

The effect of mixing combustible and inert dust on the minimum ignition energy

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Abstract

An experimental campaign was conducted to measure the minimum ignition energy (MIE) of dust mixtures made of a combustible and inert dust. Three combustible dusts – flour, grain dust and lycopodium, and three inert dusts – CaCO₃, limestone and silica sand were tested. All the combustible dusts were studied in the same manner. Mixtures of each of the inert dusts were prepared having different weight ratios, ranging from 100% to 0% of inert. At each ratio, the MIE was measured using a modified Hartmann tube. The explored energy values ranged from 4 to 4165 mJ. Three energy levels were measured, here called E₁, E₂ and E₃ according to Cavallero and Marmo¹⁾.

The effect of the inert dust diameter and chemical properties were also studied.

Keywords : dust explosion, minimum inerting concentration, MIE.

1. Introduction

Inerting or partial inerting can be used to avoid, suppress or mitigate explosions and their consequences. It is well known that many inerts, whether gaseous or solid, can be used to suppress explosions or mitigate the violence, for example by decreasing the dP/dt_{max} (Eckhoff^{2), 9)}, Dastidar and Amyotte³⁾ Jaeger⁴⁾, Dastidar et Al.^{5), 6)}. As an example, NFPA 651¹⁰⁾ suggest mixing aluminum dusts with inert dust, such as calcium carbonate, to a weight ratio of 1 : 5 to ensure the mixture cannot explode. It is not yet clear what the effect is of admixing combustible and inert dusts on the Minimum Ignition Energy (MIE) of the former, nor what the effect is on the ignition probability at a given spark energy. Explosion mitigation or suppression should be the consequence of several phenomenon which that characterize the interaction between inert particles and the flame front. However the inert dust subtracts heat, which as a consequence, is no longer available for flame

propagation. Heat subtraction occurs according to several mechanisms. First, inert particles adsorb energy until they heat to flame temperature. The heat is adsorbed at a rate that depends on the inert mass, the heat exchange mechanism and the driving force. The heat exchange mechanisms that take place are, of course, radiation and convection. Both depend on the area available to exchange as a consequence the inert particle diameter is expected to exert a great effect on the ability of the inert to suppress the flame front : smaller particles should be more efficient than larger ones. If the inert can undergo endothermic decomposition, the heat subtracted from the flame front can further increase. Chemically reactive solids, such as sodium carbonate, are expected to affect the MIE of the mixture much more than non–reactive inert dusts like silica sand. In order to verify these hypotheses an experimental campaign has been conducted to measure the MIE of dust mixtures. Three different combustible dusts – flour, grain dust and lycopodium were chosen.

Table 1 Composition of the carbonaceous and silica sands used as inerts, expressed as oxides.

Limestone N° 1 (IN1)		Limestone N° 2 (IN2)		Silica sand	
Component	%Weight	Component	%Weight	Component	%Weight
Magnesium Oxide	–	Magnesium Oxide	4.5708	Quartz	84
Aluminum Oxide	0.1695	Aluminum Oxide	0.3556	Feldspar	7.5
Silica	0.5586	Silica	1.0445	Serpentine	6.5
Phosphorous Oxide	0.0354	Phosphorous Oxide	0.033	Others	1.0
Sulphur dioxide	–	Sulphur dioxide	0.0478	Limestone	1.0
Potassium Oxide	0.1128	Potassium Oxide	0.3087		
Calcium Oxide	98.4447	Calcium Oxide	92.2522		
Titanium Oxide	–	Titanium Oxide	0.1783		
Ferric Oxide	0.5839	Ferric Oxide	1.1431		
Nickel Oxide	0.0194	Nickel Oxide	–		
Strontium Oxide	0.0656	Strontium Oxide	0.0660		

Sodium carbonate, limestone and silica sand were used as inerts, in order to understand the effect of inert characteristics such as heat capacity, particle diameter and chemical reactivity. Sodium carbonate is expected to undergo chemical decomposition during an explosion and, as a consequence, it is expected to decrease the MIE to a great extent. Limestone should be far less reactive, but endothermic decomposition could take place to some extent. Silica sand should not decompose, and it is expected to suppress the explosion by subtracting the heat corresponding to the inert temperature increase during explosion. Several mixtures of combustible and inert materials were prepared with different weight ratios (ϕ), defined as the ratio between the inert and the mixture mass, from 100% to 0% inert. The MIE was measured at each ratio in a modified Hartmann tube using a capacitive discharge circuit to generate the spark. The energy values ranged from 4 to 4165 mJ. Three energy level were measured for each mixture: E_1 , E_2 and E_3 according to Cavallero and Marmo¹⁾.

