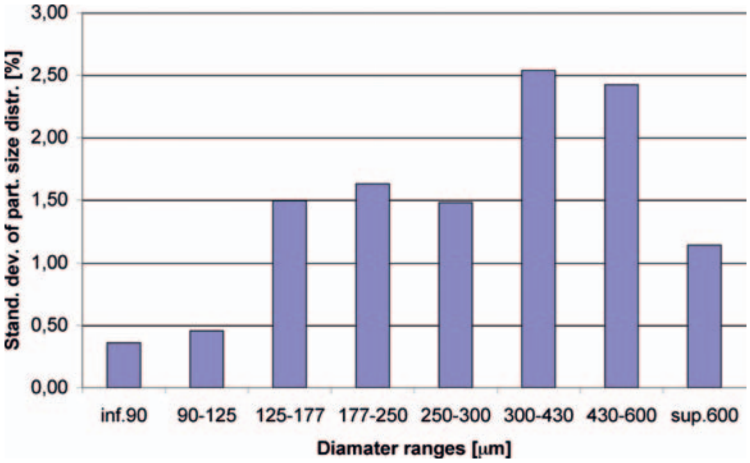
5. ANALYSIS OF LABORATORY CHARACTERISATION

Given the statistically related nature of particle size distribution measurement, repeatability is usually considered one of the most critical issues. The repeatability of the proposed measurement chain was evaluated by analysing data dispersion obtained from 16 measurements (each including sampling, loading, dispersion on the plate and optical analysis, repeated until the satisfaction of statistical parameters of Equations 3 and 4) of the same powder. The frequency distribution of particle size is measured in counts by the proposed method, but it is then converted in volume or weight (which are the same, if constant specific gravity is assumed), in order to have data comparable with sieving measurements. Results are shown in Figure 6, in terms of volume frequency distribution (a) and relative standard deviation (b) respectively. Repeatability varies from 0.4 % to 2.5 % in the different ranges (thus fully satisfying the industrial requirements) and seems generally to decrease with the diameter. This is surely related with the absolute uncertainty of the optical measurement method (see Figure 4–a), but by repeated measurements of the same images it was determined that the repeatability of the optical measurement is always below 0.2 % in all ranges. Therefore, repeatability is not affected only by optical measurement uncertainty (which could create data dispersion for the particles with diameter close to the ranges limits), but mainly by the statistical process of sampling and measuring. In fact, the 10-20 g sample which is taken from the main powder sample and measured is not exactly the same for the different measures and the powder is not disposed under the camera in the same way. This natural statistical dispersion is probably the most relevant factor and has an equally distributed relative weight over all the diameter ranges. As a consequence, the absolute standard deviation is proportional to the frequency amplitude distribution in the different ranges. This explains the reason for the higher dispersion in the ranges with higher distribution (i.e. 300-430 and 430-600 μm).

It is worth noting that the last 3 measurements (Measure 14, 15 and 16) have been performed with accurate manual sampling and loading procedure from a mixed sample. As it can be seen, the results are compatible within the repeatability interval, which is comparable for manual and automatic sampling procedures.



(a)



