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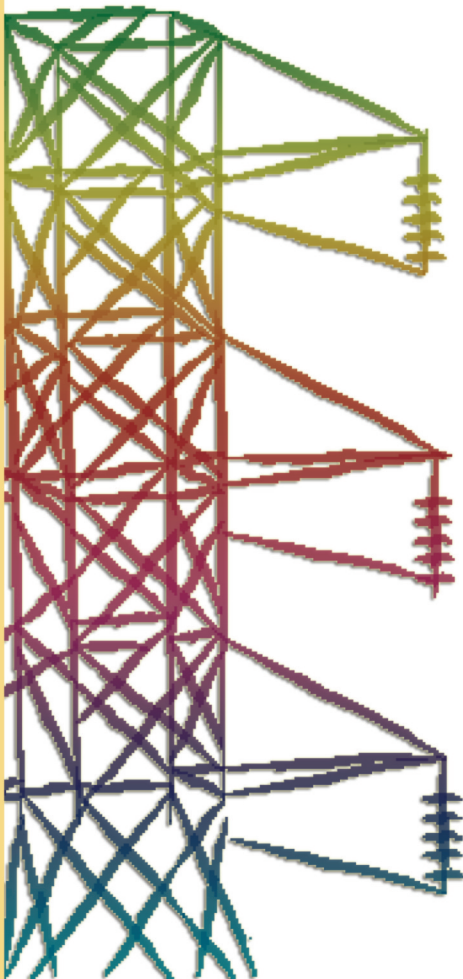


Ministero dello Sviluppo Economico

RICERCA SISTEMA ELETTRICO

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Nanoparticles Formation during High Temperature Pulverized Coal Combustion

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Abstract

A laboratory reactor has been developed to study ultrafine particles formation during pulverized coal combustion. The reactor consists of an atmospheric pressure flat laminar premixed flame homogeneously doped with pulverized coal particles. The gas mixture in which coal combustion occurs can be easily adjusted varying flame stoichiometry and diluent gas fraction. Both air and pure oxygen has been used as oxidizer while three bituminous coals have been added to the flame. The simple experimental configuration allows us to investigate the early stage of particles formation. Generated particles size distributions, in size range extending down to 1nm, were on line measured using a dilution probe and a high resolution differential mobility analyzer equipped with a Faraday cup electrometer. The results indicate that nanoparticles with size smaller than 30nm, commonly neglected at the exhaust of coal fueled combustors, are formed during pulverized coal combustion in not negligible amount. Moreover the shape of those nanoparticle size distributions is unaffected by the type of used coal.

Introduction

Coal is one of the cheapest and most abundant reserve sources of energy. Air-blown, atmospheric-pressure pulverized coal combustion furnaces are widely used for power generation despite coal combustion involves abundant pollutant emissions. Particularly fine particles (size smaller than 2.5 μ m), nitrogen oxides and carbon dioxide are of major concern because of their effect on climate change and human health. Alternative reactor configurations are being investigated to enhance energy efficiency and reduce greenhouse-gas and pollutant emissions. One of the most promising, at least in the near term, is atmospheric-pressure pulverized coal combustion in mixtures of oxygen and recirculated flue gas. This technology, especially attractive because of the possibility to convert existing conventional plants, requires enhanced oxygen levels (typically >28mol%) to maintain a suitable furnace exit gas temperature and sufficient heat-transfer rates to steam tubes. Its major advantage is preventing nitrogen oxide formation, due to removal of nitrogen from the air stream, and permitting a cheaper carbon dioxide sequestration.

The formation of particulate matter in coal-air combustion systems has been studied extensively [1-3] but only few researches have reported the size distribution of submicron particles generated from coal combustion in oxygen enriched conditions [4]. Particles from pulverized coal combustion are formed through at least two mechanisms resulting in a bimodal or trimodal fly ash particle size distribution. The residual-fragmentation pathway is the main responsible of the supermicron modes. The submicrometric particles are almost totally generated via vaporization of metals, their subsequent heterogeneous or homogeneous nucleation [5], usually in the form of refractory oxides, and growth

via coagulation or condensation [6]. The vaporization from parent coal may occur directly for metals with low boiling point or organically bonded to the char matrix. The refractory minerals vaporize via reduction reactions to volatile suboxides [7]. The vaporization chemistry is quite complex and can be promoted by some elements, particularly chlorine, involving the formation of volatile compounds.

