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Ignition and explosion of nanopowders: something new under the dust

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Abstract. This work deals with the study of ignition and explosion characteristics of nanoparticles. It has been carried out on various powders: zinc, aluminum, carbon blacks… Specific behaviours have been highlighted during the first phase of this project (Nanosafe 2). For instance, it has been demonstrated that there mainly exists two combustion regimes that are either kinetically controlled, for small size particles, or diffusion controlled, for large size particles (generally with diameters greater than 1 or 2 μm). It has been found that as the particle size decreases, minimum ignition temperature and minimum ignition energy decrease (even lower than 1 mJ), indicating higher potential inflammation and explosion risks for metallic nanopowders. Moreover, the presence of agglomerates in the nanopowders could modify their reactivity. Thus, the explosion severity of Al powders tends to increase as the specific surface area decreases, before reaching a peak for 1 μm particle size.
These results are essential for industries producing or handling nanopowders in order to propose/design new and proper prevention and protection means. Nevertheless, the validity of the classical characterization tools with regard to nanopowders should be discussed. For example, the experimental laminar flame velocity of Al dusts has been compared to a theoretical one, determined by Huang's model, which assumes that the propagation of the flame is run mainly by conduction. It has shown a good agreement. However, under certain conditions, the Al flame propagation is expected to be mainly conducted by radiation. Two hypotheses can then be made. On the one hand, it can be assumed that the 20 L sphere probably disturbs the flame propagation and thermal mechanisms by absorbing radiation (wall quenching effect). On the other hand, it has been observed, thanks to the use of a high speed camera that the preheating zone is smaller for some nanopowders than for micro-particles (figure below). It could notably be explained by the fact that the flame radiation is absorbed by the cloud of unburnt Al nanopowders.
Several other factors may have an impact on the explosion severity. If these points are correctly addressed, it will be possible to get more reliable ignition and explosion characteristics.

1. Introduction

Nowadays, nanomaterials are widely used in the industrial and research fields. As the nanomaterial production and use are going to increase, there will be more and more associated risks. But the knowledge about the hazards related to these new materials is currently limited: if some studies are

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dedicated to the determination of their potential toxicity, only very few ones concern nanopowders fires and explosions [1]. In fact, since 2002 [2], one could quote the recent extensive studies performed by the EU NANOSAFE2 project [3] during the 2005-2009 period and more recently by Wu in Taiwan in 2009-2010 [4,5].

Obviously, dust explosion should not be neglected when the dusts are combustible [6]. However, literature studies concerning the evaluation of explosion and flammability risks of micron-sized powders do not enable to evaluate the fire and explosion risk probabilities and gravities of nanopowders.

As a consequence, the main goal of this work is to study explosion and ignition risks related to nanopowders. In fact, the tested powders are more "nanostructured particles" composed by agglomerated nanoparticles, than genuine "nanopowders" with a particle size lower than 100 nm. Existing analytical and methodological tools (such as a 20L explosion sphere, the modified Hartmann tube and specific procedures, standards, etc.) initially designed to evaluate dust explosion hazards related to micropowders have been tested in order to determine their validity to assess the ignition sensitivity and explosion severity of nanopowders. Experimental results were then compared with the explosion data obtained for micron-sized powders. We will mainly focus this paper on the cases of metallic nanopowders (aluminium and zinc). Carbon nanotubes and carbon blacks have also been studied. The main results obtained with these compounds will not be presented here in detail as they have been previously published [7]. However, they will be commented and compared with those obtained with metallic powders. Other compounds such as silicon carbide have also been tested but will not be presented here. Finally, this article will specifically focus on the evaluation of the validity of the 20L sphere. The hypotheses given in a previous paper [8] will be discussed in the light of recent experiments.

2. Materials and methods

Zinc and aluminium micron-sized powders were purchased from Goodfellow and zinc nanopowders from Inframat Advanced Materials. Carbon black, multiwalled carbon nanotubes and aluminium nanopowders were provided by industrial partners.

In order to have good reproducibility between the different tests, the carbonaceous nanomaterials and zinc powders were systematically dried at 50°C under dynamic vacuum of 1 Pa during 15 minutes and then under a static vacuum of 10 Pa during 4 hours before handling. The aluminium nanopowders were tested directly as they were initially stored under dry argon atmosphere.