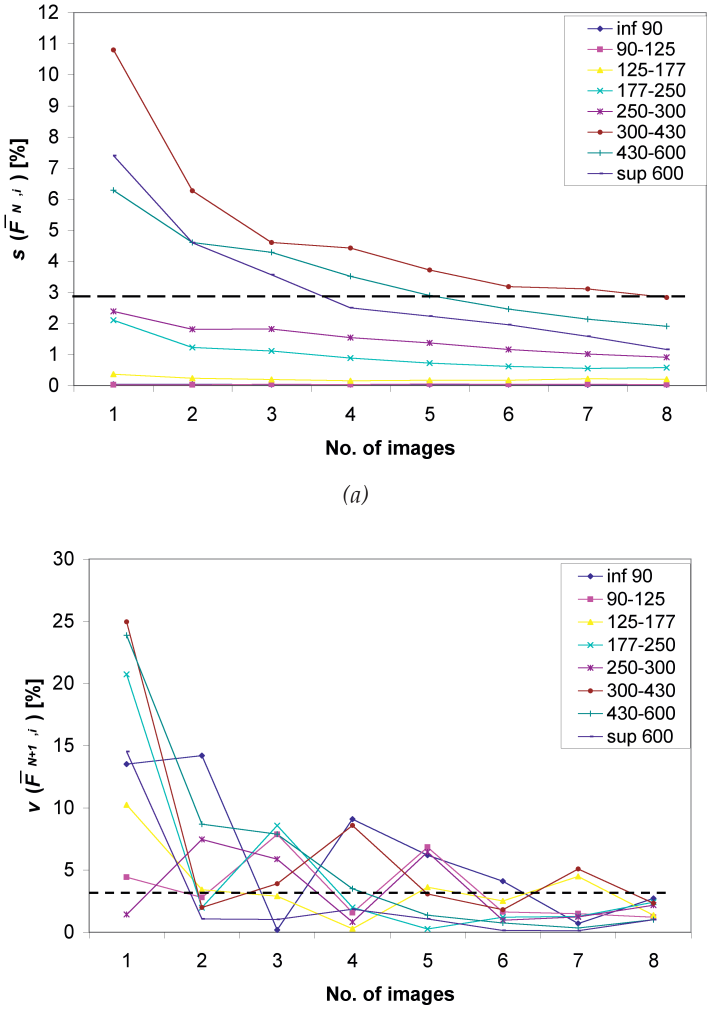
(b)

Figure 6. Repeatability test (a) and achieved standard deviation for each diameter range (b); particle size distribution computed in volume.

As a consequence of this statistical behaviour, the knowledge of the number of images necessary to have a significant representation of the population assumes an important role. As previously explained, the proposed statistical parameters of Equations (3) and (4) are used to take this decision. An example of the computation of $s(\overline{F}_{N,i})$ and $v(\overline{F}_{N+1,i})$ for 8 consecutive images is reported in Figure 7. It is possible to note that in the investigated case after 8 images both the values decrease below 3 % for all the diameter ranges, which can be considered as a satisfactory value. A higher number of images can be used to further improve the quality of the results, but a compromise must be found with the required time for the on-line control. 8 measurements take no more than 2 minutes to be performed.

In order to have a detailed characterization of the developed method, also a comparison with other measurement techniques has been performed

First of all, the sieving method was considered, which is the oldest and still the most diffuse one[3]. A number of sieves with openings of different sizes are arranged in order in a vertical stack with the sieve having the largest apertures at the top. After a 100g of powder sample has been charged on the top of the sieve, vibrator agitates the sieves stack for about 5 minutes. The particles larger than the size of each opening are retained on the upper side of each sieve. The size distribution of the powder sample is easily determined by weighing the particles retained on each sieve.



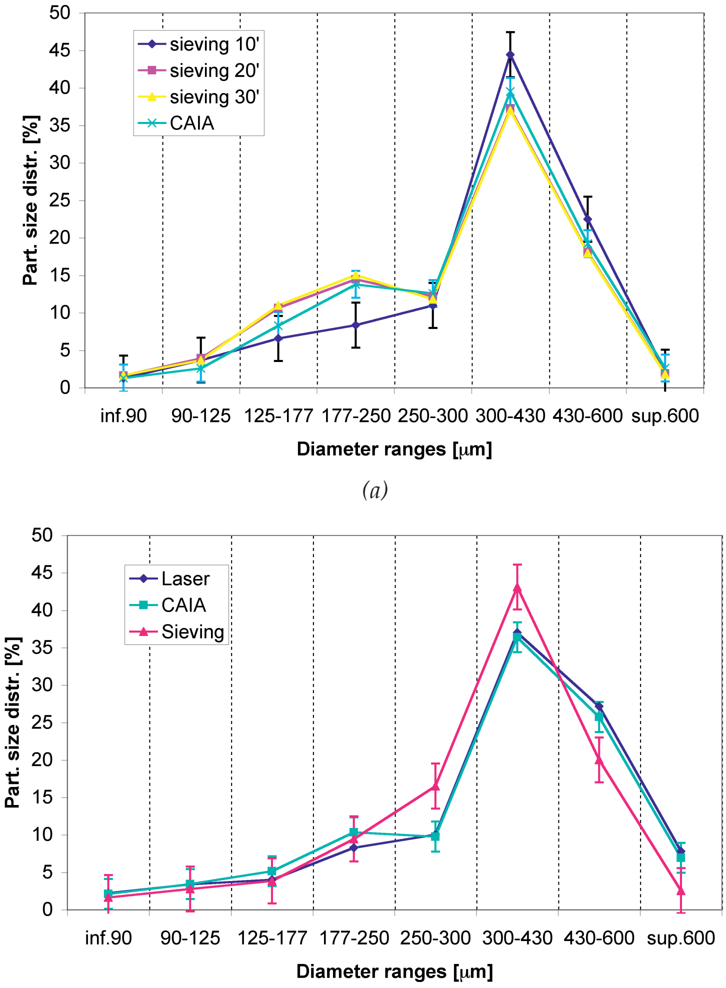
(a)

