DETERMINATION OF PM_X/TSP FRACTIONS IN CHANNELLED EMISSIONS OF THE CERAMIC INDUSTRY

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ABSTRACT

Aerosol size in atmospheric pollution phenomena is a key property, according to the recommendations of the World Health Organisation. For this reason, the most recent legislation on air quality in many countries echoes this criterion when it comes to fixing limit values. Thus, in relation to emissions, the European Pollutant Emission Register (EPER) includes, for the first time, the parameter PM_{10} applied to emissions, instead of the usual total suspended particles (TSP). In this sense, this parameter will foreseeably be extended to other regulations on emission at both European and State levels.

In the present study, a sampling methodology and mathematical data treatment have been developed and fine-tuned, which enable determining the particle size fractions (PM_x/TSP) of particulates emitted at channelled sources, using a seven-stage cascade impactor. In particular, the study focuses on the development and validation of a sampling methodology for the determination of PM_{10} particles (particles with an aerodynamic diameter ≤10 μm) and $PM_{2.5}$ (particles with an aerodynamic diameter ≤2.5 μm) in channelled emissions of the ceramic industry, calculated as PM_x/TSP fractions.

1. ENVIRONMENTAL REGULATIONS: PM_{10} AND $PM_{2.5}$

Particle size is a key property of aerosols and a major factor in atmospheric pollution processes, in addition to other features or parameters, such as particle morphology, chemical composition, chemical or physical reactivity, etc. This importance is confirmed by air quality regulations worldwide, in which settleable particles and total suspended particles are practically no longer being legislated, but, instead, regulations now focus on promulgating air quality limits in terms of PM_{10} particles. Certain countries, such as the United States, have created new regulations so that State and local authorities can develop programmes and actions to reduce $PM_{2.5}$ particle emissions into the atmosphere from power stations, factories and automobiles. The European Commission has recently proposed a strategy for quality improvement throughout Europe, in particular, proposing the regulation of the levels of $PM_{2.5}$ particles, among other pollutants. All these changes are due to the application of the recommendations of World Health Organisation, in which human health protection criteria prevail.

In the case of the Spanish environmental regulations, the transposition of European Directive 1999/30/EC (by approval in July 2002 of Royal Decree 1073/2002) modifies Spanish legislation on the evaluation and management of environmental air quality in relation to particles and other pollutants, and regulates PM_{10} levels. These regulations not only introduce an important change in the parameter to be measured, but also entail greater restrictions in the allowable limit values for air quality (immission limits).

However, in regard to atmospheric emissions from channelled sources, although the environmental regulations continue to refer to emission limits, and total suspended particles, it is in the European and in the Spanish State Pollutant Emission Register (EPER-Spain) in which the PM_{10} parameter appears for the first time, replacing the usual total suspended particles. Therefore, it is to be expected that in the near future values will be fixed for PM_{10} emission limits, and subsequently even $PM_{2.5}$, as has occurred in air quality.

For all these reasons, it has been considered of interest to study the situation of the ceramic industry in regard to PM_{10} and $PM_{2,5}$ emissions from channelled sources. For this, we have first developed a sampling methodology and studied its applicability in the ceramic industry, to determine the particle size fractions of the emission sources of greatest interest. In the middle term it is intended to have sufficient information to be able to establish toxicological emission factors that can be used in sectoral studies on inventorying emissions, toxicological studies, quality of air studies, etc.

2. SAMPLING OF PMX/TSP WITH CASCADE IMPACTORS

The sampling methodology followed in the present work is based on the total particle sampling method, adapted and simplified for ceramic industries[6], and on the special considerations for the determination of the PMx/TSP fractions, contained in the reference standards consulted, such as the German standard VDI 2066 Part 10[9], American EPA Method 201[1], and Japanese standard JIS K 0302[4].

Independently of the standards followed, it can be stated, in a general way, that the particles are separated from the gas stream in which they are suspended by the action of

different physical forces. These forces also represent the basis for the classification of the different sampling systems: samplers with filters, and other inertial devices (impactors, cyclones, centrifugal devices). These last samplers are mainly used in the determination of particle size fractions and/or particle size distributions.