In order to understand the effect of the inert dust diameter, the silica sand and limestone were classified according to size intervals, and mixtures of the combustibles and the different inert size intervals were studied.

2. Samples

Combustible dust. Grain dust, lycopodium and flour were chosen as combustible dusts. The lycopodium was provided by Sigma Aldrich, and the flour was of a commercial 00 type. The grain dust was collected in a grain mill close to Turin. The grain dust was sieved to eliminate the coarser particles and fibres. The size distribution of the flour and sieved grain dust are shown in Fig. 1.

Inert dusts. Four different inerting materials were used: silica sand, two limestones and CaCO_3 . The silica sand and the two limestones were provided by two mines in the North of Italy. The CaCO_3 (analytical grade) was provided by Sigma Aldrich. Their features are summarized in Table 1.

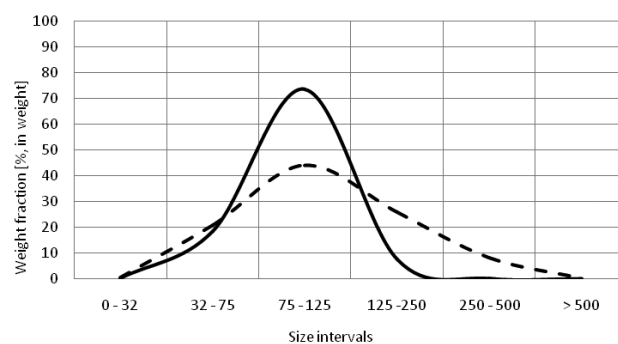


Fig. 1 Particle size distribution of the flour and grain dust samples used in the experimentation.

3. Experimental setup and procedure

The experimental setup is described elsewhere¹⁾. The experimental apparatus is a modified Hartmann tube equipped with a high voltage triggering system. The test chamber structure was constructed according to Cesana & Siwek¹¹⁾. The control system allows the minimum ignition energy to be measured in the 4 to 4165 mJ range, these being the actual values that take into account all the losses in the triggering circuit. The delay time that elapses between the dust suspension and the ignition ranges from 30 to 180 ms in 30 ms steps.

The same measuring procedure used in Cavallero and Marmo¹⁾ was adopted in this work, since it had proved to avoid any effect of spurious factors, above all the moisture content of the sample.

The silica sand and limestones (hereafter called IN 1 and IN 2) were carefully washed with water in a 5 liter tank to eliminate the finer fractions. The mixture was stirred and the dust allowed to settle for a few minutes, then the supernatant was eliminated. The procedure was repeated until a clean supernatant was obtained. The solid was recovered and dried in a stove for 24 hours at 105°C. The silica sand and limestone were classified according to size by sieving with standard sieves. The dimensional classes obtained through sieving are summarized in Table 2. The CaCO_3 was used not classified. Its size distribution is summarized in Table 3.

Each combustible dust was conditioned in a stove for at

Table 2 Mixtures studied in this work. Grey : tested, white : not tested.

Inert	Silica sand				(IN1)		(IN2)		CaCO ₃	
	Size [μm]	32–75	75–125	125–250	250–500	32–75	75–125	125–250	250–500	32–250
Flour	Grey	Grey	Grey	Grey	Grey	Grey	Grey	Grey	Grey	Grey
Lycopodium	Grey	Grey	Grey	White	White	White	White	White	White	White
Grain dust	Grey	White	White	White	White	Grey	Grey	Grey	Grey	Grey

Table 3 Size distribution of the CaCO₃ used as an inert.

