



Effect of the nozzle type on the integrity of dust particles in standard explosion tests



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ABSTRACT

The effect of the standard dust dispersers, rebound nozzle and annular nozzle, in the 20 L explosion vessel on the particle integrity has been quantified. Dispersion tests were run and measurements of the particle size before and after dispersion and of the elastic recovery of the materials were performed. The effect of the dispersion pressure was also studied. Different dusts were tested: lycopodium, nicotinic acid, ascorbic acid, anthraquinone, active carbon, and paracetamol.

Results show that particle breakage occurs at any conditions for all the dusts investigated, except for lycopodium. It is also found that dispersion through the rebound nozzle generates a more significant variation in particle size than dispersion through the annular nozzle. Furthermore, higher fractional loss is observed with increasing dispersion pressure.

All these results suggest that the measurement of the explosion and flammability parameters according to the standard procedure may lead to misleading results. Thus, a novel dust dispersion method should be proposed in order to avoid failure in measurement of dust explosion/flammability parameters.

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1. Introduction

Many industrial accidents are imputable to explosions of flammable dusts. Protection from such accidental explosions requires the knowledge of flammability and explosion parameters of dust/air and/or dust–gas/air mixtures. Most of these parameters (minimum explosible concentration, MEC; limiting oxygen concentration, LOC; maximum explosion pressure, P_{MAX} ; deflagration index, K_{St}) are evaluated through standard tests performed in a closed steel combustion chamber with an internal volume of at least 20 L, spherical or cylindrical (with a length to diameter ratio of approximately 1:1) in shape, according to standard procedures described in details in both European and American guidelines (the German Society of Engineers (VDI) Method 3673 (1995), the International Standards Organization (ISO) Method 6184/1, the American Society for Testing and Materials (ASTM) Method E 1226 (2000) and the National Fire Protection Association (NFPA) Standard 68 (1994)), as reported in [1]. One of the major requirements of the apparatus is that it must be capable of dispersing a fairly uniform dust cloud of the material.

According to the standard procedure, if the 20 L sphere is used as explosion vessel, the dust is loaded in a dust container together with compressed air (21 bar), while the explosion vessel is initially pre-evacuated

at 0.4 bar. When the valve connecting the container to the vessel is opened, the dust is injected into the vessel.

At the bottom side of the vessel, a rebound nozzle or alternatively an annular nozzle is placed to allow dispersion of the dust/air mixture inside the vessel. When the valve is opened, the pressure difference between the container loaded with dust (21 bar) and the vessel (0.4 bar) generates turbulence that decays in time. The dust/air mixture passes through the holes present in the rebound nozzle and it is pushed inside the sphere, thus creating a dust/air cloud. Such a procedure has at least three main issues: i) the control of the turbulence level present in the sphere at the moment of ignition (pre-ignition turbulence); ii) the dust dispersion, mixing and homogeneity (uniformity of the dust/air cloud); and iii) the integrity of the solid particles when injected through the rebound nozzle.

Results of CFD simulations [2–4] allowed the visualization of fluid flow, turbulence level and dispersion degree in the 20 L explosion vessel with the rebound nozzle. It has been found that the turbulent kinetic energy is not uniform inside the sphere, being very high only at the center of the sphere. Furthermore, it has been shown that the dust particles are not well dispersed within the sphere.

Kalejaiye et al. [5] used optical dust probes to measure optical transmittance through the dust cloud at six locations within the 20 L sphere equipped with the two standard nozzles – the rebound and the perforated annular nozzles – in order to have a measure of the dust uniformity inside the vessel. They tested dispersion of three dusts with different

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diameters and concentrations, showing that the transmission data were significantly lower than those calculated from theory assuming uniform dispersion and concentration equal to the nominal value in all cases. They have addressed the difference to the reduction in particle size that occurred during dispersion. To prove this, they measured the dust size before and after dispersion, showing that after dispersion the size of the dust particles significantly decreases being the diameter about half the initial value.

In this work, we studied the effect of the standard dispersion systems (rebound nozzle and perforated dispersion ring) on the particle integrity. To this end, dispersions of different dusts were performed in the 20 L vessel at different diameters and dispersion pressures. The two standard dispersion systems were used.

