Image processing method to adjust the ELPI distribution: application to the measurement of aerosol density emitted from combustion sources

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Abstract

Fly ashes from combustion sources are great of interest since they can be a risk for health and environment. Number size distribution of aerosols can be obtained using the Electrical Low Pressure Impactor (ELPI), which is dependent on the density ρ. However, the density ρ of the particles is specified by the user and assumed to be constant for the twelve stages. Besides, ρ cannot be measured with usual methods since there is not enough matter collected on each stage of the ELPI. ρ is proportional to the square of the aerodynamic diameter *dae* over the equivalent (Stokes) diameter *deq*. Our approach uses electronic microscopy to evaluate ρ at each stage to increase the accuracy of the number size distributions given by the ELPI. Firstly, particles were collected on glass substrates stuck by capillarity on the impaction stages. Then, images were acquired with an electron microscope, and finally, processing tools were applied: pretreatment (noise and contrast), segmentation (discrimination of the particles) and measurement (form factor to check the circularity and area to deduce *deq*).

This method was applied to Silica particles which are defined to have a constant density (ρ = 2.5 g.cm^{-3} with the pycnometer): density was found to be 2.2 g.cm^{-3} and 2.4 g.cm^{-3} for stages 2 (*dae* 57nm to 95nm) and 3 (*dae* 95nm to 158nm), respectively. The results match reality for fly ashes from wood combustion with ρ varying from 1.1 g. cm^{-3} to 2.4 g. cm^{-3}, respectively for impaction stages 2 to 8.

Key words: ELPI, PM, microscopy, density measurement, aerosol

Introduction

Fine particles are likely to pose a risk to environment and health because they can travel deeply into the respiratory tract. Particularly, very small particles (diameter below 1 µm) which largely dominate in number, may be responsible for some adverse health effects associated to air-pollutant exposure. These ultra fine particles get deposited in the alveolar regions of the lung where the adsorption efficiency for trace elements is up to 60-80% (1-3). Therefore, in a context of environmental policy, it would be useful to identify the sources of ultra fine particles. Cascade impactors are widely used for that purpose since they allow collecting particles and measuring the number size distribution. They can be used in many fields, e.g. particles emitted from diesel engines, ambient aerosols or other aerosols from combustion sources (4-6).

In this study, an Electrical Low Pressure Impactor (ELPI) manufactured by Dekati Ltd., Tampere, Finland, has been used to collect particles from 29 nm to 10 µm into 12 size fractions. The ELPI can be divided into three parts: the particles are first electrically charged according to their Stokes diameter, and then they are impacted on different stages according to their inertia related to their aerodynamic diameter. Finally, the induced current is measured. The number of particles depends on the induced current related both to the aerodynamic and the Stokes diameters. As explained later in the paper, the conversion of the Stokes diameter into the aerodynamic diameter needs to know the particle density. This density ρ has to be given by the user in the ELPI software. As a consequence, the density is a relevant parameter whose knowledge is necessary to get accurate size distributions. Particularly, parametric conditions in combustion processes (i.e., temperature, flow rate gas, flue gas treatments, oxygen fraction, etc…) have a great influence on the nature of particle emitted and therefore on their density (mineral fly and bottom ashes, char and tar formation,). As well, fuels nature (municipal solid waste, hazardous waste, biomass, sludge, etc…) produce aerosols with different chemical composition inducing a different density. Generally, the solid spherical particles of fly ash are called precipitator fly ash and the hallow particles of fly ash with a density of less than 1.0 g. cm^{-3} are called cenosphere fly ash. One common type of fly ash is generally composed of the crystalline compounds such as quartz, mullite and hematite, glassy compounds such as silica glass and other oxides. Bottom ashes from Municipal Solid Waste Incineration are mainly constituted of minerals as examples silica, calcite tringite and contain alkaline, alkaline-earth and heavy metals. The mineral composition of bottom ash and precipitator fly ash contributes to enhance the density of these materials in the range 2.0-2.5 g. cm^{-3} (7-9). Cenophere fly ash, which consists of hollow fly ash particles, presents low density in the range 0.4-0.7 g. cm^{-3} compared with the density of bottom and precipitator ashes. Diesel particles which mainly contain carbonaceous materials and a soluble organic fraction, present lower density values in the range of 0.3 – 0.8 g. cm^{-3} than aerosols from solid combustion processes (10).

The density ρ of the aerosol cannot be easily measured with classical experimental tools (like pycnometer) since there is not enough matter collected on each stage of the impactor in the exhaust during combustion processes. Moreover, for the calculation of the size distribution, the software uses the same ρ for each stage. However, in practice the density may vary from stage to stage.