Size [μm]	<32	32–75	75–125	125–250	250–500
%Weight	0.95	26.44	32.55	36.07	3.98

least 4 hours at 105°C, then the combustible dusts were stored in a silica gel dryer no more than two days.

The combustible and inert dust mixtures with the desired weight ratio were prepared by weighing the desired amounts of each material and mixing the mixture in a glass becker. The mixture was transferred into a silica gel dryer where it was kept until the measurements. The maximum ageing allowed for the mixture in the silica gel dryer was six hours, then a new mixture was prepared and conditioned. The mixtures studied in this work are shown in Table 2.

Each mixture sample was weighed immediately prior to the MIE measurement, and immediately transferred into the test chamber using a small aluminum tray. The sample and the tray were grounded prior to disposing of the dust in the Hartmann tube.

In each case, according to Cesana & Siwek¹¹⁾, the following energy values were measured :

- E₂: the lowest energy value at which ignition occurred at least once in ten subsequent tests ;
- E₁: the highest energy value at which no ignition was observed in at least ten subsequent tests ;
- E₃, the highest energy value at which the explosion occurred at each run, ten times, during the first dispersion of a new sample.

Each sample was submitted to a maximum of three ignition attempts, and in the case where no ignition occurred, the Hartmann tube was opened, carefully cleaned and a new sample was introduced into the test chamber.

The delay time (*t_d*) was kept constant at 180 ms, which guarantees the lowest ignition energy for each sample. The maximum delay time was used to improve the measurement sensibility to the other variables.

Preliminary measurements were made with the aim of measuring E₁, E₂ and E₃ for each pure combustible dust, as a function of the sample weight, i.e. of the nominal sample concentration in the test chamber. A minimum was generally obtained for each value, whereas E₃ showed a much stronger dependence on the sample amount (see, for example, the data in Fig. 2) than E₁ and E₂. Generally, the minimum of each E_n was found at the same dust concentration, to be equal to 833 g/m³.

The same measurements were repeated using dust mixtures and keeping the weight ratio (ϕ) constant. It was

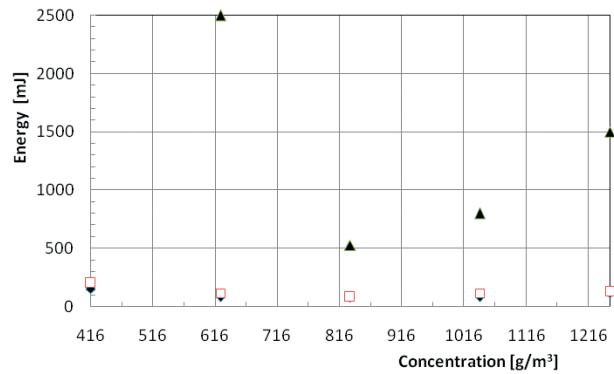


Fig. 2 E₁, E₂ and E₃ versus the nominal dust concentration in the Hartmann tube. Combustible : flour, *t_d* = 180ms. ▲ : E₃, □ : E₂, ◆ : E₁.

generally observed that each mixture exhibited minimum values for each energy level at the same nominal concentration the pure combustible dust did. As a consequence, the concentration corresponding to the minimum energy of the pure combustible dust was chosen as the working point for the mixtures obtained using the same dust. As a consequence, the MIE measurements of a given mixture were made at a constant nominal mixture concentration.

4. Results

As expected, the MIE was influenced to a great extent by ϕ . Each value (E₁, E₂ and E₃) increased when the inert amount increased. Fig. 3 shows an example of the results,