(b)

Figure 7. Standard deviation of the average values $s(\overline{F}_{N,i})$ *(a) and % difference between consecutive average values* $v(\overline{F}_{N+1,i})$ *(b) for a sequence of 8 images.*

The sieve offers the advantages of simplicity and low cost and as a consequence it is adopted as the standard technique in the ceramic industry. However, results can be affected by numerous parameters (usually an accuracy of ± 3 % is specified by producers), including uncertainties due to operator intervention in all stages of measurement. In addition adhesion to sieve and cohesion between particles may lead to poor repeatability. Finally large diameter particles may be broken by the long and strong vibration, causing a shift in the measured particle size distribution. This was demonstrated by experimental tests. Trials were performed on a 100 g sample of powder: the measurements were repeated on the same powder sieved by vibration for a time of 10, 20 and 30 minutes. The results (Figure 8-a) show a shift in the particle size distribution towards smaller diameters in the second and third measurements. This effect is due to collision between particles that occur during vibration, resulting in particle breakage. Particles of larger diameter and agglomerates, which are easier to break, divide up into smaller diameter particles, thereby influencing the weight of other close diameter ranges. Therefore the vibrating time becomes a major influencing parameter with systematic effects. After three measurements on the same powder with the sieve, the particles size distribution tended to be stationary.

At that point, the same sample of powder was taken from the sieve and measured using the proposed CAIA optical system. The information obtained was compatible with that provided by the last measurement performed with the sieving method (Figure 8-a), while in at least two of the most important ranges there is no compatibility between the first sieving measurement and the optical method. Uncertainty bars have been taken for all the diameter ranges as ± 3 % for the sieving method (data from producers) and ± 2 % (as an average value from Figure 6-b) for the optical method.



(a)

(b)

Figure 8. Comparison of sieving measurements with different vibration time and CAIA (a); comparison of measurements by sieving, laser diffraction and CAIA (b).

An interesting test was performed by measuring another powder sample with the CAIA system, a laser diffraction system and a sieve respectively (Figure 8-b). It was found that the results from the proposed optical system are entirely consistent with that made with the laser system, whereas the curve for sieving is not compatible with the others. In particular, again a shift toward smaller diameters was found due to the breakage of particles and agglomerates. However, it should be noted that whereas the distribution measured with the sieve refers to frequency distribution in weight, the distributions obtained with the optical and the laser methods express a frequency distribution in count. This distribution can subsequently be converted into volume or weight, but this process obviously requires some assumptions (such as constant specific gravity, etc.) that may introduce further deviation.

Another important factor influencing sieving results is the fact that a large number of particles of smaller diameter or of irregular shape can remain trapped inside each sieve, quite independently from the sieve size. In order to verify this effect, the CAIA method was used to measure the individual fractions separated with a sieving measurement and then collected in each sieve. The results are show in figure 9.

Note that, for all the sieves, the retained particles are highly dispersed over a much wider number of diameter ranges than expected. In some cases, it could happen that the maximum amplitude is not in the expected nominal range. Two main reasons can be identified for this:

a. smaller particles are trapped in sieves with larger mesh size,

b. irregularly shaped particles are able to pass through the mesh in the direction of their smallest dimension.

In conclusion this demonstrates that the real selectivity of sieves is quite limited and this strongly influences the final measurement accuracy. This problem can be overcome by the proposed optical method.