In the present study a cascade impactor has been used (see section 4.2). This type of apparatus has been widely used for determining particle sizes in environmental, toxicological, hygienic-sanitary, pharmacological studies, etc. There are many types of cascade impactors, which differ in design, number of impactor stages, etc. For example, there are impactors that enable direct determination of the particle size fractions of interest (PM_{10}, $PM_{2.5}$, etc.) and other types that consist of several stages, which, based on sampling characteristics (principally intake flow rate and temperature) and design characteristics, allow obtaining the size distribution of the particles emitted by a given source.

On the other hand, the particle diameter used generally in atmospheric pollution and determined by this type of equipment is the aerodynamic diameter. This diameter is that of a spherical fictitious particle of density 1 g/cm^3, which has the same aerodynamic behaviour (same end velocity in a steady air regime) as the real particle. The use of this parameter enables comparing particles with very different densities, although the aerodynamic diameter may differ notably from the real diameter, even in the case of almost spherical particles, if the density differs considerably from 1 g/cm^3.

3. OBJECTIVES OF THE STUDY

The main objectives of the present study are:

- Developing and fine-tuning a sampling method for the determination of PMx/TSP at channelled sources of the ceramic industry by means of a multi-stage cascade impactor.
- Determining the PM_{10}/TSP and $PM_{2.5}$/TSP fractions of different stages in the ceramic process and analysing the results obtained.

4. EXPERIMENTAL

In the present study, both pilot-scale and industrial-scale (ceramic industry) tests have been carried out.

4.1. SET-UP AND MATERIAL USED IN THE PILOT-SCALE TESTS

In order to conduct the study on a pilot scale, an experimental set-up has been used (Figure 1), also used elsewhere by ITC[6]. This assembly enables regulating, in a simple fashion, the gaseous stream variables (gas flow rate, particle concentration, type of solid material, etc.), which makes it highly suited for this type of study.

The origin and physical characteristics of the solid material used are:

- Composition of red-firing clays, typically used in the Castellón area for ceramic tile manufacture, dry milled, to which 1% fumed silica was added to increase

flowability and to enable appropriate, constant proportioning of this material to the gaseous stream (consisting of air).

- The true density and particle size distribution of the material used was determined. The results are: density 2.703 g/cm^3 and mean volume diameter of 99.11 μm.

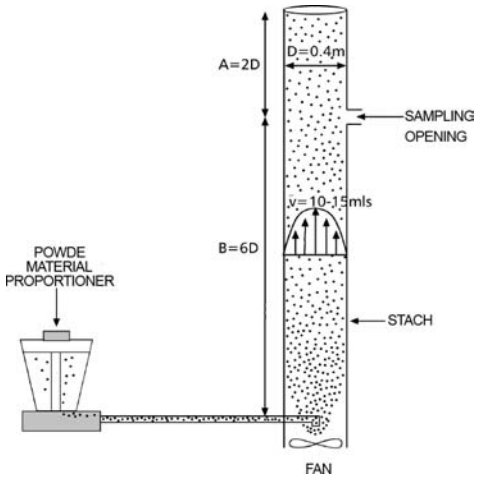

Figure 1. Detail of the experimental set-up used in the study

4.2. EQUIPMENT USED: CASCADE IMPACTOR

To conduct the present study, a MARK III seven-stage cascade impactor with a back up filter (Figure 2) was used. This apparatus has a particle diameter range of 0.3 to 20 μm and the particle separation mechanism is based on the principle of inertial impaction, where the cut-off diameter of each stage (d_{50}) designates the particle size at which half the particles will be captured and half will pass on to the following stage. The gravimetric measurement of each stage enables determining the total mass fraction in each aerodynamic size range.

Figure 2. Detail of the disassembled MARK III cascade impactor

In addition, the resolution of the impactor and particle separation accuracy can be affected by the intake velocity and the mechanical and dimensional design of the apparatus. In this sense, the manufacturer provides a series of graphs in which the cut-off diameter of each stage is plotted versus flow and temperature of the gas, from which it is possible to evaluate, by interpolation, the cut-off diameter of each stage for different sampling conditions.

Further aspects that determine the effectiveness of the capture and subsequent separation by particle size – a problem for this type of impactor – is the possibility that the particle could bounce or escape from the capture surface. To minimise this problem, the impaction plates can be covered with a thin layer of grease and/or a maximum mass is established per stage, which, depending on the standard or reference document, ranges from 10 to 20 mg.