The ultrafine fraction ($D < 100$ nm) of generated particles has the highest human health impact since toxicological results have shown that particles toxicity increases with decreasing particles size and depends on their numerical concentration [8]. Also the efficiency of the filtering devices decreases with decreasing particles size [9]. Despite these evidences, nanoparticles ($D < 30$ nm) are commonly not measured at the exhaust of pulverized coal furnaces mainly because it is assumed that their intense Brownian coagulation not allow surviving exhaust condition.

Recent advances in diagnostics showed that, at high temperature, the coagulation rate of nanoparticles drops dramatically orders of magnitude for size below 10nm both for carbonaceous [10] and metal nanoparticles [11-14]. Thus they could be emitted in not negligible amount. In this paper we present an experimental study of ultrafine particle formation during high temperature pulverized coal combustion as a function of the coal type, combustion gas composition and temperature.

Experimental methodology

Flat premixed flame reactor

The reactor consists of a fuel lean flat laminar premixed flame homogeneously doped with pulverized coal particles, monodisperse in size, and operated at atmospheric pressure. This reactor ensured that most of

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the particles experience similar temperatures and gas composition and velocities across the flame. To this aim the coal was previously milled in a Planetary Mono Mill (Pulverisette 6, Fritsch) using tungsten carbide bowl and balls, and subsequently suspended in ethanol. The suspension, with coal mass concentration of 1.88%, was intensively sonicated to fragment agglomerated coal particles. After this treatments, more than 95% of the suspended coal particles have size smaller than 30 μ m as verified by the dispersion granulometric analysis (Hydro 2000S, Malvern Instrument).

Three high volatile bituminous coals have been used for the experiment. The proximate and the ultimate analysis of these coals are given in tab.1. Their main differences regards ashes, volatile fraction and chlorine content. The Indonesia coal-10025 has the highest chlorine and volatile content while coal-8556 is the less volatile and chlorinated and the most enriched in ashes. The Colombia coal-10045 is an intermediate one.

Table 1
Used coals proximate and ultimate analysis.

	Coal-10025 Indonesia		Coal-10046 Colombia		Coal-8556	
	as rec'd	dry	as rec'd	dry	as rec'd	dry
Moisture [%]	3.11		4.90		2.20	
Volatile [%]	40.20	41.49	38.31	40.28	26.76	27.36
Ashes [%]	7.40	7.64	6.11	6.42	13.99	14.30
Fixed C [%]	49.29	50.87	50.68	53.3	57.64	58.94
HHV [Kcal/Kg]	7245	7478	6862	7216	6725	6725
LHV [Kcal/Kg]	6938	7198	6560	6957	6494	6500
C [%]	71.68	73.98	70.95	74.60	69.43	70.90
H [%]	5.62	5.44	5.32	5.03	4.50	4.35
N [%]	1.38	1.42	1.32	1.39	1.92	1.96
S [%]	0.53		0.40		0.44	0.44
Cl [ppm]	164		101			30.80

A Berglund-Liu-type Vibrating Orifice Aerosol Generator (VOAG model 3450, TSI) generates 80 μ m monodisperse coal suspension droplets. The VOAG syringe pump was immersed into an ultrasonic bath, thermostated at 40°C, while providing a constant suspension flow of 0.30cm³/min through a 40 μ m orifice oscillating at a frequency of 20KHz. The ultrasonic bath assures the suspension stability, preventing particles sedimentation or agglomeration into the syringe with the consequent orifice clogging, and thus the coal steady flow of 4.5mg/min.