An accurate measurement of the density for each class of size is not only essential for the evaluation of the number size distribution done by the ELPI, but also in the characterization of the studied aerosol.

This paper presents a method to evaluate ρ using image processing tools, as the density is proportional to the Stokes (or equivalent) diameter and the aerodynamic diameter. The aerodynamic diameter is known from the ELPI since each stage is characterized by its lower cut off aerodynamic diameter. As for the equivalent diameter, it can be estimated from images. Images have been acquired with a scanning electron microscope whose resolution allows a precise measurement of sizes.

In the first part, the ELPI limitations are depicted. Then, in the second part, the protocol for the particle collection with the ELPI and for the correction of the distribution is described. Finally, based on the distributions obtained on Silica particles, whose density is well known and constant all over the size range, the validity of the method is discussed and extended to the determination of the density of tar and fly ashes from biomass and wood waste combustion.

Experimental section

1. Formation and collection of aerosols

Three types of aerosols were collected on 22 mm diameter glass substrates purchased from ROTH before image acquisition by Scanning Electron Microscopy (SEM).

A mineral polydispersed aerosol was produced by flowing 20 mg of SiO_2 from SOVITEC in a tubular reactor. The electrical impactor was connected at the end of the reactor to collect the silica aerosol during 30 minutes. Two experiments were conducted resulting in namely experiment 1 and experiment 2.

The density of the silica was previously measured in solution in 2-propanol with a pycnometer. The density of SiO_2 was found to be equal to 2.5 ± 0.4 g.cm^{-3}.

An aerosol from natural beech wood combustion was collected with a residential wood fireplace purchased from FONDIS SA during 1h of run with the ELPI connected on the chimney.

The last aerosol was collected during small-scale incineration tests of wood waste (wood treated with Copper, Chromium and Arsenic fungicide) with a vertical fixed grid furnace at burning temperatures in the range of 850 °C – 1000°C and air flow rate of 5. 5 Nm^3. h^{-1}. The ELPI was placed in the flue gas treatment, upstream the wet air-pollution control system and a cyclone with a cut-off diameter of 40 μm designed to collect fly ashes. A density of 1.2 ± 0.2 g. cm^{-3} was measured for particles collected in the cyclone with a pycnometer in solution in 2-propanol.

2. Electrical Low-Pressure Impactor (ELPI): limitations

The ELPI is made of three parts: the particles are firstly electrically charged by the corona charger, and then, they are impacted according to their inertia in the impactor. Finally, the size distribution is evaluated from the induced current of the pre-charged particles (11). The charged particles induce a current when impacted on a stage. Each stage is electrically insulated with Teflon and a real-time measurement of the current I is obtained by electrometers. This current is then converted into a size distribution of the aerosol. The distributions (number size, mass size, diameter size...) depend on the conversion of the measured current to a concentration of particles. The relation between the concentration C_i of particles per stage i and the current I is given by the following equation (1):

$$C_i = \frac{I}{P.n.e.Q} \qquad (1)$$

Where C_i is the number concentration of particles per second of sampling ($cm^{-3}.s^{-1}$), P the penetration through the charger, n the number of charges per particle, e is the charge of an electron and Q is the flow rate (9.82 $L.min^{-1}$). The factor $P \times n$ characterizes the charger efficiency and depends on the Stokes diameter *Deq* which in the ELPI is related to the equation 2 (14):

$$d_{eq} = \frac{d_{ae}}{\sqrt{\frac{\rho.C_{d_{eq}}}{\rho_0.C_{d_{ae}}}}} \qquad (2)$$

Dae is the diameter of a particle having a density of one and the same velocity than the real particle and ρ_0 is the standard density ($1g.cm^{-3}$). The aerodynamic cut off diameter of the particles for a considered stage is characteristic of the ELPI. C_{dx} (x=eq or ae) are the Cunningham slip-correction factors (11-12).

In his analyse of the particles Morphology effect, DeCarlo (17) defined the relation between the volume equivalent diameter d_{ve} and the aerodynamic diameter d_{ae} by the equation 3:

$$d_{ae} = d_{ve}\sqrt{\frac{1}{\chi}\frac{\rho.C_{d_{ve}}}{\rho_0.C_{d_{ae}}}} \qquad (3)$$

The dynamic shape factor χ is a correction factor for non-spherical particles. This factor is equal to 1 for spherical particles and for compact aggregates (17) and greater than 1 in other cases. For spherical particles this equation 3 becomes equivalent to the equation 2 with d_{ve} corresponding to d_{eq}

As a result, d_{eq} can be directly determined by measuring the diameter of spherical particles on the images of impacted particles on glass substrate. In our case the studied nanoparticles are mostly spherical (at least 70% of particles have a form factor above 0.7). Consequently equation 2 is applicable and for silica particles, taking into account the measured density of 2.5 ± 0.4 $g.cm^{-3}$, table 1 expresses the influence of the density on the variation between d_{ae} and d_{eq}.