4.3. PMx/TSP DETERMINATION WITH A CASCADE IMPACTOR

The following activities were performed on a laboratory and industrial scale:

- Study of the validity of the sampling procedure followed for the determination of the PM_x/TSP fractions by means of the cascade impactor. In particular, the validity of the sample extraction method, and of the conditioning and weighing of the filters has been evaluated. For this, pilot-scale and industrial-scale tests were performed, and the results obtained of total particle concentration (hereafter TSP) were compared with the traditional sampling train (one filter) and with the impactor.
- Studying and evaluating the repeatability of the applied test method (on a pilot scale).
- Comparing the results of the PM_{10}/TSP and $PM_{2.5}$/TSP fractions obtained with the impactor on a pilot scale by means of stack samplings, with those obtained by performing dry particle size analysis of the material with a laboratory laser diffraction instrument.
- Development and application of a mathematical treatment to determine the PM_{10}/TSP and $PM_{2.5}$/TSP fractions from the results obtained with the cascade impactor.
- Performance of sampling campaigns at different ceramic company sources, applying the methodology developed in the previous study phases to determine the PM_{10}/TSP and $PM_{2.5}$/TSP fractions.

4.3.1. Determination of TSP at ceramic sources with the cascade impactor.

To conduct this phase of the work on a pilot and an industrial scale, two types of samplings were performed:

- **Single-stage samplings (usual probe):** These samplings, made with the isokinetic probe in the usual configuration (a filter), following the procedure set out in the reference standard used[2-3] and applying the studied simplifications (simplified method for the ceramic industry), enabled obtaining total particle concentration (TSP), designated $\mathbf{C_T}$ in this case.

- **Seven-stage samplings (cascade impactor):** These samplings were conducted with the isokinetic probe; however, the single-stage filter was replaced with the cascade impactor, following the procedure indicated previously. This test was used to determine: total particle concentration (TSP), designated $\mathbf{C}_I$ in this case, particle size distribution of the material, and the PM_x /TSP fractions.

All the tests were carried out by sampling at a single point (centre point of the stack).

4.3.2. Comparison of the PM_x/TSP fractions obtained by different methods

In order to verify the validity of the PM_x/TSP fractions obtained with the cascade impactor, a sample of the solid material proportioned in the pilot scale set-up was subjected to particle size analysis by the dry method with laser diffraction in the laboratory.

In order to be able to compare the results obtained with the cascade impactor and the laser diffraction method, it is necessary to bear in mind that these techniques calculate, respectively, the aerodynamic diameter (spheres with density 1 g/cm^3) and volumetric diameter (considering the real density of the material). The relation between these two diameters can be found, simply, from the calculation of the mass of a given particle that has been subjected to testing in an impactor and in a laser diffraction instrument:

Impactor:
$$m_p = \frac{\pi}{6} \cdot \rho_{assumed} \cdot D^3_{aerodynamic} \qquad (1)$$

Laser diffraction:
$$m_p = \frac{\pi}{6} \cdot \rho_{real} \cdot D^3_{volume} \qquad (2)$$

As the mass of the particle must be equal in both cases, taking into account that the assumed density is equal to 1 g/cm^3, for the calculation of the aerodynamic diameter with the impactor, combining Equation (1) and Equation (2) gives:

$$D_{volume} = \frac{D_{aerodynamic}}{\sqrt[3]{\rho_{real}}} \qquad (3)$$

4.3.3. Mathematical treatment of the experimental results

The experimental results obtained directly with the cascade impactor are the mass retained in each stage (plates) plus that of the terminal filter, while an aerodynamic cut-off diameter is also associated with each stage. The cut-off diameter of each impactor stage varies in terms of the flow rate and temperature of the sampled gas, which, applying the manufacturer's design equations enables obtaining, in a simple way, a differential particle size distribution.

In order to be able to process the results of the particle size distributions found more simply and directly, and to calculate with greater accuracy the PM_{10}/TSP and $PM_{2.5}$/TSP fractions, the results obtained have been fitted to a cumulative log-normal

distribution. This type of (log-normal) distribution is commonly used in studying the particle size distribution of particulate materials

From a mathematical standpoint, operating with a log distribution assures that all the values obtained for the diameter are positive (logarithms), i.e. the results have a physical meaning across the entire range of studied diameters.