The 80 μ m monodisperse suspension droplets, once generated, were suddenly dispersed into 2.3l/min (STP) of an oxidizing gas, preventing their coalescence and the resulting aerosol was fed to the burner. The latter was properly designed both to stabilize the ethanol fueled premixed flame and to homogeneously feed the pulverized coal particles into the reacting mixture. The burner is similar to that used by Arabi-Katbi et al. [15] and it consist of two coaxial stainless steel tubes. The inner tube (18mm ID) was used to carry the reacting dispersion of suspension droplets into the oxidizing gas. Tube walls are thermostated at 90°C to prevent ethanol condensation. A 40mm long Mullite Zirconia

honeycomb (400CPSI, CTI s.a.) was placed on the top of the inner tube. The outer tube supplies a ring (24mm ID and 34 OD) used for flowing 9l/min (STP) of the sheath Ar to minimize the perturbation by the surrounding air and reduce flame flicker. A flat plate is also placed at 90mm of height above the burner (HAB).

Ethanol completely evaporated through the honeycomb generating almost monodisperse 20 μ m coal agglomerates and the gaseous premixed fuel lean mixture. Coal particles were homogeneously carried by the gaseous stream into the premixed ethanol flame stabilized on the honeycomb mouth (~160mm/s cold gas velocity). Coal particles devolatilize, ignite and oxidize downstream of the flat flame front at the high burnt gas temperature.

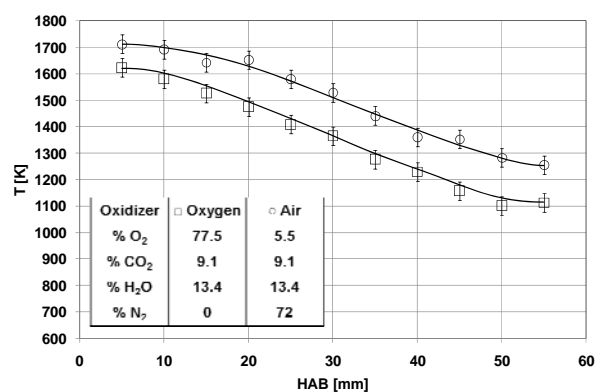


Fig. 1. Axial temperature profile versus height above the burner and molar composition of hot gasses downstream of the flat flames obtained using Oxygen (circles) and Air (squares) as oxidizer.

We report results obtained using both pure oxygen and air as oxidizing gas. The resulting flame equivalence ratios are 0.15 and 0.71, respectively. Flow reactor temperature was measured on-axis downstream the flames front using a 250 μ m Pt/Pt-13%Rh thermocouple (Type R, Omega Engineering) and corrected for the radiative losses. We fed pure ethanol, despite coal suspension droplets to avoid particles deposition on the junction while measuring temperature. The measured temperature profiles of flames obtained using oxygen and air as oxidizer are shown in fig.1. We also reported the molar composition of the hot gases in which coal particles combustion occur.

Particle residence times can be estimated assuming particles velocity equals to that of hot gases. It was about 60ms at HAB=50mm for both flames.

Differential Mobility Analysis

Generated particles Mobility Diameter (MD) distributions are on-line measured using a dilution probe and a TapCon 3/150 Differential Mobility Analyzer (DMA) system equipped with a Faraday Cup Electrometer detector [16]. The measurements were performed operating the DMA in two modality with nominal MD measurements range from 0.6nm to 28nm and from 2.1nm to 100nm, respectively.

We used horizontal tube rapid dilution probes (8mm ID, 0.5mm wall thickness) to suddenly cool and dilute sampled aerosol and transport them to the DMA [17]. The aerosol was drawn through a pinhole, drilled on the probe walls, while particle free dilution N_2 steadily flows into the tube. The Dilution Ratio (DR) was regulated performing a slight underpressure (tens of mmH₂O) in the probe, manually controlled with a rotary vane vacuum pump. The dilution N_2 flow rate is also adjusted to this aim, from 16.5 l/min to 29.51 l/min.

Three probes, with 0.3mm, 0.8mm and 1.5mm sampling orifice diameters, were used to perform DRs in different range of values. The sampling flow rates, performed at several underpressure, were previously calibrated using a 0.3mm probe [16]. The sampling flow rates performed with different pinhole diameter were evaluated from the calibrated values with a Bernoulli approximation. The corresponding performed DRs were ~30000 (0.3mm orifice, 29.5 l/min N_2), ~4000 (0.8mm orifice, 29.5l/min N_2), ~2000 (0.8mm orifice, 16.5l/min N_2) and ~1000 (1.5mm orifice, 29.5l/min N_2).