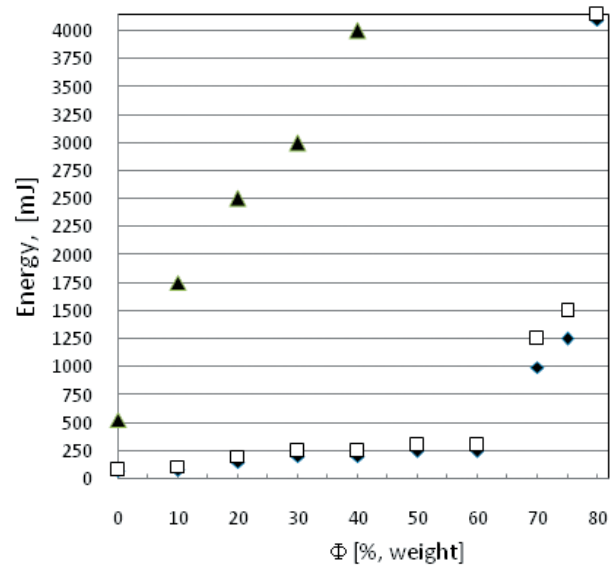


Fig. 3 E₁, E₂ and E₃ for the Flour—IN 2 (125–250 μm) mixture. The measurements were made at 833 g/m³, *t_d* = 180ms. ▲ : E₃, □ : E₂, ◆ : E₁.

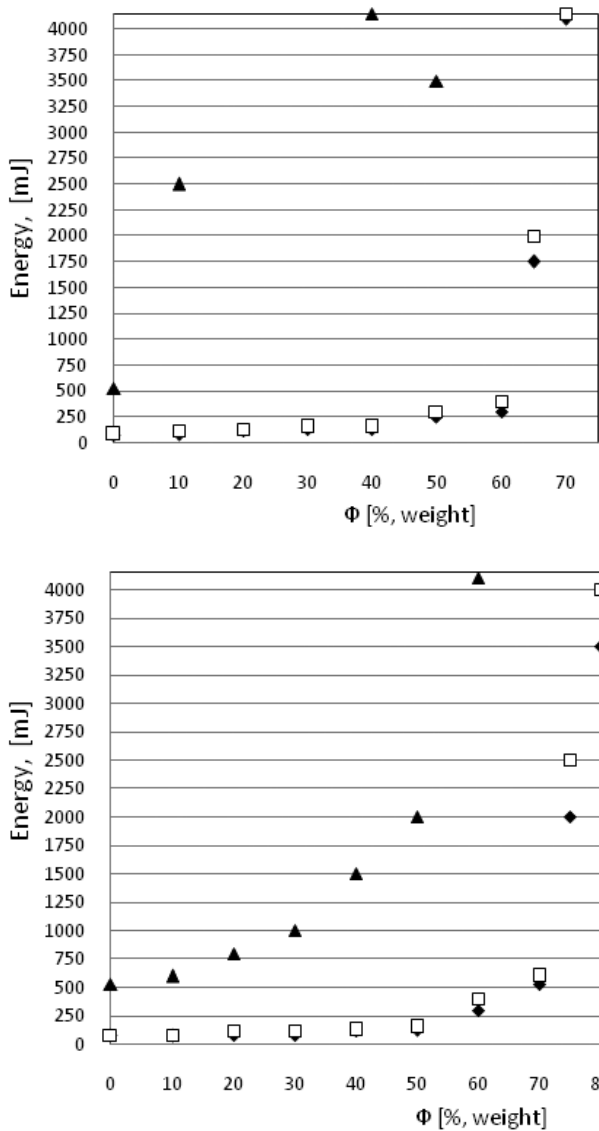


Fig. 4 E_1 , E_2 and E_3 for the Flour—Silica sand mixture of different sizes. Inert diameter 75–125 μm (left), 250–500 μm (right). The measurements were made at 833 g/m^3 , $td = 180\text{ms}$. \blacktriangle : E_3 , \square : E_2 , \blacklozenge : E_1 .

where the typical trend of E_1 and E_2 with respect to ϕ , is evident: at low ϕ , the two energy values increase linearly with ϕ . When the amount of inert is further increased the E_1 and E_2 slope rise and the dependence changes to exponential. This is a general trend that was observed whatever combustible and inert were used. A sort of critical ratio ϕ_c , that divided the linear field from the exponential one, seems to exist. ϕ_c depends to a great extent on the inert characteristics, it is much lower in case of strongly reactive solids, such as CaCO_3 .