On the other hand, the particle size distributions found can be fitted to unimodal or bimodal distributions in certain cases. This last case is normally because particle generation in the process occurs by two different, simultaneous mechanisms. The larger-sized particles usually correspond to particles generated by mechanical processes (crushing, swept by gases, etc.), whereas the finest particles can originate from thermal processes that give rise to sublimation phenomena, such as condensation in the gaseous stream, producing fine particles[8].

The fit to a log-normal distribution has been made using the **Solve** function of the Excel spreadsheet, performing the minimisation by the Newton method. The distribution fit parameters allow readily obtaining the PM_X/TSP fraction.

The scheme followed for the calculation of PM_X/TSP and $PM_{2.5}$/TSP is shown in Figure 3.

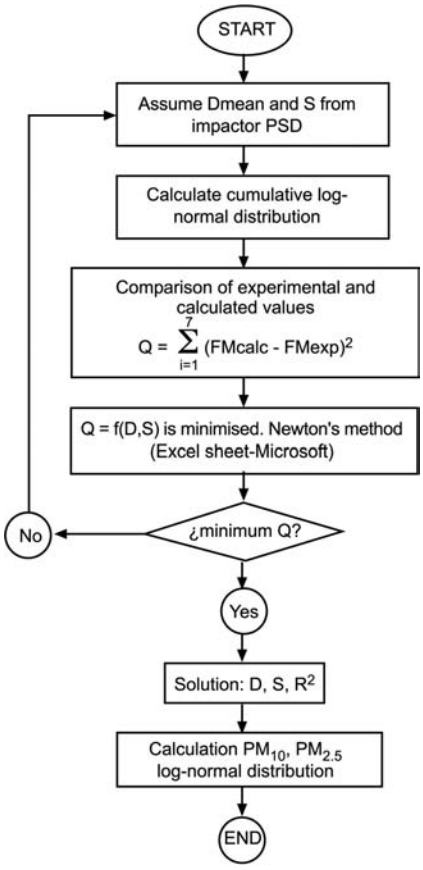

Figure 3. Flow chart of the mathematical treatment used to calculate PM_{10}/TSP and $PM_{2.5}$/TSP

5. RESULTS ON A PILOT SCALE

5.1. DETERMINATION OF TSP WITH THE CASCADE IMPACTOR

The results obtained in the different pilot-scale samplings have been plotted in Figure 3. In this phase of the work total particle concentration (C_T) has been determined consecutively with the traditional sampling train, and the total concentration with the cascade impactor (C_I), in both cases applying the same simplified sampling methodology of[6] detailed in section 4.3.1.

Although not plotted in Figure 4, the uncertainties of the results obtained in every sampling have been calculated. The criterion applied has been the same in both types of samplings (a single filter and a cascade impactor); however, in the case of the results obtained with the cascade impactor, the uncertainty was greater, due to the weighing of the filters, since in the case of the impactor, 8 filters are weighed (one for each cascade impactor plate, plus the back up filter). In addition, when the calculated uncertainty is below 10% of the particle concentration, a theoretical uncertainty of 10% is applied.

Figure 4 shows good correlation has been obtained between the pairs of values from the samplings. This indicates that the methodology used in sample extraction, with

all the simplifications considered in determining the total particles using a traditional sampling train, is applicable in the case of the cascade impactor for determining total particle concentration.