Ignition sensitivity is often referred to as a combination of three parameters characterizing the ability of the particles to be ignited and to generate explosions. These parameters are the following ones: the minimum ignition energy (MIE), the minimum ignition temperature (MIT) and the minimum explosive concentration (MEC). Here, only the variations of minimum ignition energy will be considered. In our study, MIE was determined using a modified Hartmann tube (Mike 3 - Kühner AG) in accordance with IEC standard 1241-2-3.

The flame propagation has also been studied by using the same apparatus. The flame propagation process was recorded by a high speed video camera (Phantom V9 – up to 15000 pps). The relation between the measured flame speed and the burning velocity could be expressed as a function of the front flame surface and the area of the tube cross section. The flame speed dependence on the variation of the gas densities has also been taken into account in order to determine the laminar burning velocity.

The measurements of dust explosion severity, i.e. P_{max} (maximum overpressure), and $(dP/dt)_{max}$, (maximum rate of pressure rise), were performed in a 20 L spherical vessel in accordance with the EN 14034 standard. These parameters were finally obtained through a number of tests with step-wise increase in dust concentration.

3. Particles agglomeration

It was observed by TEM that carbon blacks tend to form agglomerates (weak cohesion forces) but also aggregates (strong cohesive forces) [3]. For aluminium and zinc samples, MEB observations as well as particle-size distributions measurements performed in isopropanol by a laser light diffraction analyzer (Mastersizer, Malvern Instrument), have shown that the agglomeration level is lower than for carbonaceous compounds. Nevertheless, a few agglomerates of 20 to 30 µm could be observed, for single aluminium nanoparticles of 100 nm diameter. The specific surface area was determined for the nanoparticles by BET measurements. Results are summarized in the table 1.

Table 1. Particles size characteristics of the samples

Nanopowders	BET specific surface area (m^2/g)	Particle mean diameter (µm)
Aluminium 100 nm	23	0,17
Aluminium 200 nm	10.5	0,28
Aluminium 3 - sample A	0.74	3
Aluminium 7 - sample B	0.25	7
Aluminium 27 - sample C	0.11	27
Aluminium 42 - sample D	0.08	42
Zinc 120 nm	19	120
Zn 38 µm	-	38

The powder densities have been measured with an helium pycnometer. By coupling the results obtained by BET and helium pycnometer, an average primary grain size can be calculated by considering that particles are isolated, cylindrical (or) spherical with a monodisperse size distribution. For well dispersed particles, such as metallic powders, the diameter d_{BET} could be linked to the Sauter diameter.

Other characteristics should be determined for a better understanding of the nanoparticles suspension behavior in a carrier gas. The dustiness measures could give a good estimation of the ability of the powder to be dispersed, but a force assessment (Van der Waals, electrostatic, capillary forces…) is also required to characterize the energy applied to powders to deagglomerate. It mostly depends on the surface properties of the nanoparticles (surface reactivity, particle diameter ...). A study is carried out at present in this field.

4. Nanoparticles ignition

Various authors have shown that the combustion time of large aluminium particles varies in a d^n law with n varying between 1.5 et 2 [9], which is typical of a shrinking core combustion model under diffusion control. In the case of nanometric particles and for temperatures lower than 1000°C, authors determined that the combustion time obeys a d^n law with n equals to 1.6 [10]. This model would indicate a faster reaction with regard to a d_p^2 expression which could be imparted to a specific effect of the alumina covering layer [9]. These authors explain this behaviour by a facilitated diffusion of aluminium through this layer, probably due to curvature effects. Another study of nanoparticles states that n index equals to 0.3 [11], which would indicate even faster reactions.

To sum up, the difference of combustion mechanisms between the nano and micron-sized particles can be understood as the result of two different regimes (a diffusion controlled and a kinetically controlled regimes). For aluminium nanoparticles, oxidation at low temperatures (lower than 1000°C) is ruled by diffusion mechanisms. It should be stressed that the change of combustion regime is observed above the nanometric size range [3].

Previous studies have also demonstrated that as the aluminium particle size decreases in the nanometric range, the inflammation temperature decreases (from 2300 K for 100 µm particles to 1200 K for 100 nm). It suggests that inflammation or explosion risks are inherently higher for nanopowders [12]. This behaviour has also been confirmed by minimum ignition energies measurements for aluminium and zinc particles (table 2). It could be noted that nanopowders have very high ignition sensitivities (notably low MIE, even lower than 1 mJ for aluminium) compared to those of micro-particles.