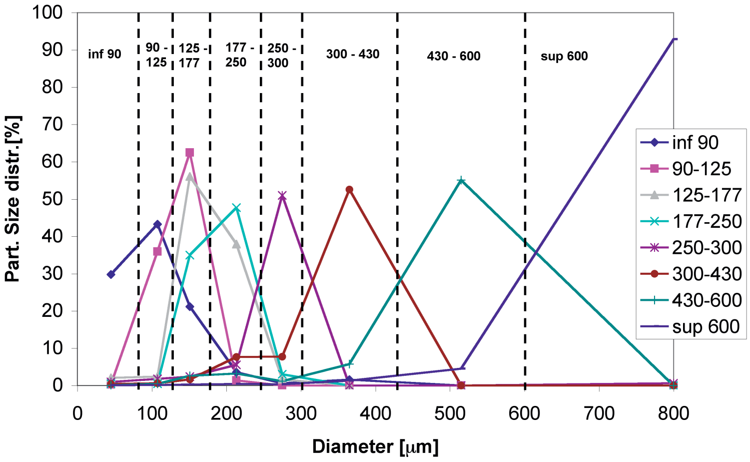


Figure 9. CAIA measurements on the single fractions found in each sieve after measurement (distribution computed in volume).

6. ON-LINE APPLICATION

The system has been finally installed and tested on the ceramic production lines of Leonardo 1502 Ceramica and Gruppo Panaria. Figure 10 shows the installation details. The system has been positioned at the exit of the drier and used for production monitoring. In Figure 11 an example of result is shown, reporting the trend of all the different powder fractions in a 450 minutes production interval. It is interesting to note quite important variations, which could not be detected with traditional discontinuous sieving measurements.

For example, the fraction within the 430-600 μm range is increasing of more than 10 % in about 100 minutes, thus showing a size distribution moving towards larger particles. The observed variations are well above the repeatability limits of the proposed measurement techniques, thus indicating real production variations. The achieved accuracy and repeatability satisfy the industrial requirements.

The availability of such data is clearly very useful for several important production issues. First of all, the data, coupled with information on the moisture content, can be used to control the main atomising parameters. Automatic alarms can be set to notify immediately when production specifications are no more met: this is particularly useful for the monitoring of critical phases, as the beginning of a new production, or the verification of the nozzle wear. Finally the data can be automatically stored and used for historical analysis or average parameter extraction.

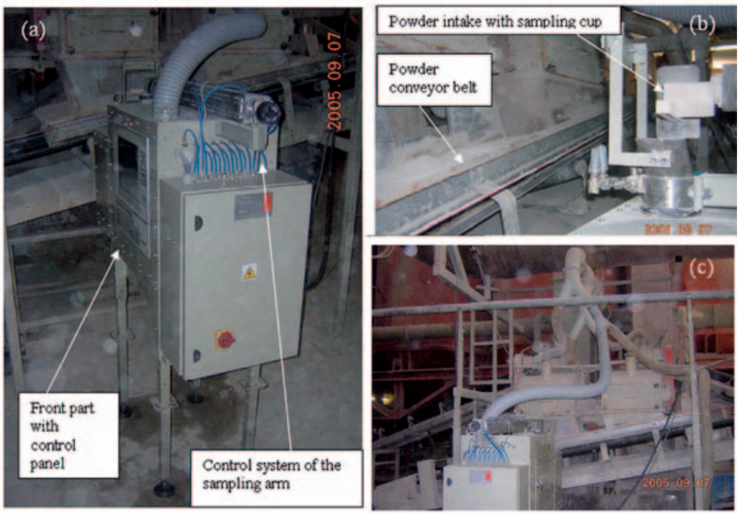


Figure 10. On-line implementation of the system: the industrial machine (a); details of the sampling and loading device (b); general view of the installation at the exit of the drier (c).