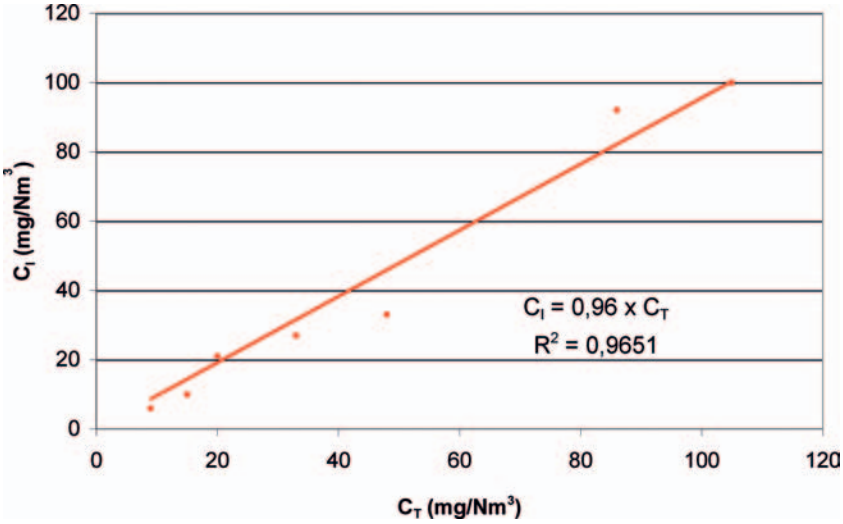

Figure 4. TSP concentration obtained with the cascade impactor (C_I) versus single-stage conventional sampling (C_T).

5.2. REPEATABILITY OF THE SAMPLING METHOD AND MATHEMATICAL FIT

The plots in Figure 5 show the particle size distributions obtained with the cascade impactor in a series of four consecutive samplings conducted on a pilot scale, keeping all the variables steady (of the stream and the material) to evaluate the repeatability of the method used.

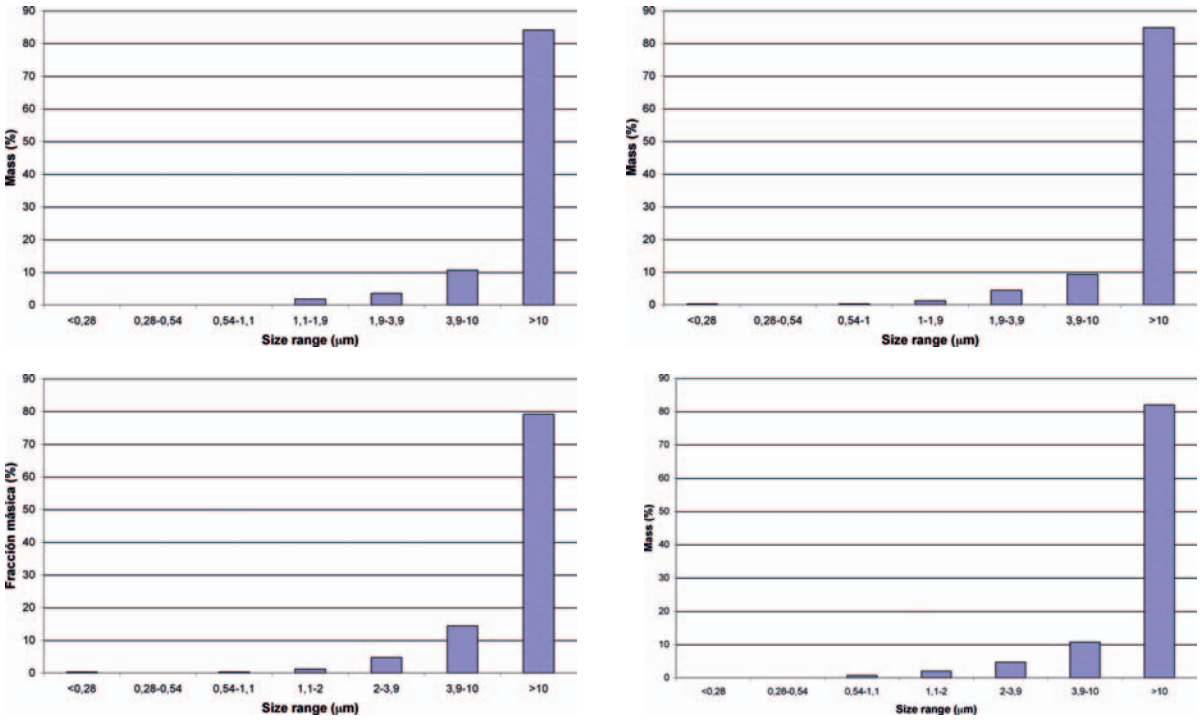

Figure 5. Particle size distributions obtained with the cascade impactor in the laboratory scale study.