The ELPI technology also implies that the aerosol separation into size fractions depends on aerodynamic characteristic, whereas the measured current depends on the density ρ which can often not be a priori known, The figure 1 presents the influence of the density on the number size distribution giving by the ELPI software for an aerosol collected during the incineration of wood waste (CCA treated wood). The number distribution shown in the Figure 1 typically characterises an aerosol emitted from combustion sources with 99 % of the number of particles having a diameter below 1 µm (13). An increase of 20% of the density significantly enhances the number of particles calculated by the software for impaction stages with cut-off diameter below 1 µm. On impaction plates 1 to 4 (7 nm < d_{ae} < 95 nm), the increase of number of particles was observed to be enhanced in the range of 17% to 23 %. Density is therefore a relevant parameter. But it can not be easily measured with classical experimental tools (like pycnometer) since not enough matter is collected on each stage of the impactor during combustion processes.

Another problem is that this information is considered by the software to be identical for each stage. The assumption of constant density over the whole range is known to be a limitation of the instrument (14). Indeed, ρ may vary with the size according to the sampled aerosol and its chemical compounds.

The imprecise and global knowledge of the density ρ will induce errors in the evaluation of d_{eq}, thus in the characterization of the aerosol and number size distribution. The proposition of this study is to measure d_{eq} for each stage i to deduce ρ for each stage. The equivalent diameter of the particles impacted on the substrate was measured with image processing techniques.

3. Method and correction protocol

The control process goal is to enhance the evaluation of the particle size distribution analysis by evaluating ρ on each ELPI stage. The aerosol is sampled by the ELPI and the particles are collected on glass substrates. The glass substrates avoid inhomogeneous background in the images. This problem occurs with other substrates such as quartz or Teflon. In addition, particles are more likely to conserve their initial shape: when impacted on fibre substrates, the particles are likely to condense around the fibres. For example, Silica particles conserve their shape, as observed before and after the impaction with the Electron Microscope.

Then, gold-covered substrates are placed under Scanning Electron Microscope (SEM) for image acquisition. The SEM used is an FEI model Quanta 400. It was chosen related to its resolution of about 10 nanometres in classical conditions.

Finally, the measure of the equivalent diameter d_{eq} is obtained via the image processing tools of ImageJ software (15). The results are combined with the aerodynamic diameter d_{ae} of the studied stage to calculate the density ρ. This value can be reintroduced into the ELPI software to correct the size distribution according to equation 2.

Results and discussion

1. Image processing – Measure of the equivalent diameter

The diversity and the complexity of the images led us to set up adapted processes to treat two kinds of images: either when particles are piled up either when they are more spread out (Figure 2).

In this second case, an automated process made up of 4 steps was set up (shown in the Figure 3) applying the following protocol:

- pre-treatment to reduce the noise
- segmentation: discrimination of the particles
- post-treatment: separation of close particles
- Measures:
    - equivalent diameter d_{eq}

$$d_{eq} = 2.\sqrt{\frac{Area}{\pi}} \quad (4)$$

Considering aliasing phenomena, the precision of the measure was evaluated to be 6 % for d_{eq}. It leads to a 12 % error on the estimate ρ.

- circularity *FF*

The form factor informs on how spherical an object is:

$$FF = \frac{4\pi.Area}{Perimeter^2} \quad (5)$$

If FF=1, the particle is spherical. The lower the FF value is, the more elongated the particle are. If the 2D particle is circular, then, the 3D particle is likely to be spherical and the equivalent diameter measured on the 2D projected surface of the particle can be considered as the volume equivalent diameter (d_{ve} in equation 3). If the segmented object is not spherical, then the d_{eq} is not considered for the calculation of ρ. Only objects with an at least 0.8 circularity were kept to calculate the mean equivalent diameter d_{eq} and its standard deviation. Other objects are likely to be badly segmented, non circular or piled up particles. As stated before, non circular particles represent a small amount of impacted particles. Piled up particles are supposed to be the result of particles impacted on the same area, rather than aggregates formed before impaction.

For instance, Figure 3 shows the image processing protocol:

- on 213 particles measured (particles with less than 50 pixels were rejected since they either are from noise or too small; thus the error is too important), 174 have circularity lower than 0.8.
- the average equivalent diameter of the 39 remaining particles is 6 pixels (34 nm) with a standard deviation of 3.4 pixels (9 nm).
- for piled up particles, the segmentation and the post-treatment were granted by a semi-automated procedure based on the active contour segmentation called "Snake" (16).