Table 2. Minimum ignition energies of Al and Zn particles

Particles	Al/42 µm	Al/3 µm	Al/200 nm	Al/100 nm	Zn/38 µm	Zn/120 nm
MIE (mJ)	440	14	7	< 1	> 1000	19

A simple model can be developed to take the variation of MIE as a function of the particle size into account. Thus, the MIE can be defined as the energy required for a given cloud of particles to sustain ignition and inflammation in a medium at an initial temperature T_u while the burning medium is at a final temperature T_{ad}, the flame adiabatic temperature [3]. Hence, one can write, for a spherical volume element of diameter D_c (critical diameter for which the "Van't Hoff and Taffanel Le Floch" conditions apply):

$$\int_{T_u}^{T_{ad}} \bar{\rho}.\frac{\pi}{6} D_c{}^3 .\overline{C_p}.dT = \int_0^{\tau_b} \left(Q_{generated} + Q_{ignition} - Q_{lost}\right).dt$$

where Q is an heat, T_{ad} and T_u, the adiabatic flame temperature and the unburned gases temperature, $\bar{\rho}$ and $\overline{C_p}$ are respectively the average density and heat capacity of the volume element. At ignition, $Q_{ignition}$ equals the MIE. By considering the mass conservation, the MIE could be described by the following relationship:

$$MIE \approx \frac{\pi}{6}.\overline{\rho_u}.\overline{C_p}.\frac{T_u}{T_{ad}}.\left(T_{ad} - T_u\right).D_c^3$$

By introducing τ_b, the combustion time, it can be assumed that: $MIE \propto \tau_b{}^{3/2}$. For the two different combustion regimes, one obtains an MIE which depends on dp^3 for kinetically controlled regime ($\tau_p \propto d_p{}^2$), which is consistent with Kalkert and Schecker's model [6], or on $d_p{}^{3/2}$ for the diffusion controlled regime ($\tau_p \propto d_p$). These two dependencies are in accordance with the variation of MIE expressed in table 2. For aluminium particles, the transition between the oxidation regimes occurs on a diameter range from 400 to 2000 nm. No experimental data are available for the carbonaceous particles, their MIE being greater than 1 J, which exceeds the limit of the modified Hartmann tube. Nevertheless, it has been demonstrated that the minimal ignition temperature of a carbonaceous particle cloud decreases and then increases as a function of the particle size [3]. The presence of this minimum value is probably due to the fact that, at lower particle sizes, there are stronger attractive interactions between particles. As a result, agglomeration/aggregation phenomena occur. This amount of energy should be added during the ignition process in order to oxidize the powders, which could explain the increase of the MIT for very small particles.

5. Explosion severity

In the literature, it was often observed that dust becomes more and more explosive up to a certain size limit at which it will plateau. For coal, [6] reports that the limiting particle size, below which there is no further increase in the maximum explosion pressure or the rate of pressure rise, is about 50 µm. [13] reports a plateau at a particle size of roughly 50 µm for flour, about 40 µm for methylcellulose, and polyethylene, but shows no sign of threshold for PVC. For metal dusts, especially the more

reactive metals like aluminium and magnesium, the limiting particle size should even be smaller than for other dusts. No size limit could be found in the literature [6] for this kind of materials.

Experimental results related to the explosion severity of tested nanopowders are summarized in the table 2. The influence of the specific surface area on the explosion severity of zinc, aluminium and carbonaceous compounds is illustrated on figures 1 and 2.

