Effect of carbon black nanoparticles on methane/air explosions: Influence at low initial turbulence

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Abstract. Nanoparticles are widely used in industrial applications as additives to modify materials properties such as resistance, surface, rheology or UV-radiation. As a consequence, the quantification and characterization of nanoparticles have become almost compulsory, including the understanding of the risks associated to their use. Since a few years ago, several studies of dust explosion properties involving nano-sized powder have been published. During the production and industrial use of nanoparticles, simultaneous presence of gas / vapor / solvents and dispersed nanoparticles mixtures might be obtained, increasing the risk of a hybrid mixture explosion. The aim of this work is to study the severity of the explosion of carbon black nanoparticles/methane mixtures and understand the influence of adding nanopowders on the behavior of the gas explosions. These results are also useful to understand the influence of soot on the efficiency of the gas combustion. Two grades of carbon black nanoparticles (ranging from 20 to 300 nm average diameter) have been mixed with methane. Tests have been performed on these mixtures in a standard 20 L explosion sphere. Regarding the scale precision, the lowest concentration of carbon black nanoparticles was set at 0.5 g.m⁻³. Tests were also performed at 2.5 g.m⁻³, which is still far below 60 g.m⁻³, the minimum explosive concentration of such powders previously determined in our laboratory. The influence of carbon black particles on the severity of the explosions has been compared to that of pure gas. It appears that the use of carbon black nanoparticles increases the explosion overpressure for lean methane mixtures at low initial turbulences by c. 10%. Similar results were obtained for high initial turbulent systems. Therefore, it seems that carbon black nanoparticles have an impact on the severity of the explosion even for quiescent systems, as opposed to systems involving micro-sized powders that require dispersion at high turbulence levels. Concerning the increment in the maximum rate of pressure rise, the addition of carbon black nanoparticles increased it by a factor of 1.15 in the case of lean fuel mixture. However, this behavior is only observed at high initial turbulence levels. The increment on the maximum rate of pressure rise is higher for powders with lower elementary particle diameter, which is notably due to the fragmentation phenomena that promotes the heat exchange.

1. Introduction

Nanoparticles have been progressively used in a wider variety of applications because of the different properties they have compared to those of bulk materials [1]. Nanoparticles are used to modify certain properties (particularly resistance, rheology, hardness, and magnetization) and, due to their greater
surface area, they appear better suited for catalysis and biological applications [2]. The past few years have witnessed a transition in the production of certain products from traditional processes to nanoscale level manufacturing. Such materials are now widely used in cosmetics, paints, tires, catalysts and pharmaceuticals [3, 4]. The European Commission suggests that a nanomaterial should be defined as a material in which more than 50% of the particles in the number size distribution (in unbound state, in agglomerates or aggregates) have a size between 1 nm and 100 nm [5]. The rapid increase in nanoparticles applications have boosted the number of studies regarding the toxicity and explosion hazards of combustible nanoparticles dust [1, 3, 6]. However, there is still a limited amount of data and analysis on the explosion properties in this field [7]. Some authors focused their researches on characterizing the difference of the explosive properties between nano- and micro- dust. Dobashi [8], for instance, found that for coal, polyethylene and PVC, the explosion violence and the ignitability of powders are significantly increased when the particle size decreases until a certain size limit below which the explosion risk plateaus. The explosion severity and ignitability have also been studied for metallic dust of different sizes. Jiang et al. [9] measured the explosion severity in the standard 20L sphere for a mean aluminum particle diameter between 75 µm and 100 nm, obtaining more violent explosions for smaller particles. Similarly, Mittal [10] found that the explosion severity increases when the diameter of the particles decreases from 125 µm to 400 nm, but the violence of the explosion is lower for magnesium dust in the 30 - 200 nm range. However, the likelihood of an explosion increases significantly for the nano-range magnesium with respect to the minimum explosive energy (MIE) and the limiting oxygen concentration [10]. Similar results have been obtained for titanium, aluminum, carbon nanotubes and carbon black, powders whose ignitability increases as the particle diameter decreases and whose explosion severity increases until a plateau is reached [11-14]. In fact, this plateau and the similar explosive behavior of some micrometric and nanometric particles could be explained by the difficulty to obtain a homogeneous dispersion of elementary nanoparticles at high concentrations (greater than the minimum explosive concentration). This is mainly due to agglomeration: during the dust dispersion, the primary particles subjected to attractive forces will be rapidly transformed into larger structures [4, 15-17]. Ignition of a nano-dust cloud could be achieved before any agglomeration phenomena happens at high initial turbulence levels and at a short ignition delay time, conditions that are difficult to meet under normal industrial operations [15]. Even though nano-dust explosions have been studied in the past years, further and deeper analysis is required to understand the complex heat radiation and the behavior of the front flame. Dispersed nanometric dust may have an influence in complex solid-gas mixtures, simultaneous presence of gas/vapor/solvents and dispersed mixtures could be obtained during the production and industrial use of nanoparticles, increasing the risk of a hybrid mixture explosion. Moreover, such mixtures are systematically encountered during the combustion of organic dust, notably on the devolatilization phase, leading to a hybrid mixture of pyrolysis gases and char/unburnt powder [18].