Earlier work on sooting laminar premixed flames examined the dilution probe effects and found that particle coagulation in the probe may significantly change the shape of the size distribution [18,19].

Experimentally, particle coagulation can be suppressed by varying the DR in order to attain a critical value, above which the shape of the size distribution does not change and the number eventually changes with inverse proportionality to the amount of dilution. However, following this procedure it was found that the dilution required to follow faithfully the size distribution of nanoparticles smaller than 5nm reduced the concentration of much larger particles (MD>30 nm) to concentrations right at the limit of the dynamic range of the electrometer. This effect limits somewhat the possibility of simultaneously characterizing particles in the whole ultrafine size range with the present set-up.

The highest DRs, preventing smallest nanoparticles coagulation, were used with the DMA operating in the 0.6nm to 28nm range. Measurements with the DMA operating in the nominal MD range between 2.1nm and 100nm were performed at lower dilution allowing to detect larger particles.

Particle size distribution measurements were performed positioning the sampling pinhole on axis HAB=50mm. The numerical size distribution of aerosol in flame were obtained performing the correction for the applied dilution. The particles volume fraction distribution were also obtained by multiplying the measured numeric size distribution by the particles volume considering that MD is slightly larger than the particles real diameter because of an effective diameter (0.5 nm for air) of particles-free gas sheath in the electrostatic classifier [20].

The particles number concentration and volume fraction were calculated by integrating the measured size distributions.

Results and discussions

Fig. 2 shows photographs of the Coal-10025 combustion using oxygen and air as oxidizer. Quite similar pictures were captured for the other two coals.

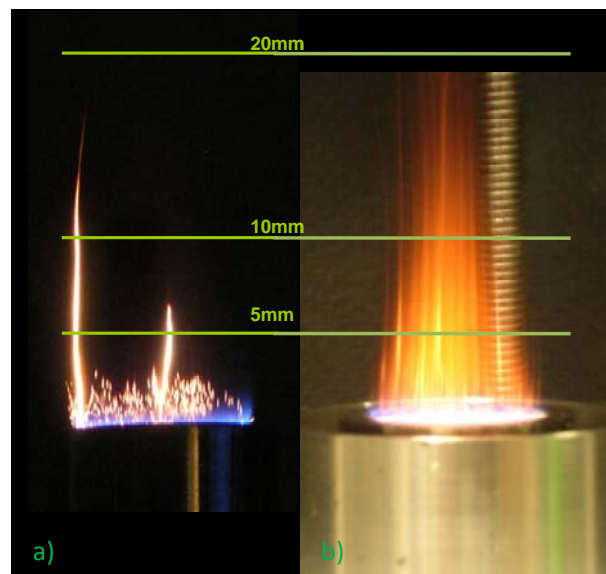


Fig. 2. Pictures of the coal doped flat premixed flames using as oxidizer: a) oxygen; b) air. The green lines indicate the length scale.

The premixed flat flame zone is evidenced by the blue light emission at the bottom of the reactor due to ethanol vapor combustion. Coal devolatilization occurs very quickly and the incandescence from the burning char particles is visible beginning from low HAB (1mm) in the reactor. The char particles combustion temperature is higher for the oxygen oxidizer case as evidenced by the incandescent intensity and color. Char burnout, evidenced by the endpoint of the incandescent particle traces, occurs much faster for the oxygen (at HAB=3mm) respect to the air oxidizer case (at HAB=30mm) because of the higher oxygen content. This behavior is in agreement with results reported in the literature [21].

The images show that coal devolatilization and char ignition and oxidation had completely run out at HAB=50mm where samplings were performed. The picture of flame obtained using oxygen also shows larger and more persistent incandescent object on the edge of the reactor. They are larger coal particles formed on burner walls and erratically carried into the flame by cold gasses. Anyway, measurements were performed on reactor axis and, thus, were not affected by these random perturbations.

Dilution Ratio effects

Measurements at several DRs were performed using oxygen as the premixed flame oxidizer. The obtained size distributions are shown in fig. 3 both in number and volume concentration. It is clear in these plots that the size distribution moves towards larger MD by decreasing DR. Moreover particles larger than 30nm are

not revealed using the highest DR because their number concentration in the probe decreases below the electrometer sensitivity.