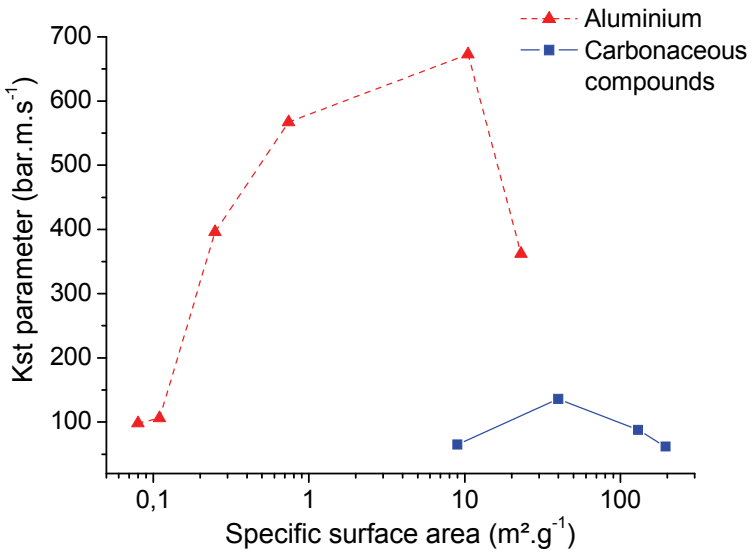


Figure 1. Evolution of the Kst parameter for aluminium and carbonaceous compounds as a function of the specific surface area

Table 3. Explosion severity parameters of metallic powders

Powders	P_{max} (barg)	$(dP/dt)_{max}$ (bar/s)	Kst (bar.m.s^{-1})	Explosivity Class St
Aluminium 100 nm	8.2	1340	362	St3
Aluminium 200 nm	9.5	2420	673	St3
Aluminium 3 µm	9.8	2090	567	St3
Aluminium 7 µm	9.1	1460	396	St3
Aluminium 27 µm	7.5	400	106	St1
Aluminium 42 µm	7.2	360	98	St1
Zinc 120 nm	3.9	223	61	St1
Zn 40 µm	3.7	71	19	St1

The explosion severity tends to increase as the particle size decreases/the specific surface area decreases, before reaching a peak for 1 µm particle size in the case of aluminium powders (approximately 2 m²/g for P_{max}) [14]. After this peak has been reached, the explosion severity finally tends to decrease as the particle size decreases/the specific surface area increases. If we focused on the aluminium nanopowders, we observed that the 200 nm particles explode more violently than the 100 nm particles. This is probably due to the degree of passivation of the aluminium that could be different for these two kinds of particles, and also due to the degree of agglomeration, which is probably more important for the 100 nm particles. A similar behavior has been observed for carbonaceous compounds: some carbon blacks with a rather high specific surface area exhibit a low explosivity, which is due to the size of their agglomerates (up to 300 µm in some cases) [3]. Once again, as for micro-particle ignition, the agglomeration phenomenon has a major influence on the particle

explosivity and the explosion characteristics of nanopowders, especially their maximum rates of pressure rise are sometimes lower than those of powders with diameters lower than about 10 µm (table 3).

For aluminium, the same behavior observed for the maximum explosion pressure is obtained for the maximum rate of pressure rise, which presents a maximum at approximately 4 $m^2.g^{-1}$ of specific surface area [3]. The same evolution is observed for the carbonaceous compounds but with a peak for higher specific surface area (figure 1).

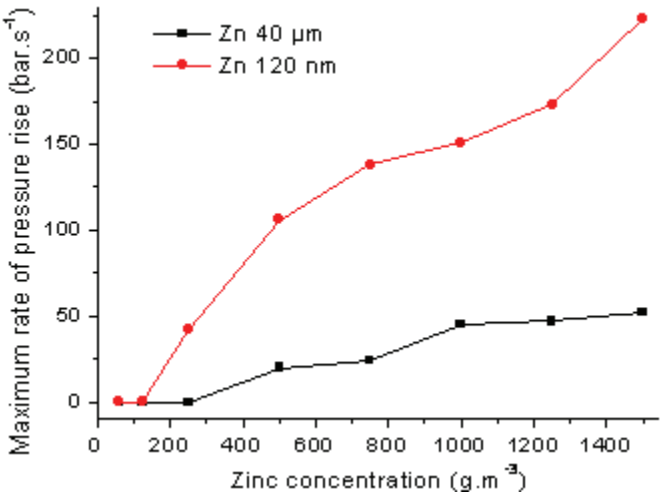


Figure 2. dP/dtm of zinc nano and micro particles versus dust concentration

Only two different particle sizes distributions of zinc have been tested. It could be seen on the figure 2 that the maximum of the dP/dt_m is not reached for concentrations ranging from 0 to 1500 $g.m^{-3}$. Nevertheless, variations of more than 300 % are observed between the maximum rates of pressure rise of zinc micro and nano powders. Tests should be carried out with 1 or 2 µm zinc powders in order to confirm the previous trends.