It is of particular interest to observe the effect of different inert properties on the same combustible. Fig. 4 shows the effect of a change in the inert particle diameter. As a general rule it is evident that a decrease in the inert particle size results in an increase in E_1 , E_2 and E_3 at the same ϕ . The data shown in Fig. 4 refer to silica sand as the inert. Since silica sand cannot undergo decomposition, the more efficient inerting effect of the finer particles must be due to heat exchange phenomena and not to a chemical

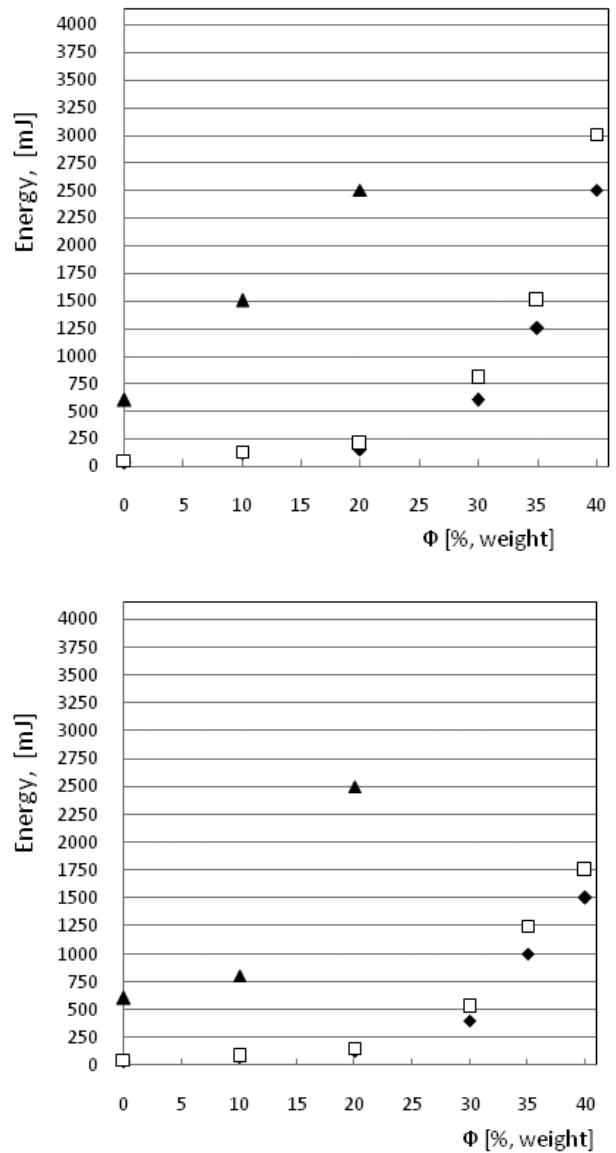


Fig. 5 E_1 , E_2 and E_3 for the dust grain-IN mixture (32 μm), right, and dust grain- CaCO_3 , left. The measurements were made at 833 g/m^3 , $td = 180\text{ms}$. \blacktriangle : E_3 , \square : E_2 , \blacklozenge : E_1 .

reaction. This general trend confirms that fine inert particles subtract the heat of combustion from the reacting mixture more efficiently. As a consequence, the amount of inert which is necessary to increase the ignition energy above a given value increases as the particle diameter grows.

The effect of a change in the inert type is shown in Fig. 5, where E_1 , E_2 and E_3 of the dust grain mixed with IN 1 and CaCO_3 , respectively, can be observed. Note that CaCO_3 was not size-classified, hence the size of the inert used in the mixture in Fig. 5 has the size distribution as that in Table 2. It is easy to note that the CaCO_3 has a much larger mean diameter than the inert in Fig. 5 (right). Despite the higher inert diameter, the energy necessary to ignite the dust grain- CaCO_3 mixture is much higher than the energy necessary to ignite the dust grain-IN mixture. It is evident that CaCO_3 is much more efficient in suppressing the explosions. This effect should be due to the chemical decomposition of calcium carbonate which