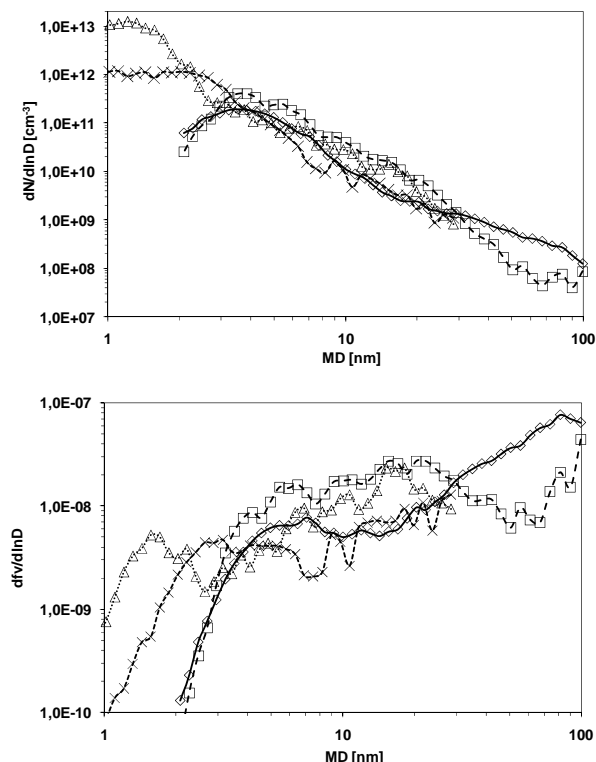


Fig. 3. Measured number (top) and volume (bottom) size distributions using the Coal-10025 and Oxygen as premixed flame oxidizer: \diamond DR=1127; \square DR=2437; \times DR=4190; \triangle DR=28789.

The biggest differences among the results are observable for size smaller than 4nm. The particles in this size range are the most abundant at the highest DR while they quickly disappear with decreasing DR because of their intense Brownian coagulation into the probe. The size distribution shows almost the same profile for MD larger than 4nm except that the accumulation of growing particles occurs at larger MD for lower DRs. The particles accumulate at 3nm, 15nm and 70nm for DR \sim 4000, \sim 2000 and \sim 1000 respectively. These sizes roughly corresponds to the mode of all the measured size distributions that can be recognize at about 4nm, 15nm and 70nm (only at the lower DRs) while a 1.5nm mode can be observed only using the highest DRs. The size distribution measured at the lowest DR allows observing the main characteristics of generated particles in the size range between 8nm and 100nm while the highest DR is required to correctly characterize also particles smaller than 10nm.

The measured number concentration of particles with size between 2nm and 100nm is $5 \cdot 10^{11} \text{cm}^{-3}$ for the highest dilution. It decreases with decreasing DRs down to $2 \cdot 10^{11} \text{cm}^{-3}$ for DR \sim 1000. Almost the totality (more than 96%) of this particles have size smaller than 30nm. The number of particles smaller than 2nm, detected using the highest DR, is of the order of $5 \cdot 10^{12} \text{cm}^{-3}$ but

decreases with increasing dilution because of coagulation.

The total ashes volume fraction can be easily evaluated performing a material balance. It is $1.7 \cdot 10^{-8}$ assuming unitary density for ashes. The measured volume fraction of ultrafine particles ($D < 100 \text{nm}$) is about $5 \cdot 10^{-8}$, regardless of used dilution. This so high value, representing 30% of ashes, is only indicative because it could be influenced by uncertainty (a factor of three) in the evaluation of the DRs. Anyway ultrafine fraction represents a large amount of all generated particles. Moreover nanoparticles smaller than 30nm are also not negligible because they represent about a third of ultrafine particles volume. The particles smaller than 2nm, detected using the highest DRs, represent a small but not negligible fraction (\sim 1%) of ultrafine particles.

These preliminary results do not allow distinguishing the amount of volatile matter at flame temperature nucleating into the probe because of sudden dilution respect to that nucleated into the flame. The high cooling rate (order of 10^5Ks^{-1}) that gas suffer into the probe involve its homogeneous nucleation rather than condensation [5]. For this reason this volatile matter is probably responsible for the smallest mode of the size distributions, with modal MD of 1.5nm, detected at the highest DRs and giving a small contribution to total particles volume fraction.