Table 1 details the results of the PM_{10}/TSP and $PM_{2.5}$/TSP fractions obtained from the sampling results (Figure 5) and after applying the mathematical fit detailed in section 4.3.3. The calculation of the fractions of interest, PM_{10}/TSP and $PM_{2.5}$/TSP, can be made with sufficient accuracy, since these lie within the range of diameters for which the cascade impactor is designed (0.1-20 μm) and, in fact, display good repeatability, as indicated by the values in Table 1.

SAMPLING	CALCULATION OF PM_x/TSP (%)	
	PM_{10}	$PM_{2.5}$
1	15.8	2.7
2	15.2	3.1
3	17.9	4.0
4	20.5	3.2

Table 1. Values of fit to a log-normal distribution and calculation of the PM_{10}/TSP and $PM_{2.5}$/TSP fractions

The results obtained (Figure 5 and Table 1) indicate that the methodology used for the determination of the particle size distributions and the subsequent calculation of PM_{10}/TSP and $PM_{2.5}$/TSP exhibit a good degree of repeatability.

5.3. COMPARISON OF THE PMx/TSP VALUES OBTAINED WITH THE CASCADE IMPACTOR VERSUS THOSE OBTAINED BY LASER DIFFRACTION

Table 2 shows the mean PM_{10}/TSP and $PM_{2.5}$/TSP values obtained from the cascade impactor sampling results detailed in Table 1, together with the PM_{10} and $PM_{2.5}$ values obtained by dry particle size analysis with laser diffraction in the laboratory.

In order to compare the results obtained by the laser diffraction and cascade impactor methods, Equation (3) has been used.

PMx/TSP	Impactor	Laser diffraction
PM_{10} / TSP (%)	17.4 ± 4.4	17.3
$PM_{2.5}$ / TSP (%)	3.3 ± 1.0	3.5

Table 2. Comparison of the PM_{10} and $PM_{2.5}$ fractions obtained by the two methods used.

The results obtained by both methods practically coincide. Therefore, in accordance with these data the determination of the PM_{10}/TSP and $PM_{2.5}$/TSP fractions by cascade impactor appears appropriate or, at least, has an accuracy similar to that of the laser diffraction method.

6. RESULTS ON AN INDUSTRIAL SCALE

6.1. DETERMINATION OF TSP AT CERAMIC SOURCES WITH THE CASCADE IMPACTOR

Just as in the pilot-scale study, the sampling methodology used with the impactor has been validated on an industrial scale. For this, particle samplings were conducted

at three ceramic sources of differing natures, specifically in a spray dryer, in a press section extraction system, and in a fusion kiln for the manufacture of ceramic frits. The particle samplings were performed consecutively, determining the total particle concentration (C_T) and particle concentration with the impactor (C_I). Table 3 sets out the resulting data.

The uncertainty of the measurements was calculated applying the criterion detailed in section 5.1. In the case of the industrial fusion kiln source, the calculated uncertainty is significantly high in both cases; this is because the high particle concentration present in the gaseous stream clogs the filters, so that the sampling time is less than the time considered in the procedure and the calculated uncertainty is very high. However, this situation does not affect the objective of this study phase, which seeks to validate a procedure.

SOURCE	C_T (mg/Nm^3)	C_I (mg/Nm^3)
Spray dryer (after FVH)*	105 ± 11	100 ± 10
Presses (after FM)*	25 ± 3	19 ± 3
Frit fusion (before FM)*	630 ± 154	734 ± 323

** FVH: Wet filter; FM: Baghouse*

Table 3. Total particle concentration obtained by the simplified method for the ceramic industry (C_T) and with the cascade impactor (C_I).

The industrial-scale data confirm the pilot-scale findings, and display good correlation between the total particle concentration obtained by the traditional method and that determined with the cascade impactor.

7. DETERMINATION OF PM_{10}/TSP AND $PM_{2.5}$/TSP AT CHANNELLED SOURCES OF THE CERAMIC INDUSTRY

Applying the methodology developed in the present study, a PM_{10}/TSP and $PM_{2.5}$/TSP measurement campaign was carried out at several ceramic companies, considering different ceramic processing stages. The studied sources have been divided into two groups, based on process temperature, since the literature reports that this characteristic notably influences the size and composition of the particles emitted at these sources[8].

Table 4 sets out the results corresponding to the sources whose stream is at ambient temperature (known as cold sources or emissions), while Table 5 details the data corresponding to the processing stages with higher temperature emissions (hot sources).