Flame velocities of 0.15, 0.19 and 0.69 $m.s^{-1}$ have been measured for aluminium dusts of 42, 0.12 and 3 µm diameters respectively; which confirms the previous findings (figure 3). Flame velocities of zinc particles have not been compared because the micro-particles do not ignite for ignition energies lower than 1 J, which is the maximum energy available with this apparatus.

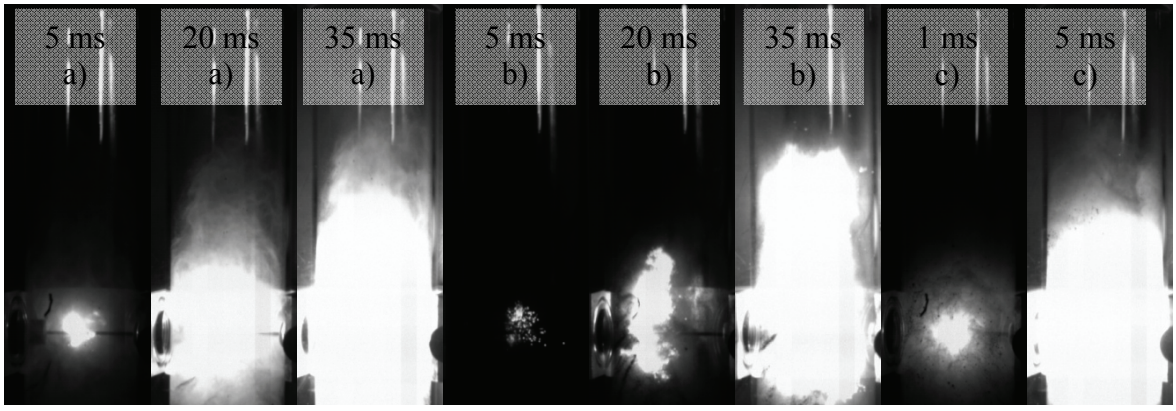


Figure 3. Flame propagation of Al dust explosion in a modified Hartmann tube. Dust concentration: 900 $g.m^{-3}$; ignition energy: 1000 mJ; a) Al 42 µm, b) Al 120 nm, c) Al 3 µm.

It should be stressed that the specific surface area of the particles should not be considered as the only factor influencing the particle explosivity, the chemical composition and the structure of the compound should also be taken into account.

6. Standards and set-up validity

So far, no literature data enables us to validate experimental data for nanopowders, it is necessary to evaluate the validity of the experimental setups and procedures, and especially the validity of the 20 L explosion sphere. Thus, some potential issues influencing the reliability of the experimental results will be developed in the following chapter [14].

6.1. Pre-ignition of the metal nanopowders.

It was observed that aluminium nanopowders were sometimes pre-ignited during the injection process into the 20 L explosion sphere [14]. Pre-ignition phenomenon may lead to an increase of the initial temperature and initial pressure as well as an increase of alumina content. However, no pressure increase was experimentally recorded. The potential heat produced by the pre-ignition phenomenon might be directly transferred through the walls of the dust storage within the vessel. Therefore, it may be considered that pre-ignition phenomenon only leads to an increase of the alumina content within the aluminium nanopowders as well as a decrease in the initial oxygen content. Consequently, it may be assumed that explosion severity may be lowered by pre-ignition.

6.2. Intrinsic oxide content.

Alumina or zinc oxides can decrease absolute explosion severity of these powders. As alumina is produced during the injection process of aluminium nanopowders in the explosion sphere, it is necessary to reduce the impact of an inadequate storage. In this way, the argon sealed bag provided by the industrial partner were opened 1 h before the explosion parameters were determined. Thus it can be considered that the experimental results are just slightly influenced by the initial alumina content.