2. Methods

2.1. Dispersion tests

Dispersion tests were performed adopting the usual procedure reported in the ASTM E1226 standard using Siwek's sphere equipped with both the rebound nozzle and the perforated ring (Fig. 1). For each substance reported in Table 1, 10 g was charged into the sample container, pressurized at 10, 20 and 30 bar (10 and 30 bar were also used in order to study the effect of the initial pressure on the particle fragmentation process) and dispersed in the pre-evacuated bomb at a pressure such that after dispersion the final pressure is equal to that of the atmosphere without triggering the ignition. After a suitable time interval (usually 5–10 min) necessary to allow the particle settling, the bomb was opened and the powders were collected paying attention to recover as much as possible of the initial sample fed (generally no less than the 80% of the initial mass was recovered). Recovered samples were characterized measuring both the diameter distributions and by Scanning Electron Microscopy (SEM) analysis. Particle distributions were determined using a laser granulometer Mastersizer 2000 (measuring range: 0.02 ÷ 2000 µm) under stirring (3500 rpm) and both with (to avoid particle agglomeration) and without ultrasounds. SEM images were acquired using a EDX FEI-Inspect S, Column E-SEM W (Filament: tungsten) system with an accelerating voltage of 200 V ÷ 30.0 kV equipped with an Energy Dispersive X-ray (EDX) probe and, depending on the sample analyzed, with 100, 800, 2000 and 10,000× magnification operated in ETD (Everhart–Thornley Detector) modality.

In Table 1, the dusts tested are given together with their mode diameter as measured with stirring or ultrasound assisted.

2.2. Elastic recovery of materials

Force–displacement curves for multiple compression cycles were used to obtain information about the elasticity of the materials [6]. Tablets of the different materials were prepared by compacting the powder in a circular die, 13 mm in diameter, using a Z010 from Zwick/Roell (Ulm, Germany) universal testing machine equipped with a 10 kN load cell.

Each sample was compressed multiple times up to a compression pressure of 7.5 MPa and at a speed of 0.5 mm/min, and then



Fig. 1. Perforated dispersion ring (left) and rebound nozzle (right).

Table 1
Particle mode diameter, D (µm).

Dust	D [µm] stirring 3500 rpm	D [µm] ultrasound assisted
Active carbon	60	60
Anthraquinone	30	21.2
Ascorbic acid	475	–
Lycopodium	34	34
Nicotinic acid	423	34
Paracetamol	134	150

immediately decreased at the same speed, before being ejected from the die. Work done in each compression cycle was calculated by integration of each force/displacement curve. When this work became constant, this force–displacement value was assumed as the work done to produce the elastic deformation during compaction and it was an indicator of the elasticity of the materials. Elastic recovery (ER) is defined as percentage of axial expansion of the compact after ejection, relative to its height at maximum pressure:

$$ER = 100 (h - h_c) / h_c \quad (1)$$

where h_c and h are the heights under compression and after ejection, respectively.

The plastic deformation takes place during initial compression and, after a certain number of compression cycles, elastic deformation becomes predominant.

3. Results

In Figs. 2 and 3, the granulometric distribution as obtained for all the dusts investigated in this work is shown. The blue curve refers to the granulometric distribution of the original sample, while the red and green curves refer to the granulometric distribution after dispersion through the rebound nozzle and the annular nozzle, respectively.

After dispersion, the granulometric distribution significantly changes in all cases, except for lycopodium that is not affected by the dispersion in the vessel. It is also worth noting that the dispersion through the rebound nozzle generates a more significant change in particle size than the dispersion through the perforated dispersion ring. Ascorbic acid results as the dust with the highest fragmentation since the mode diameter reduces from 475 to 168 µm.

It is worth saying that in the case of nicotinic acid the results obtained with the ultrasound assisted methodology are different from those obtained with only stirrer. The curves showed above refer to the results obtained by ultrasound assisted methodology. This difference has to be addressed to the presence of agglomerates in the original sample.

In Fig. 4, the SEM images of the original sample and the samples dispersed through the rebound nozzle and the perforated dispersion ring are shown for all the dusts investigated.

From the SEM images, the high fragmentation of ascorbic acid dust can be observed. Furthermore, it appears that the lycopodium particles remain unchanged, behaving like sponges. The SEM images also confirm the presence of agglomerates in the nicotinic acid samples.

From granulometric distribution, we evaluated the mode diameter. In Table 2, the mode diameter of the original sample and the samples dispersed through the rebound nozzle and the perforated dispersion ring are given for all dusts. In the same table, the mode diameter variation ($\Delta D = (D_{in} - D_f) / D_{in} 100$) is also given.

The dust breakage seems to be slightly lower during the passage through the perforated dispersion ring.

From the literature, it is known that the fractional loss per impact (ξ) increases with increasing linear dimension of the original particle (D) and the dependence on D changes as a function of the particle breakage mechanism [7]. In the case of a particle breakage mechanism based on chipping (lateral cracks), the fractional loss may be calculated

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