Oxidizer gas effects

Figure 4 shows a comparison between the size distributions measured using oxygen and air as oxidant. The size distributions were obtained at DR \sim 1000 to focus on a wider size range. Similar comparative discussion could be performed on size distribution measured at higher DRs not reported in this paper.

Both the size distributions show a quite similar shape. The numerical concentration distributions decrease of about three orders of magnitude moving from MD=4nm up to MD=100nm showing three modes centered at about 4nm, 15nm and 70nm. Particles smaller than 3nm that are slightly less numerous in the air oxidant case but the main difference regards the relative contribution of the modes centered at 15nm and 70nm. The size distribution of particles generated using air as oxidizer shows more distinctly this two modes with a slight larger amount of particles in the 15nm mode and slightly less in the 70nm mode respect to that formed using oxygen as oxidizer. This is more evident observing the volumetric size distributions.

The number concentration and volume fraction of measured ultrafine particles ($D < 100 \text{nm}$) are almost the same ($2 \cdot 10^{11} \text{cm}^{-3}$ and $5 \cdot 10^{-8}$, respectively) using both the oxidizer. Nanoparticles smaller than 30nm represent almost the total number of ultrafine particles (\sim 99.5%) using both the oxidize. Nanoparticles volume fraction is also not negligible and it is a larger fraction of ultrafine particles volume fraction using air (40%) as oxidizer respect to oxygen (20%).

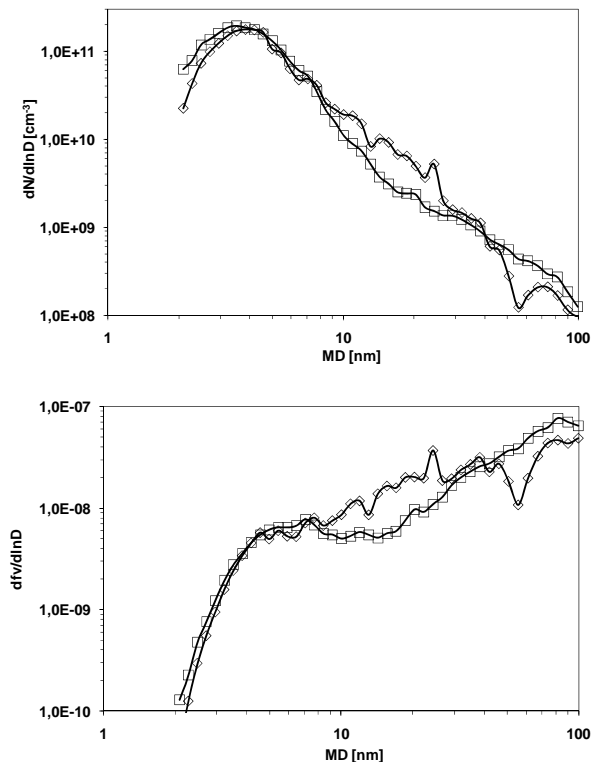


Fig. 4. Measured number (top) and volume (bottom) size distributions using the Coal-10025, DR ~ 1000 and both the premixed flame oxidizer: \square Oxygen; \diamond Air.

The differences between the two cases are very small considering the big differences of the condition in which coal combustion occurs. First of all the gas temperature differs of about 100K. Moreover the oxygen concentration is more than fourteen time lower using air respect to oxygen as oxidizer. Consequently coal combustion is much slower and occurs at lower temperature as qualitatively indicated by the flames observation. The amount of nanoparticles with size smaller than 30nm results to be a considerable fraction of generated particles both in number and volume terms, regardless of operative conditions.

Coal type effect

Measurements at the highest DRs were performed using the three different types of coal. The highest dilution allows to clearly distinguish nanoparticles smaller than 30nm.

Figure 5 show the measured number and volume size distributions. We measured almost the same size distribution despite the differences in the type of used coal. Three modes, at 1.5nm, 5nm and 15nm, are distinguishable. The slight differences regard a lower amount of particles in the 5nm mode using the coal-10046 and a higher amount of particles in the 15nm mode using the coal-10025. The coal-8556 with the highest ashes content generates a slightly larger amount of particles larger than 20nm. These differences could be simply attributed to measurements uncertainty.