The benefit of this step is the test of a new substrate: glass substrates allow the impaction of the particles with the ELPI and well suit to SEM observations. Also, according to the SEM observations of the Silica particles before and after the sampling, the substrate does not seem to alter the aspect of the particles. For a given plate, according to this procedure, the area of the particles was measured and the equivalent diameter was deduced. A size distribution of the particles impacted on each considered ELPI stage was established. The distribution is then fitted to a Gaussian distribution from which the mean value was calculated.

2. Impaction/Experiences protocol and results: Silica, Tar and Fly ashes particles from wood combustion

The method was firstly tested on polydispersed Silica particles since the density can be previously measured. Experiments with Silica were organized in two steps: in the first step, smaller particles were collected (up to stage 4) whereas the second step collected bigger particles (from stage 4 to stage 10).

In further experiences, the method was applied to tar emitted from natural wood combustion and fly ash from wood waste incineration.

- *calculation of the expected values of the diameter of silica particles impacted on each stage (d_{eq})*

Knowing the silica particles density (ρ=2.5 ± 0.4), the aerodynamic lower cut off diameters d_{ae} specific of the ELPI (Table 1), the Cunningham coefficient, the expected d_{eq} can be calculated with the equation 2. Table 1 gives the comparison between these aerodynamic cut-off diameter and the calculated equivalent diameters. For instance, the silica particles impacted on stage 2 should vary from 22 nm up to 50 nm, for the minimum and maximum values of the cut-off diameters. However, since the cut-off diameter is given for 50% efficiency, some particles collected are bound to be slightly smaller and bigger than these values. Size distribution of silica particles on certain stage will be established after microscopy observations.

- *Treatment of experience 1*

With ρ = 2.52 g. cm^{-3}, the number distribution given by the ELPI is represented on the Figure 4 where each bin represents a stage: from left to right: stage 1, stage 2…stage 10, stage 11 and stage 12. In this experience, particles were mainly collected on stages 1, 2 and 3. However, only the particles on the stages 2 and 3 were studied since the resolution of the SEM is not sufficient for stage 1 (d_{eq} expected being less than 25nm).

The observation of the stage 2 by SEM gives 1098 particles measures. They are classified into 13 size fraction according to the diameter precision measurement with ImageJ tools (Figure 5). The three more important bins with horizontal lines represent the particles whose size is exactly within the expected range for ρ=2.52 g.cm^{-3} (from 25 nm to 44 nm, Table 1), corresponding to 74% of the measured particles. Considering a density variation of 15%, 86% of the particles are within the expected range (22 nm to 50 nm, Table 1).

Using a reverse approach, the distribution Figure 5 can be used to determine the mean density ρ of particles impacted on stage 2. This repartition of the particles can be fitted as a Gaussian distribution with a mean diameter at 38 nm. Thus, the average equivalent diameter of the particles impacted on stage 2, d_{eq} =38 nm, is used to compute the density according to equation 2 and computing d_{ae} as the mean of the upper and lower cut-off aerodynamic diameters of the considered stage:

$$d_{ae} = \frac{d_{c2} + d_{c3}}{2} = \frac{57 + 95}{2} = 76\ nm \qquad (6)$$

d_{c2} being the lower cut-off diameter of stage 2 and d_{c3} the lower cut-off diameter of stage 3 (Table 1).

It comes from equation 2 that the average density of the particles is 2.2 g. cm^{-3}. This value stands in the range defined by the experimental measures with the pycnometer (2.5 ± 0.4 g. cm^{-3}).

The same reasoning is applied to the particles measured on stage 3 where 94% of the particles stand in the expected range, from 39 nm to 80 nm (Table 1). The equivalent diameter of the associated Gaussian distribution is 62.4 nm. Since the cut-off aerodynamic diameters of stage 3 are 95 nm and 158 nm (mean d_{ae} = 127 nm), the density calculated with equation 2 is $\rho = 2.4$ g.cm^{-3}. This value confirms the potential of the method since it is in agreement with the experimental pycnometer measurement of 2.5 ± 0.4 g. cm^{-3}.

- *Treatment of experience 2*

The second experience done with Silica permitted an analysis of the distribution of bigger particles. The images of the particles impacted on the upper stages were acquired with a SEM. The size of the particles observed on stages 6 to 10 varies significantly, from nanometer particles up to micrometer particles. This corroborates the fine particles losses phenomena occurring on the upper stages (12).