The use of the cascade impactor to determine the particle size distributions in the emissions with low particle concentrations (dryers or emissions after treatment) entail the problem of requiring long sampling times. For this reason, the sample quantity collected in each plate is usually very low, which affects the reliability of the resulting particle size distributions.

Characteristics of the sampled source				Goodness of fit R^2	Calculation of PMx/TSP (%)	
process stage	Ref.	Sampling point	C_I (mg TSP/Nm^3)		PM_{10}	$PM_{2,5}$
Milling VH (1)	M1	After FM(2)	<5	0.9942	67.5	38.3
	M2		<5	0.9684	77.0	61.2
Milling VS (1)	M3	After FM(2)	<5	0.9279	88.3	68.5
	M4		<5	0.9544	62.0	34.3
Pressing	P1	Before treatment	109	0.9974	19.0	1.45
	P2	After FM	25	0.9951	24.1	2.11
	P3	After FM	19	0.9658	29.0	13.6
Glazing	E1	Before treatment	233	0.9903	36.7	11.3
	E2	Before treatment	27	0.9519	66.9	26.5
	E3	After FM	<5	0.9912	88.2	65.6

(1) VH: Wet method, VS: Dry method; (2) FM: Baghouse

Table 4. Results of the cold PMx/TSP samplings at ambient temperature sources or cold sources (T<40ºC).

Characteristics of the sampled source					Goodness of fit R^2	Calculation of PMx/TSP (%)	
Process stage	Ref.	Sampling point	T^agases (ºC)	C_I (mg TSP/Nm^3) (1)		PM_{10}	$PM_{2.5}$
Spray dryer	A1	Before treatment	81	>1000	0.9884	88.7	46.5
	A2	After FVH(2)	62	52	0.9654	95.4	59.4
	A3	After FM(2)	73	8	0.9704	94.4	60.6
Dryer	S1	Without treatment	115	<5	0.9735	83.7	64.9
Single-firing tile kilns	H1	Without treatment	157	14	0.9967	99.9	97.1
	H2	Without treatment	193	8	0.9125	99.0	91.2
Frit kilns	F1	Before treatment	108	734	0.9709	78.9	45.8
	F2		218	223	1.0000	76.3	62.9
	F3		243	269	0.9994	69.4	68.7
	F4	After FM(2)	102	<5	0.8450	89.0	45.0

(1) The TSP concentration is expressed in oxygen content under real conditions.
(2) FM: Baghouse; FVH: Filter by wet method

Table 5. Results of the PMx/TSP samplings at medium- and high-temperature ceramic sources (hot sources).

By way of example, Figure 6 shows some of the particle size distributions obtained in the industrial-scale samplings.