In a previous study [19], the influence of low concentration of carbon black nanoparticles on gas mixture explosion has been analyzed. The flame front is modified from a semi-parabolic to a non-uniform shape, the latter caused by multiple perturbations occasioned by the burnt particles [19]. In the present study, the influence of the turbulence on the explosion severity and on the reaction products of carbon black/methane/air is analyzed. This work aims to document the influence of nanoparticles dispersion on hybrid gas/solid mixtures to understand the complex heat transfer, the changes on the laminar flame velocity and the possible effects of dispersed nano-carbon particles (such as soot nuclei) in the combustion chemical reaction.

2. Materials and Methods
In this study, Printex XE2 and Corax N550 (Orion) have been chosen as carbon black nanoparticles. The characteristic diameter of the powder $d_{50}$, the BET specific surface (using Brunauer-Emmett-Teller method) and the equivalent BET diameter are reported in Table 1. It appears that the primary nanoparticles are arranged in agglomerates of micrometric size. Moreover, Printex XE2 presents a much
larger specific surface area than Corax N550 does, which may have an impact on their respective reactivity and physical properties.

Table 1. Main characteristics of the nanopowders [13]

| Nanopowders | BET specific surface area (m²/g) | BET diameter (nm) | d₅₀ (µm) |
|-------------|---------------------------------|------------------|----------|
| Printex XE2 | 950                              | 3                | 10       |
| Corax N550  | 40                               | 75               | 15       |

The explosion severity of nanopowders/methane hybrid mixtures has been assessed by measuring the maximum overpressure Pₘₐₓ and the maximum rate of pressure rise (dP/dt)ₘₐₓ. The measurements of the explosion severity were performed in a 20-liter spherical vessel. In order to study the influence of an initial concentration of soot in a methane/air mixture, low concentrations of carbon black particles were chosen. With regard to the scale precision, the lowest concentration of carbon black particles was set at 0.5 g.m⁻³. In addition, tests were also performed at 2.5 g.m⁻³, which is far below 60 g.m⁻³, the minimum explosive concentration of such powders [13]. Samples of burnt gases were taken without dilution and analyzed by micro gas-chromatography (Varian 490 MGC). Flame propagation test have been also performed but will not be detailed here.

![Image of Printex XE2 and Corax N550](image_url)

Figure 1. Scanning Electron Microscopy images of Printex XE2 (left) and Corax N550 (right)

The influence of the initial turbulence was characterized by varying the ignition delay tᵧ (i.e. time between the beginning of the air pulse and the ignition at 100 J with chemical igniters). The severity of the explosion depends on the turbulence level, which is necessary for the dust dispersion especially for microparticles. The influence of the initial turbulence is studied by changing the delay time between the beginning of the dust dispersion and the ignition of the mixture. The intensity fluctuations estimated using the PIV method were 3.4 and 1.04 m.s⁻¹ at 60 and 120 ms, respectively. Due to the low settling velocity of nanoparticles, a quiescent case, which corresponds to the addition of dust without a pulse of air, was also considered.