The proposed method was used to extract d_{eq} and evaluate ρ for the particles of the upper stages. All the results are summarized in the Table 2. The density values which correspond to the ones expected are in bold. For the stage 9, the distribution is more lognormal, shifted to the lower sizes, while the d_{ae} considered is still the mean value of a Gaussian distribution. This can explain why the density is slightly greater than the maximum expected (2.9 g.cm^{-3}). Moreover, the correlation factor is slightly lower than the one of the 'good' results. As for the stages 6 and 7, the distribution is more spread out and the identification with a Gaussian is not valid anymore. The correlation factor is indeed pretty low, thus the result can not be taken into account. The fact that these distributions are so wide probably means that the stages 6 and 7 are affected by the fine particle losses and by the rebounds. Stages 8 to 10 are probably affected by the losses too, but these particles were too small compared to the impacted ones to be measured.

Then, according to this test done with Silica particles, the method used to determine the density of the particles is valid when the Pearson correlation factor r is significant (higher than 94%).

- *Application to wood combustion experiments*

Once the method validated, it was firstly applied to fly ashes particles from wood waste incineration (Figure 6). Particles diameters on stage 2 were measured and the average of the equivalent diameter evaluated at 75 nm, with a Pearson correlation factor of 98% (distribution shown in the Figure 7). Thus, the equivalent density for d_{eq}=75 nm and d_{ae}=76 nm was 1.0 g.cm^{-3}. As the variance for the equivalent diameter is 10 nm, then $0.9 \leq \rho \leq 1.2$ g.cm^{-3}. The density range found for the particles impacted on stages 2 is rational for such an aerosol.

In a second time, the method was applied to an aerosol collected on the ELPI stages during the combustion of natural beech wood logs in a domestic fireplace purchased from FONDIS SA. Stage 2, stage 3 and stage 5 were analysed by SEM.

As shown in Table 3, the density of the aerosol ρ_1 resulting from image processing and calculated according to equation 2 presents large variation all along the impaction stages. This result underlines that the formation of particles in the exhaust during a combustion process occurs with different sizes related to different densities.

Number (N_1) and mass (M_1) of particles were calculated with the software of the ELPI applying values of density ρ_1 resulting from image processing for stages 2, 3 and 5, respectively. They were compared to those obtained with the usually reference density ρ_2 of 1 (N_2 and M_2). As shown in table 3, the influence of the density on the number of particles calculated by the software on each stage is significant: the relative difference between ρ_1 and ρ_2 is in the same order of magnitude that the relative difference between N_1 and N_2, for the three impaction stages, respectively.

The influence of a variation of the density on the mass is poor with a relative difference between M_1 and M_2 below the precision of the technique. When multiplying the density by three for a stage, the mass of the particles is overestimated by a factor of 7%.

The main benefit concerns the evaluation of the density of the aerosol depending of its size. Experiments from natural wood logs combustion in a domestic fireplaces demonstrate a large variation of the density of the particles emitted in the exhaust related to their diameter. Regards to the environmental regulations concerning pollutant emission factors, the number size distribution of particles emitted in the exhaust can be calculated with a better precision using the real density for each impaction stage of the ELPI. However, when the size of the particles is widespread, all the particles cannot be acquired on a same picture because of the resolution of the microscope. This problem mostly occurs for the upper stages, causing a truncated distribution. When a wide range can be observed and measured, the result is still biased if the differentiation between the lost and impacted particles is not done. Evaluation of the different losses will lead to a better approximation of ρ. Indeed, rebounds and fine loss particles are well known as being limitations of the ELPI. The distribution may then not only be wide, but also bimodal (especially for the intermediate stages), which wrongs the Gaussian model. In other cases, a lognormal distribution was observed. Then, another model to fit the distribution must be studied to enhance the precision of the method.

Thus, this method permits an evaluation of ρ, even though the variation for its value is pretty large. This variation would probably be reduced when the losses are taken into account and when a better model is applied for fitting the distribution.

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TABLES

Table 1: Equivalent diameter versus apparent density for Silica aerosol

	Stage	1	2	3	4	5	6	7	8	9	10
Cut-off diameter for 50 % efficiency	Dae (µm)	0.029	0.057	0.095	0.158	0.264	0.384	0.616	0.953	1.610	2.400
ρ = 2.14	Deq (µm)	0.014	0.029	0.050	0.089	0.159	0.240	0.398	0.627	1.075	1.615
ρ = 2.50	Deq (µm)	0.012	0.025	0.044	0.078	0.141	0.215	0.361	0.573	0.985	1.482
ρ = 2.90	Deq (µm)	0.010	0.022	0.038	0.070	0.127	0.196	0.332	0.529	0.914	1.377

	Experience 1		Experience 2				
Stage	2	3	6	7	8	9	10
Dae (nm)	76	127	500	785	1282	2005	3205
Deq (nm)	38	62.4	800	858	834	1118	1771
Correlation Factor (%)	99	94	85	81	97	93	95
ρ (g. cm^{-3})	**2.2**	**2.4**	0.4	0.8	**2.2**	2.9	**2.2**