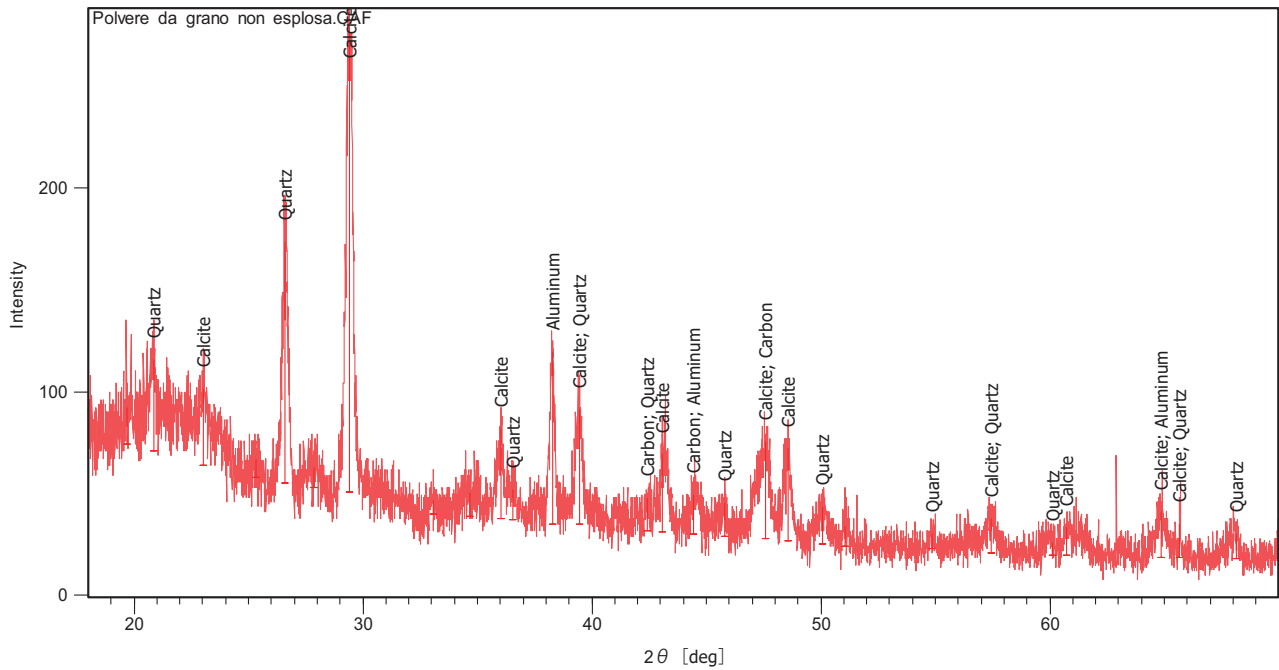


Fig. 6 X-ray diffraction for the dust grain-CaCO₃ mixture, not exploded.