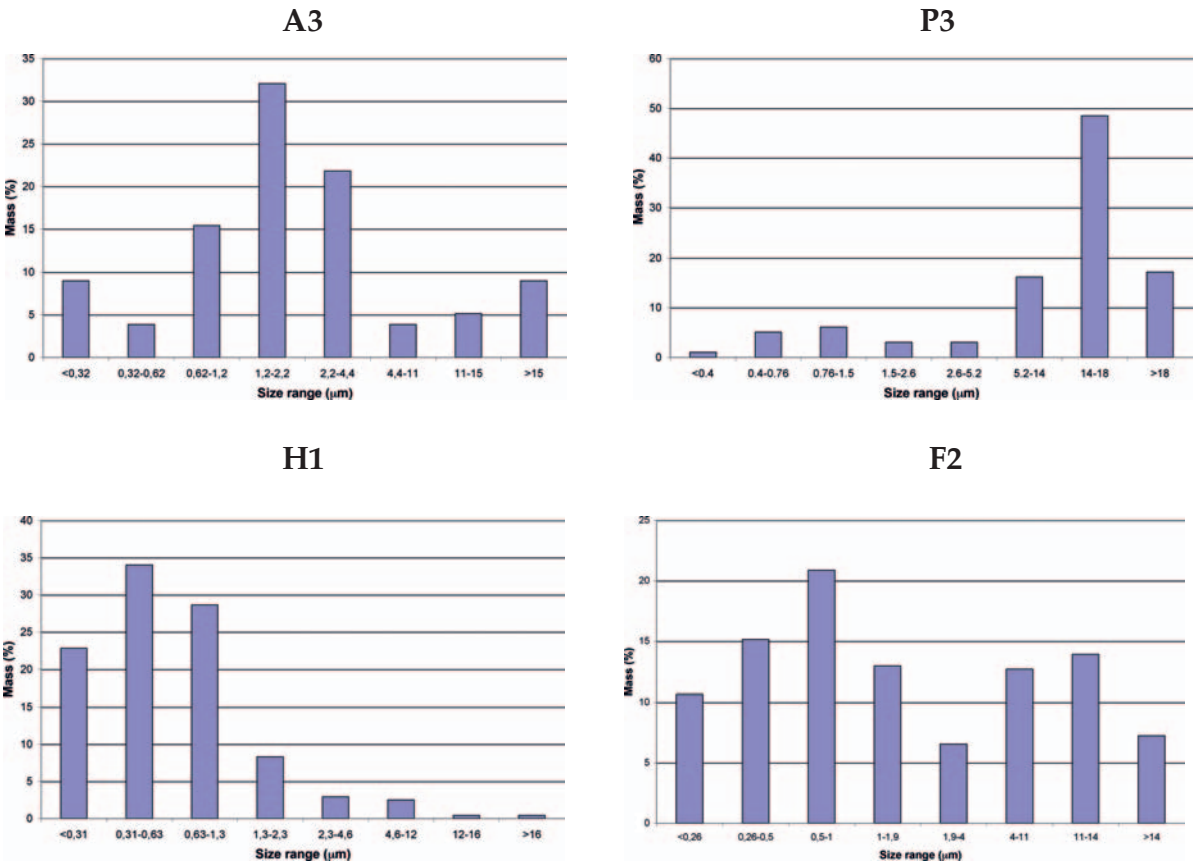

Figure 6. Example of particle size distributions obtained at different industrial sources of the ceramic industry.

Analysis of the results obtained (Tables 4 and 5, and Figure 6) indicates:

- The particles present at hot sources exhibit particle size distributions made up of smaller particles and, therefore, with higher mean values of PM_{10}/TSP and $PM_{2.5}$/TSP than in the sources at ambient temperature. These outcomes match expectations, since in the ambient temperature processes the particles are formed by mechanical processes, while in the hot sources, in addition to a mechanical origin, particles can originate by steam condensation processes when temperature decreases, or by chemical reactions between compounds (secondary particles).
- In the particle size distributions obtained after the treatment system, despite the limitations remarked in the determination, higher mean values of PM_{10}/TSP and $PM_{2.5}$/TSP are found than before treatment. Note, furthermore, that the fractions after the treatment stage depend, in addition, on the characteristics of the process, the treatment system used and its operation, so that these fractions can exhibit greater variability.
- The lowest PM_{10}/TSP and $PM_{2.5}$/TSP values are obtained in the pressing stage, probably because granulated raw materials (spray-dried granules) are used, whereas the highest values (smallest particles) are recorded in the glazed tile firing stage, in which the particles have formed mainly by condensation processes.

8. CONCLUSIONS

- The results obtained in the present study demonstrate that the cascade impactor is a good instrument for quantifying the total particle concentration emitted by channelled sources in ceramic and like industries (compared with the conventional TSP measurement method).
- The particle size distributions obtained with the cascade impactor can be fitted with good accuracy to log-normal distribution functions, provided the particle size of the particulate material studied is within the cascade impactor range of use. This greatly simplifies data treatment, allowing mathematical modelling and comparison of particle size distributions at different sources.
- The methodology developed has been used to characterise the particle sizes in an important number of channelled emissions in the ceramic industry, and to calculate the fractions of greatest environmental interest (PM_{10}/TSP and $PM_{2.5}$/TSP). This characterisation has shown that the particles present at the hot sources contain a greater percentage of smaller particles than at the ambient temperature sources, due to the different formation processes of these particles.
- The information obtained in the characterisation of the industrial sources needs to be completed with further data, particularly on the treated emissions, to be able to establish, reliably, PM_{10} and $PM_{2.5}$ emission factors in the ceramic industry.

9. ACKNOWLEDGEMENTS

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