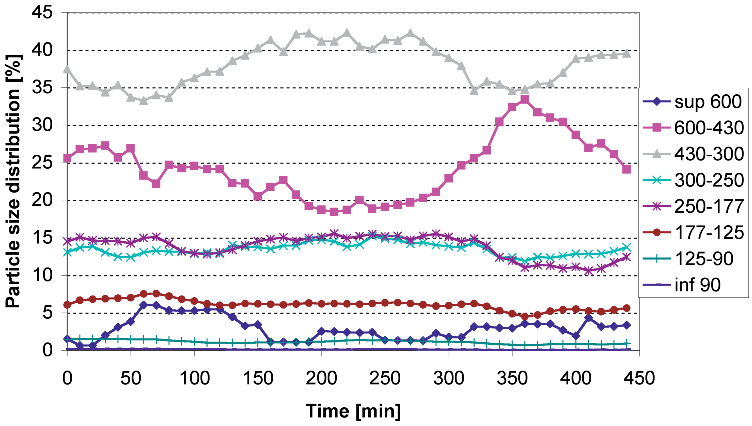


Figure 11. Time history of particle size distribution during 450 minutes of production (measurements every 10 minutes).

7. CONCLUSIONS

In the present work a new system for the on-line measurement of ceramic powder granulometry has been developed, analyzed and implemented at the industrial level. The system is based on Computer Aided Image Analysis (CAIA) by a high-resolution camera and dedicated algorithms for image processing. The industrial implementation has been approached, in such a way as to have a completely automated system for powder sampling and measurement ready to supply information for the control of the atomizing process.

A detailed performance and uncertainty analysis was performed, in particular in relation to state of the art systems, as those based on sieving or laser diffraction. It was shown that sieves may present uncertainty in the results due to the strong influence of the vibration and to the low selectivity caused by adhesion to sieve and cohesion between particles, which may lead to poor repeatability.

The proposed optical system solves these problems and makes the measurement procedure fully automated. The achieved repeatability (maximum absolute deviation of about 2.5 % in the ranges with higher concentration) is fully compatible with the industrial requirements. Additionally the system can estimate parameters such as the shape of particles and agglomerates and to assess other production anomalies, allowing for an improvement in quality. Statistical parameters are used to establish when the measurement can be stopped, i.e. when the analysed powder sample can be considered as representative of the produced population. The system has been finally implemented and successfully tested in real production lines.

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REFERENCES

[1] De Andrade R.M., Paone N., Revel G.M., “On-line measurement of ceramic powder granulometry by computer-aided image analysis”, Proceeding of the International Conference Qualicer 2000, March 12-15 2000 Castellon, Spain, Registration Number CS – 75/90.

[2] Allen, T., 1997, “Particle Size Measurements”, Chapman & Hall, Powder Technology Series, London.

[3] Arai, Y., 1996, “Chemistry of Powder Production”, Chapman & Hall, Powder Technology Series, London.

[4] Venturi, V., 1992, “Tecnologia Ceramica – Le Piastrelle”, Gruppo Editoriale Faenza Ed. S.p.A., Faenza, Italy.

[5] Hulst, 1981, “Light scattering by small particles”, Dover Publications Inc, NewYork.

[6] Hackley, V. A., 1996, “Ultrasonic characterization of particle size and charge”, Proceeding of the 5th Congress of Chemical Engineering”, Second International Particle Technology Forum, Track 2, July 14 – 18, San Diego, CA. American Institut of Chemical Eng., New York, pp. 577 – 562.

[7] Patent n. WO2005026699 A1 (International Application Number PCT/EP2004/010022): *Method for Continuously Controlling the Particle Size Distribution of Powders used in the Ceramic Industry, and Plant for its Implementation*

[8] Weeks, A. R., 1996 “Fundamentals of Electronic Image Processing”, SPIE Optical Engineering Press, Washington.

[9] Jain, A. K., 1989, “Fundamentals of Digital Image Processing”, Prentice Hall Inc, New York.

[10] ISO *Guide to the Expression of Uncertainty in Measurement*, ISO, Geneve, 1993, ISBN 92-67-10188-9.

[11] Paone, N.; Riethmuller, M. L.; 1989; “Uncertainty in a Particle Image Displacement Velocimetry Experiment: Measurements Inside a Centrifugal Pump”, 3th International Conference on Laser Anemometry – Advances and Applicatios, ASME, University College of Swansea, 26th – 29th September, pp38.1 – 38.11