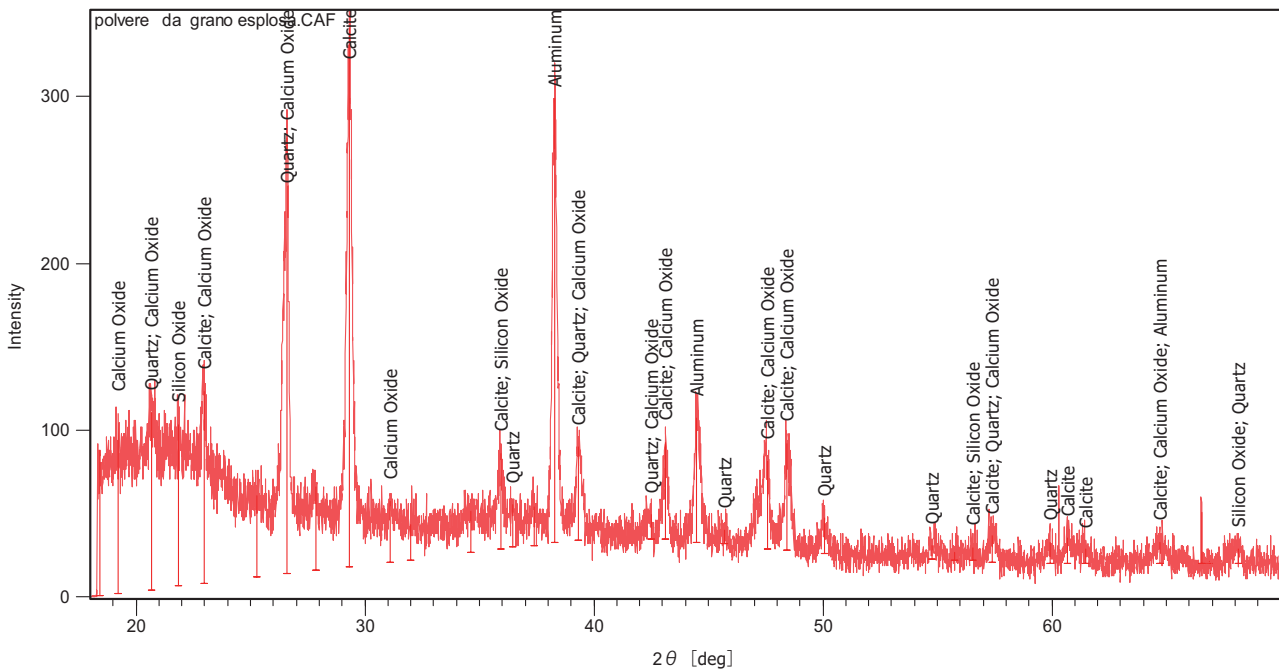


Fig. 7 X-ray diffraction for the dust grain-CaCO₃ mixture, exploded.

forms CaO. To prove this phenomenon, several mixture samples were analyzed by X-ray diffraction to highlight the decomposition of the sodium carbonate and of IN 1 and IN 2. Blank measurements were made on the pure inert dust and on the unexploded mixture. Measurements on the exploded residual were then made on the dust collected from the Hartmann tube after the explosion.

The results of the X-Ray diffraction are shown in Fig. 6 (unexploded, used as a blank) and Fig. 7 (exploded). Although CaO was absent in the blank sample, traces were found in the sample collected after the explosion.

The inerts from natural limestone (IN 1 and IN 2) were far less reactive than CaCO₃, and their ability to increase the MIE was similar to that of silica sand.

5. Inerting mechanism

The trend of E_1 and E_2 versus ϕ seems to agree with a two mode interaction between inert dust and the flame front. The first mode deals with the heat subtraction consequence of heat capturing by a convection/chemical reaction, the second mode deals with the optical shielding of inert particles versus the radiant heat propagation. The first mode is more efficient at low ϕ , the second is more efficient at high ϕ .

To confirm this hypotheses, a model of the MIE was postulated as follows.

First of all a method to calculate the MIE from the available data was developed. It is known (Bernard et. Al.⁷, Baudry et. Al.⁸) that ignition probability varies