Table 2: Results of the analysis on Silica particles collected during experiences 1 and 2

	Stage 2	Stage 3	Stage 5
mean Dae (nm)	76	126	324
mean Deq (nm)	69	82	157
ρ_1[a] (g. cm^{-3})	1.1	1.7	3.0
N_1 : number of particles for ρ_1	3.0 10^{12}	6.9 10^{12}	1.1 10^{12}
N_2: number of particles for ρ_2[b] = 1.0 g.cm^{-3}	2.5 10^{12}	3.5 10^{12}	4.3 10^{12}
Relative difference between ρ_1 and ρ_2 (%)	11	43	67
Relative difference between N_1 and N_2 (%)	16	50	63
M_1: mass of particles collected for ρ_1 (mg)	0.53	3.2	6.7
M_2: mass of particles collected for ρ_2 (mg)	0.52	3.3	7.2
Relative difference between M_1 and M_2 (%)	2	3	7

[a] ρ_1 was calculated according the equation (2) resulting from image processing
[b] ρ_2 is related to a spherical particle of density 1 g. cm^{-3}

Table 3 : Influence of the density on the number and mass of particles calculated by the ELPI.

FIGURES

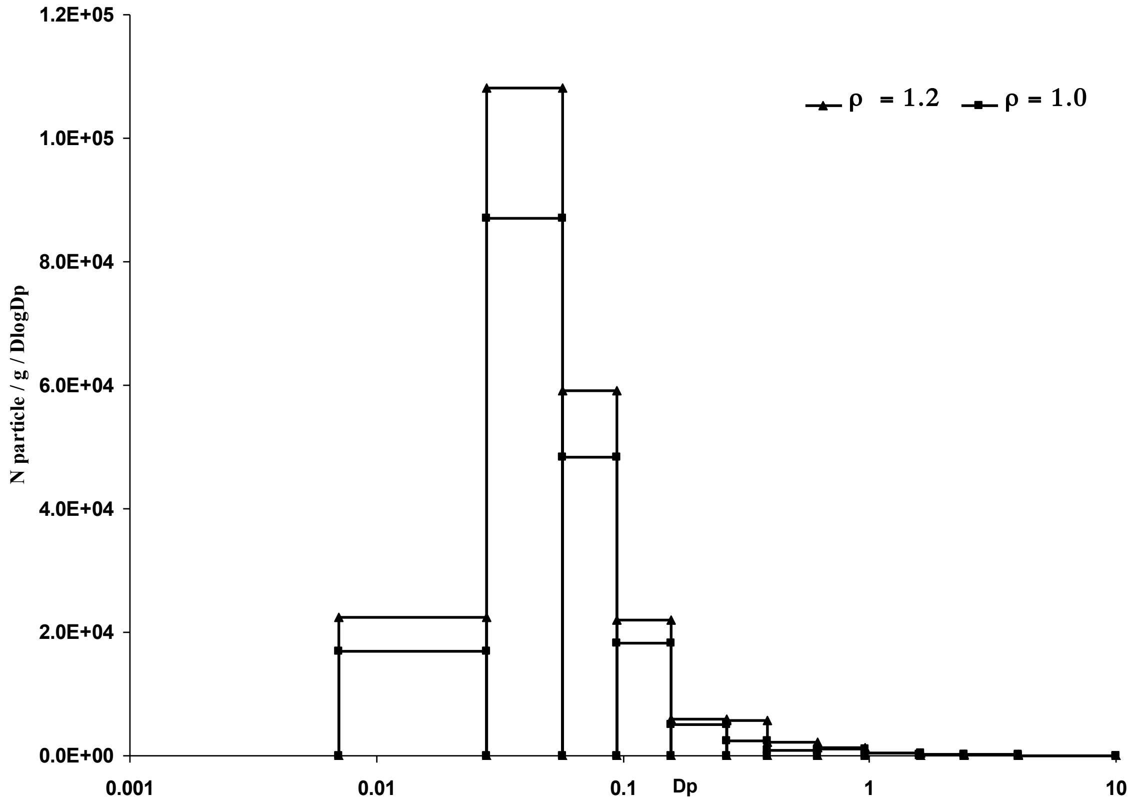
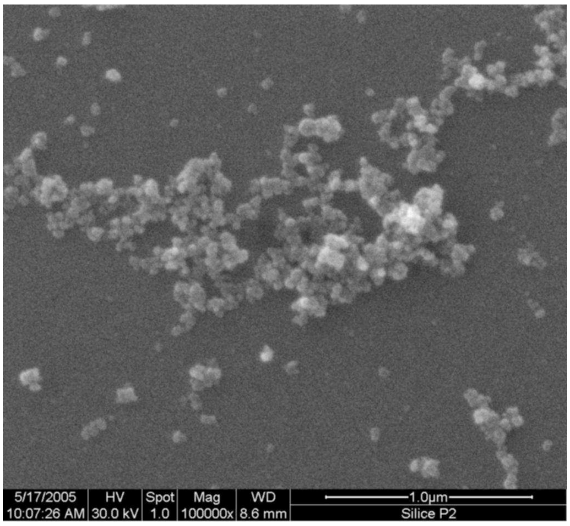