6.3. Efficiency of the dispersion process during injection.

The degree of dispersion of nanopowders may also decrease the explosion severity. This effect was previously shown by Eckhoff [6]. The authors investigated the influence of the degree of dispersion of maize starch grains on the rate of pressure rise during explosions in the 1.2 L Hartmann bomb and showed a consistent increase of (dP/dt), as the effective particle size decreases, when the agglomerates sizes within the tested materials were lowered. Therefore, measured explosion safety parameters may only be reliable in order to assess explosion risks related to the storage or cleaning operations. The explosion risk related to the industrial production process may be underestimated as there are often well-dispersed nanoparticles within the process. However, it was also shown recently by [15,16], that agglomeration could make ignition of nanoparticles easier than for deagglomerated nanoparticles. Consequently it may be assumed that explosion severity could be increased by considering a simple flame propagation model such as Cassel's one [6].

6.4. Quenching effect of the 20L explosion sphere.

Flame propagation mechanism may also be potentially affected by nanoparticles in the dust cloud. For instance, it was shown experimentally by [17] that an aluminium flame could propagate under a mechanism mainly conducted by radiation rather than by conduction. It can then be assumed that the explosion severity is lowered due to heat losses of the flame to the walls of the explosion sphere. As it currently remains difficult to determine the accurate influence of the agglomeration, pre-ignition, and intrinsic alumina content on the explosion severity, the potential quenching effect of the 20 L sphere was studied through a theoretical investigation.

6.5. Comparison between experimental results and the modeling of the laminar flame velocity.
The explosion pressure P_m and rate of pressure rise $(dP/dt)_m$ were combined through the semi-empirical relation of Silvestrini [18] in order to calculate an experimental laminar flame velocity. It has been compared to a theoretical one, determined by the model of Huang [19], which was initially established to predict laminar flame velocity in an aluminium dust cloud. This model assumes that the propagation of the flame is run mainly by conduction.

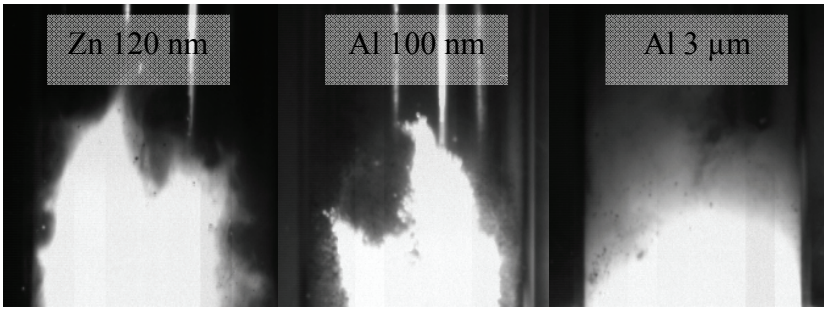


Figure 4. Flame propagation of zinc and aluminium dust explosion in a modified Hartmann tube. 900 $g.m^{-3}$ of powder at 30 mJ for tv of 120 ms. Nano Zn - Nano Al - 15µ Al

It has been demonstrated that a Huang-like model could be used to represent our experimental data, i.e. that the propagation flame is mainly conducted by conduction within the 20L sphere. However, it should be reminded that it is experimentally [17] and theoretically [20,3] expected that the flame propagation within nanodust cloud is probably mainly conducted by radiation.

Two hypotheses can thus be made. One the one hand, it can be reasonably assumed that the 20 L sphere does not enable to get reliable data as it probably disturbs the flame propagation and thermal mechanisms by absorbing radiation (quenching due to wall effect). On the other hand, it has been observed, thanks to the use of a high speed camera and the visualization of the propagating flame edge structure (figure 4) that the preheated zone is smaller for nanopowders (100 nm) than for micro-particles. It could notably be explained by the fact that the flame radiation is absorbed by the cloud of unburnt aluminium nanopowders. A bichromatic pyrometer will also be used in order to check if this modification of the flame behavior in the visible range could be due to a change in the radiation wavelengths (flame temperature modification). Similar tests have been carried out on 200 nm particles and more radiations have been observed than in the 100 nm case.