The number concentration measured for particles in the size range between 2nm and 28nm is $5.0 \cdot 10^{11} \text{cm}^{-3}$ using both coal-10025 and coal-8556 and $3.9 \cdot 10^{11} \text{cm}^{-3}$ using the coal-10046. The particles smaller than 2nm are about $5.3 \cdot 10^{12} \text{cm}^{-3}$, regardless of the type of coal.

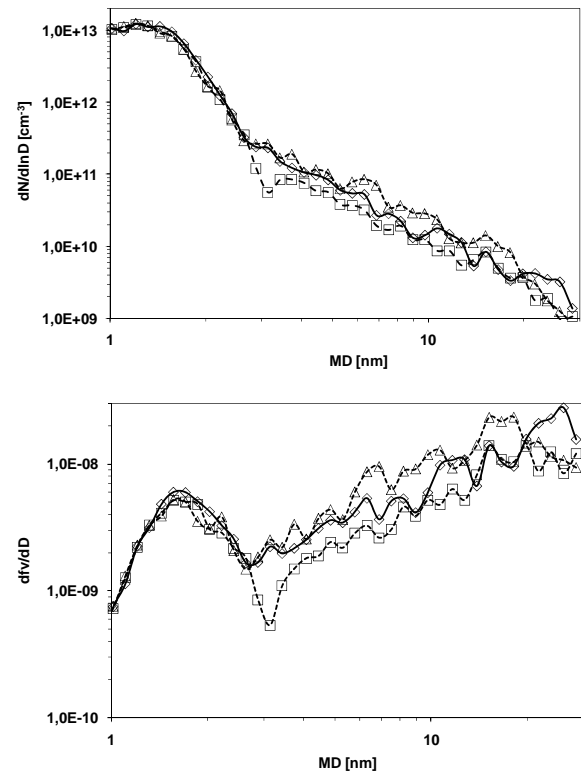


Fig. 5. Measured number (top) and volume (bottom) size distributions using DR ~ 30000 and oxygen as the premixed flame oxidizer: Δ Coal-10025 \square Coal-10046; \diamond Coal 8556.

The total ashes volume fractions, evaluated by a material balance assuming unitary density, are $1.7 \cdot 10^{-7}$, $1.4 \cdot 10^{-7}$ and $3.1 \cdot 10^{-7}$, for coal-10025, coal-10046 and coal-8556, respectively. The measured nanoparticles ($D < 28 \text{nm}$) volume fractions are 15 % ($2.6 \cdot 10^{-8}$), 12% ($1.7 \cdot 10^{-8}$) and 7.7% ($2.4 \cdot 10^{-9}$) of total fed ashes using coal-10025, coal-10046 and coal-8556, respectively. About a tenth of these values are attributable to particles smaller than 2nm. These percentage are only indicative because of DR evaluation uncertainty. Nevertheless nanoparticles represent a not negligible fraction of ashes. The higher amount of nanoparticles generated from coal-10025 probably depends on highest chlorine content respect the other two coals. The amount of nanoparticles decreases in percentage with the lower chlorine content of coal-10046 and coal-8556. Indeed chlorine promotes the vaporization nucleation pathway and thus the formation of ultrafine particles [5].

Conclusions

This study concerns the development of a new laboratory reactor allowing to investigate ultrafine particles formation during high temperature coal combustion in a wide range of operative condition in

term of temperature and gas composition. It consists of a fuel lean flat laminar premixed flame homogeneously doped with pulverized coal particles. A high resolution DMA is used to measure the size distribution of particles down to 1nm.

Measurements at several probing dilution were performed to tune the experimental technique for coal combustion occurring in an oxygen enriched and poor gas mixture. Three types of sub bituminous coals were tested. The results clearly showed that the particles nucleation modes are characterized by a size distribution almost independent from the type of coal and from the combustion condition, at least in the investigated cases. Nanoparticles smaller than 30nm are the most abundant in number and they also give a not negligible contribution to total ashes volume fraction.

Acknowledgments

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