Figure 1 : Influence of the density on number size distributions for CCA treated wood aerosol



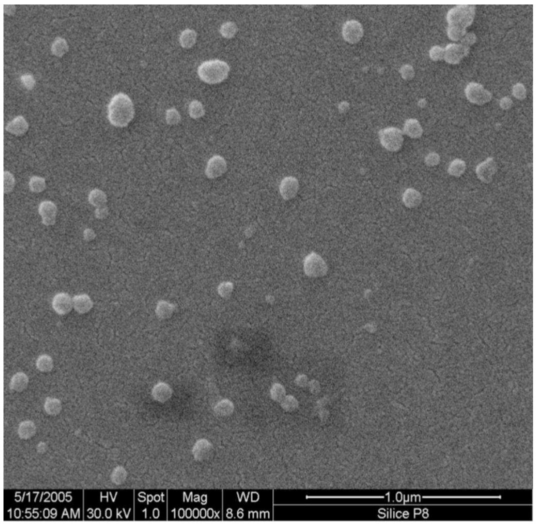


Figure 2: Piled up Silica particles on stages 2 (on the left) and 8 (on the right)

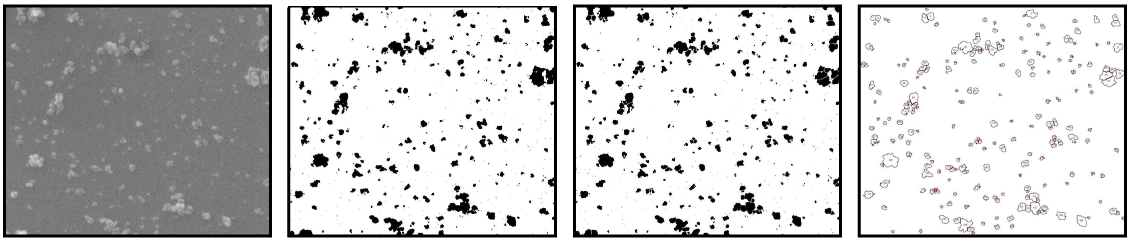


Figure 3: Example of an automated treatment (from left to right): pre-treated image; segmented image; post-treated image; labels of the measured particles.