3. Results

3.1. Dispersion Tests

Dispersion tests have been made for Printex XE2 and Corax N550 in order to analyze the stability of a dust cloud some seconds/minutes after the injection. To accomplish this, the mobility particle size was
measured at different times after the powder injection by sequentially using a Differential Mobility Analyzer (Electrostatic Classifier – TSI 3080) and an Ultrafine Condensation Particle Counter (UCPC – TSI 3776). Twenty seconds after the dispersion, the estimated mean mobility diameters of the Printex XE2 and the Corax N550 were 245 nm and 346 nm, with a total concentration of $2.1 \times 10^5$ and $2.4 \times 10^5$ particles.cm$^{-3}$, respectively (Table 2). The DMA - UCPC results must be analyzed carefully because the particle size distribution analysis takes two minutes, time in which agglomeration and sedimentation phenomena occurs. However, the results obtained show that, even 3 minutes after the dispersion, the total concentration of dust is not negligible and the mean mobility diameter does not change with respect to the first measurement. It should be added that, due to the sampling process, a depression would be generated inside the reservoir, so a dilution with air is necessary to continue the DMA-UCPC tests which may explain the significant reduction of the total concentration of particles, in addition of the dust sedimentation phenomenon. These results suggest that the agglomeration phenomenon, even at high initial turbulent conditions, is limited because of the low concentration of dispersed dust. As consequence, it has be demonstrated that a stable dust cloud of agglomerates is generated and is still present at the ignition delay times of 60 and 120 milliseconds studied in this article.

### Table 2. Carbon Black mean mobility diameter and total concentration after dispersion

| Nanopowders | Time after dispersion (s) | Mean Mobility Diameter (nm) | Concentration (particles.cm$^{-3}$) | Ambiance Concentration (particles.cm$^{-3}$) |
|-------------|---------------------------|-----------------------------|--------------------------------------|--------------------------------------------|
| Printex XE2 | 20                        | 245                         | $2.1 \times 10^4$                    |                                             |
|             | 190                       | 241                         | $1.5 \times 10^5$                    | $6.3 \times 10^3$                          |
| Corax N550  | 20                        | 346                         | $2.4 \times 10^5$                    |                                             |
|             | 180                       | 350                         | $1.6 \times 10^5$                    |                                             |

3.2. Explosion severity and burnt gases characterization

The maximum overpressure and the maximum rate of pressure rise for hybrid mixtures methane/air/Printex XE2 for different dust concentrations at quiescent conditions are shown in Figure 2. As pointed out in a previous work [19], in the case of Corax N550, the influence of the dispersion of nanoparticles agglomerates on methane/air gas explosions depends on the fuel equivalent ratio $\varphi$. For lean fuel mixtures ($\varphi < 1$), the severity of the explosion seems to increase when the concentration of Printex XE2 is augmented from 0.5 to 2.5 g.m$^{-3}$, but it drops for rich fuel mixtures. At low fuel concentration, dust dispersion may generate a deformation of the flame surface, causing an acceleration of the flame and an increase in the explosion severity. However, for fuel rich mixtures, heat radiation transfer appears to offset the effect of particles or agglomerates on the surface of the flame, attenuating the severity of the explosion. These results show that the risk of a gas explosion may be incremented when low concentrations of carbon black nanoparticles are present in the mixtures, even in the absence of high turbulence to guarantee the total dispersion of a nano-dust cloud.
Figure 2. Influence of the carbon black concentration on the maximum overpressure - $P_{\text{max}}$ (left) and on the maximum rate of pressure rise - $\frac{dP}{dt}_{\text{max}}$ (right) for a Printex XE2/Methane/Air mixture under quiescent conditions.

For systems involving solid fuels, the initial turbulence level of the system is critical to generate an explosion and is determinant of the severity parameters. A similar behavior of the explosion severity has been observed at an initial velocity fluctuation of 1.05 m.s$^{-1}$ (see Figure 3). Nevertheless, a higher turbulence level seems to cause a higher increase in the maximal explosion overpresssure for rich fuel mixtures at lower concentrations of carbon blacks. This result suggests that the initial turbulence level increases the powder deagglomeration, thereby amplifying its effects on the flame surface and on the heat radiation transfer. Even when the turbulence level was increased, no ignition of the mixture resulted at 12% v/v methane concentration due to a turbulence quenching generated by the turbulent cloud around the ignition point. The previous result was not observed for Corax N550/methane/air mixtures [19], probably because of the greater primary particle diameter.

Figure 3. Influence of the carbon black concentration on the maximum overpressure – $P_{\text{max}}$ (left) and on the maximum rate of pressure rise – $\frac{dP}{dt}_{\text{max}}$ (right) for a Printex XE2/Methane/Air at an initial turbulence level $u' = 1.04$ m.s$^{-1}$.