Figure 5 sums up some key parameters governing the flame propagation phenomenon. The difference of combustion mechanisms between the nano and micron-sized particles can be understood as the result of two different regimes (a diffusion controlled regime for large powders and a kinetically controlled regime for small ones). It is clearly shown on figure 5 that the diffusion controlled regime ($1/d_p$ law) can explain the evolution of flame velocity for large particles. Concerning the heat transfers, it is interesting to consider the Mie parameter $\pi.d_p/\lambda$ (λ being the wavelength) and the balance between light scattering and absorption for nanopowders (change from Mie to Rayleigh theory). In the case of aluminium combustion, the limit is obtained for 100 to 120 nm particles. This could give information on the modification of radiation and light absorption observed between 100 and 200 nm powders. In figure 5, the flame velocities presented are those deduced from Silvestrini's law from the 20 L sphere explosivity data [18]. They differ from those previously quoted and obtained by using the modified Hartmann tube and considering both gas expansion and flame stretching. Thus, the influence of the vessel quenching could not be neglected.

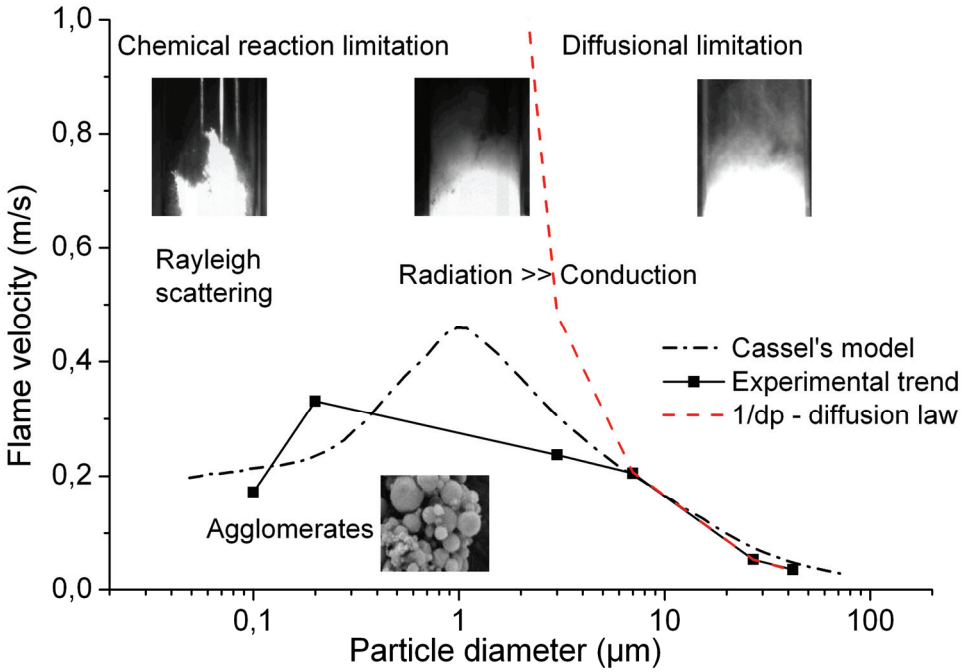


Figure 5. Influence of the Al particle size on the flame velocities of aluminium dust clouds: key parameters

7. Conclusions

During this study, it has been found that the inflammation risk of aluminium powders greatly increases with the use of nanopowders, thus delineating high potentials for ignition risks in facilities manufacturing such powders. Nevertheless, the presence of agglomerates could reduce the reactivity of the powders, and therefore, their explosion severity.

Some experimental issues should be considered in order to characterize the ignition and explosion risks of such powders. The explosion severity of nanopowders may be affected by pre-ignition phenomenon, the intrinsic alumina content of the aluminium, the agglomeration/aggregation level of the nanopowders and the quenching effect of the 20 L explosion sphere. Consequently, some improvements are needed to avoid the premature ignition of ignition sensitive nanopowders and to reproduce the agglomeration level observed in the industrial process. In all cases, it should be reminded that extrapolation of the obtained data and their use for vent sizing purpose, might be impossible by the way of the widely used “cubic law” [6]. In fact, this semi-empirical relationship assumes that the flame thickness is relatively thin; this thickness being increased by the radiation phenomenon. It induces that this law could become null and void for flame propagation mainly governed by radiation. Nevertheless, the importance of the radiation phenomenon and light absorption in the dust cloud should be discussed as a function of the particle size. Finally, the impacts of agglomeration on explosion severity and sensitivity for nanopowders are still not fully understood and should be further explored in the future. For instance, the particle size distribution of the dust dispersed in air will be determined; the results will obviously be different from those obtained by laser light diffraction in a liquid suspension or by SEM or TEM on a dust layer.

Acknowledgements

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