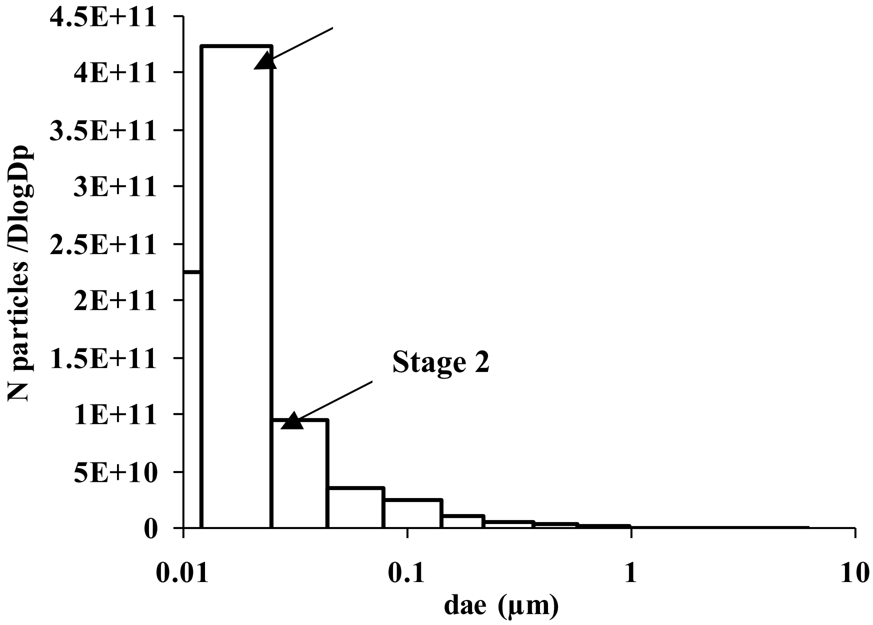


Figure 4 : Number size distribution of Silica – Experience 1

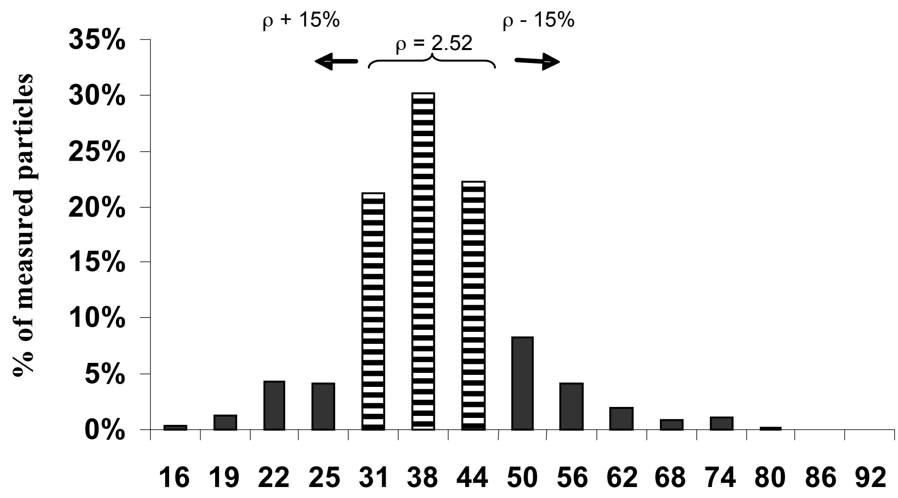


Figure 5 : Number size distribution of Silica particles impacted on stage 2 (1098 measures)

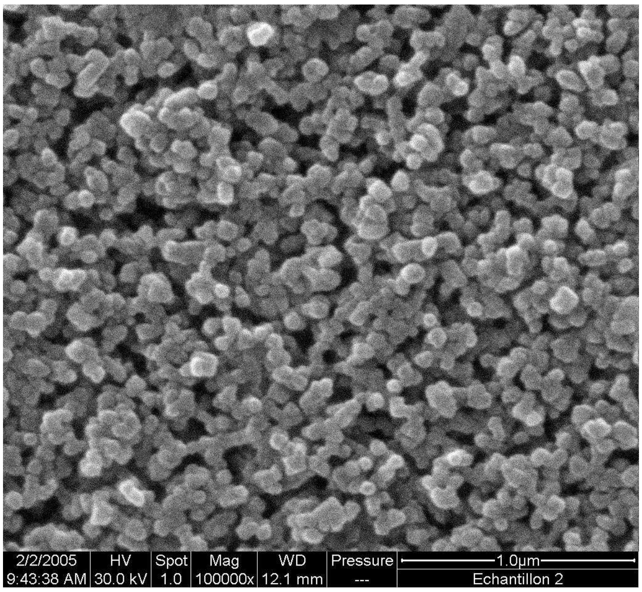


Figure 6: SEM images of fly ash particles impacted on stage 2 during wood waste incineration

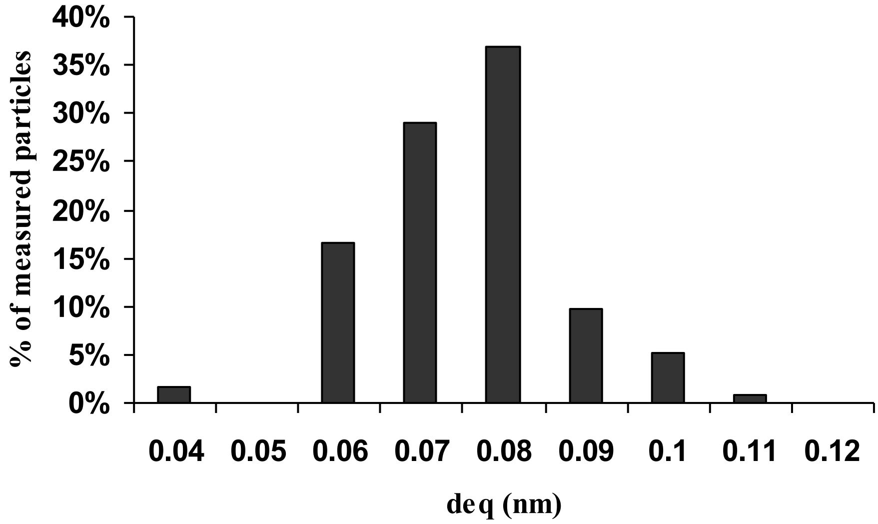


Figure 7 : Number size distribution of fly ash particles on stage 2 (114 measures) produced by wood waste incineration