Measurements of the volume fractions of the burnt gases were obtained using the micro gas-chromatography in order to analyze the effect of the carbon black and of the initial turbulence level on the chemistry of the combustion reaction. Figures 4 and 5 present the CO$_2$ volume fraction and the
CO/CO$_2$ ratio of the burnt gases for a gas mixture and hybrid mixture explosion, respectively. Under quiescent conditions, the maximum CO$_2$ volume fraction is obtained for stoichiometric conditions. The presence of an initial turbulence seems to increase the conversion of CO to CO$_2$ for lean mixtures but this conversion decreases for rich mixtures. Nevertheless, the turbulence level does not have an influence on the conversion of CO to CO$_2$ for the gas mixtures. For hybrid mixtures, the CO$_2$ volume fraction and CO/CO$_2$ ratio have similar gas explosions trends when the initial turbulence is augmented. However, the highest CO$_2$ volume fraction is obtained for the highest initial turbulence level as shown in Figure 5. The heterogeneity of the mixture increases the higher the turbulence level, generating a local equivalent ratio that may explain the difference of conversion of CO into CO$_2$ [19].

![Figure 4](image1.png)

**Figure 4.** Influence of the turbulence level on the final CO/CO$_2$ ratio (left) and on the final CO$_2$ molar fraction (right) for a Methane/Air mixture.

![Figure 5](image2.png)

**Figure 5.** Influence of the turbulence level on the final CO/CO$_2$ ratio (left) and on the final CO$_2$ molar fraction (right) for a 0.5 g.m$^{-3}$ Corax N550/Methane/Air mixture.

Comparing the explosion severity for Printex XE2/Methane/Air mixture at different initial turbulence levels (Figures 2 and 3), it appears that the maximum rate of pressure rise is greatly increased at high turbulence levels, evidencing a close relationship between the turbulence level and the chemical combustion reaction. The influence of the turbulence level and combustion interactions on the explosion severity of this type of complex mixtures should be studied to design the appropriate prevention or protection devices. The micro gas-chromatography results suggest that there is a relation between the
final molar CO$_2$ fraction and the maximum rate of pressure rise, in which the highest value of (dP/dt)$_{\text{max}}$ is obtained when the maximum conversion of CO to CO$_2$ is reached (Figure 6). Therefore, the conditions that promote the chemical reaction of oxidation of CO, as a higher initial turbulence level, will generate a higher release of chemical energy and, consequently, an increase on the maximum rate of pressure rise. Similar results were obtained regarding the influence of the carbon black dispersion on the maximum rate of pressure rise (Figure 2 - right), where the higher explosion severity was obtained when the maximum concentration of CO$_2$ was produced (Figure 7).

![Figure 6. Influence of the turbulence level on the maximum rate of pressure rise - dP/dt$_{\text{max}}$ (left) and on the final CO$_2$ molar fraction (right) for a 2.5 g.m$^{-3}$ Printex XE2/Methane/Air mixture.](image)

The presence of a carbon black cloud affects the combustion reaction, even if the amount of solid fuel is negligible with respect to the methane volume fraction. Figure 7 shows the influence of the Printex XE2 initial concentration on the CO$_2$ and CO molar fraction for hybrid mixtures explosions under quiescent conditions. The explosion severity seems to increase due to the presence of dispersed agglomerates, phenomena that appears to promote the conversion of CO$_2$ for lean fuel mixtures. In addition, this conversion rate decreases for a fuel equivalent ratio higher than 0.8. The CO production seems to increase for lean mixtures when 2.5 g.m$^{-3}$ of Printex is added to the system, outcome that is not observed when using Corax N550 dust. Firstly, this result shows again that both powders have high stability when dispersed and influence the methane/air gas explosion. However, such impact on the explosion becomes more important for powders with a higher specific surface. The stable elementary particles and dispersed agglomerates seem to affect the flame surface (observed thanks to flame propagation tests), and, because they undergo oxidation reactions, the concentration of the final gases and the violence of the reaction vary. Secondly, the negative effect on the explosion severity for rich fuel equivalent ratio may be caused by the improvement of the soot nucleation phenomenon due to primary nuclei when initial carbon particles are added into the combustible mixture.
3.3. Flame propagation velocity from 20L sphere tests

The burning velocities of carbon black nano-particles and methane/air hybrid mixtures were estimated using the thin-flame model postulated by Dahoe [20] using the explosion severity results. The model assumes that the flame is an infinitely thin flame that separates the inner burnt region and the outer unburnt gas. The rate of pressure rise and the maximal overpressure are related to the burning velocity as:

$$\frac{dP}{dt} = \frac{3(P_e - P_0)}{R_{\text{vessel}}} \left(1 - \left(\frac{P_0}{P}\right)^\gamma \frac{P_e - P}{P_e - P_0}\right) \left(\frac{P}{P_0}\right)^\gamma S_u$$

where $P_e$ is the maximal overpressure, $P_0$ is the initial vessel pressure and $S_u$ is the burning velocity.

The estimation of the burning velocities for the hybrid mixtures Printex XE2/methane/air and Corax N550/methane/air at an initial turbulence level of $u' = 1.05 \, \text{m.s}^{-1}$ are presented on Figure 8. An estimation of the burning velocity is possible by equation 1. However, it should be underlined that this relationship may underestimate the burning velocity, because the flame thickness is often not negligible at high pressure and for systems involving dispersed dust clouds. Likewise, the equation 1 suppose a perfect spherical propagation of the front flame, which may not be present because of the interactions of the flame with the internal elements of the sphere, and also, because of inertial effects created by the dispersion. Shuy et al. [21] reported a flame velocity of methane/air at stoichiometric concentrations of approximately 240 cm.s$^{-1}$ at an initial turbulence of velocity fluctuation $u' = 1 \, \text{m.s}^{-1}$, which is higher than our estimated value of 174 cm.s$^{-1}$. The addition of 0.5 g.m$^{-3}$ of Printex XE2 generates an increase of 20% on the burning velocity for fuel lean mixtures compared to gas mixtures, while a decrease is present for fuel rich mixtures. In addition, the burning velocity is considerably reduced when the carbon black concentration is augmented to 2.5 g.m$^{-3}$, even if the maximal overpressure seems to be improved with the presence of a Printex XE2 dust cloud (Figure 3). Similarly, the presence of a dust cloud of Corax N550 generates a decrease of the burning velocity with respect to gas explosions. The previous behaviour could be explained by two possible phenomena. On the one hand, the increment on the heat radiation transfer caused by the dispersed dust may generate heat losses on the propagating front and will be enhanced at a higher turbulence level. On the other hand, the cloud dust may generate changes on the flame surface, generating local flamelet regimes and reducing the overall flame velocity, which
has been observed on the flame propagation effects. Ameliorations on the experimental flame velocity at different turbulence levels must be implemented in order to avoid underestimation of the protection devices at lean fuel mixtures and overestimation at rich fuel mixtures when low concentrations of dust may be present in the system.

**Figure 8.** Burning velocity calculated by the thin flame model for Methane/ Printex XE2 /Air (left) and Methane/Corax N550/Air (right) for an initial turbulence level $u^* = 1.04 \text{ m.s}^{-1}$.

4. Conclusions

This work studies the influence of nanoparticles on the explosion severity of gas explosion. The results obtained show that, even when adding carbon blacks at low concentration (well below the minimum explosive concentration), the heat transfer phenomena and the combustion reaction can be modified. The explosion severity tests and the burnt gas characterization suggest that:

a) The cloud of nanopowders remain highly stable some minutes after the dispersion, with a constant mean mobility diameter. The dust cloud formed, notably composed of agglomerates, has a mean diameter of 240 nm for Printex XE2 and 370 nm for Corax N550.

b) The explosion of nanoparticles can be hardly obtained under quiescent conditions, despite the high stability of the dust. Nevertheless, the explosion severity of methane/air mixtures increases when low concentrations of carbon black nanoparticles are present in the system, even at quiescent conditions. In addition, at high turbulence level, the presence of carbon black nanoparticles generates a negative effect on the ignition sensitivity for fuel rich mixtures.

c) The addition of carbon black particles for fuel lean mixtures seems to promote the oxidation of CO, which will generate a higher release of chemical energy and, consequently, an increase in the maximum rate of pressure rise. The presence of carbon blacks (or as a consequence, soot) has effects on gas explosion severity which can vary as a function of the turbulence level. Such influence can lead to explosion quenching and has to be further studied in order to design appropriate prevention or protection devices as well as propose inherent safety measures.

d) The specific surface area of the dispersed particles have a great influence on the explosion severity of carbon black/ methane/ air mixtures. At high turbulence levels, the deagglomeration effects on heat transfer and flame surface are amplified for powders with higher specific surface.

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