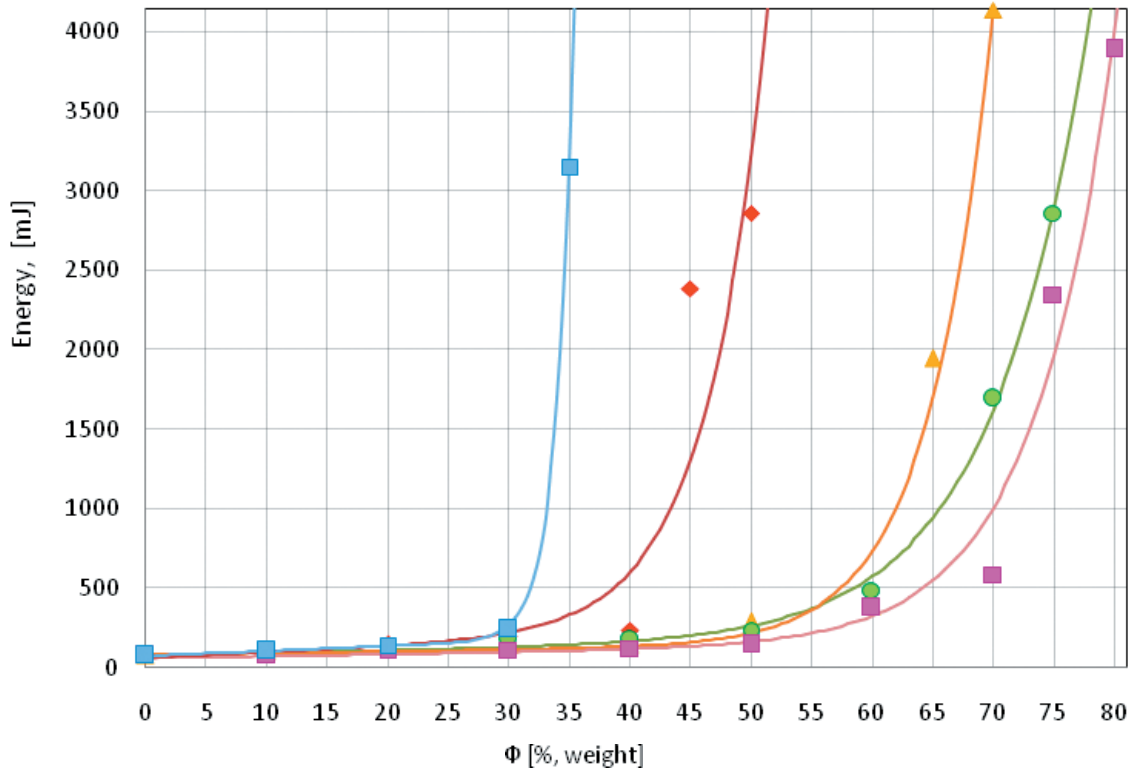


Fig. 8 MIE of the flour–CaCO₃ (light blue), flour–IN1 and flour–IN 2 mixtures. Data refer to different inert sizes: 32–75 (red); 75–125 (yellow); 125–250 (green); 250–500 (pink). Solid lines: model prediction (equation 3).

between E_1 and E_2 , being null at E_1 . It is not clear what ignition probability should correspond to the MIE, some Authors (Bernard et. Al.⁷, Baudry et. Al.⁸) assumed a value ranging between 5 to 10% as a proposal, despite in principle the MIE should be the energy value that cause the ignition probability to rise from zero to a finite positive value. In this paper we assumed as an estimate of the MIE a value depending on the ignition probability measured at E_2 . To this purpose MIE was calculated on the basis of E_1 , E_2 and the ignition probability at E_2 according to Equation 1.

$$MIE = E_2 + (E_1 - E_2) \cdot \left(\frac{w_1 (\ln(1 + P_1) / \ln 2)^{0.5} + w_2 (\ln(1 + P_2) / \ln 2)^{0.5} + w_3 (\ln(1 + P_3) / \ln 2)^{0.5}}{w_1 + w_2 + w_3} \right) \quad (1)$$

where P_n are the ignition probabilities at energy E_2 at the n^{th} resuspension of a given sample (maximum resuspension attempts equal to three, according to the norm), and w_1 , w_2 and w_3 are arbitrary weights, 0.4, 0.3 and 0.3, respectively.

The ignition probability at E_2 was defined as:

$$P_i = \frac{n_{ig.}}{n_{att.}} \quad (2)$$

where P_i is the ignition probability, $n_{ig.}$ is the observed number of ignitions and $n_{att.}$ is the number of ignition attempts needed to obtain $n_{ig.}$

The data calculated with Equation 1 are shown in Fig. 8. The data were fitted with Equation 3, where a , b , c and d are parameters and ϕ is the inert weight ratio as defined above.

$$y = a + b \cdot \phi + c \cdot e^{(d \cdot \phi)} \quad (3)$$

The meaning of the terms in equation 3 agrees with the two-mode interaction cited above. The linear term $b \cdot \phi$ stands for heat subtraction which is somewhat proportional to the amount of inert in the mixture, and the exponential term $c e^{(d \cdot \phi)}$ stands for the optical shielding effect.

The agreement between the data and model prediction was very good, whatever the combustible or inert dust used, and whatever the size of the inert. The model parameters obviously changed as the dust and size classes changed.

6. Conclusions

The effect of admixing combustible and inert dusts on the MIE has been measured at various weight ratios, inert and combustible characteristics, and inert dimensions.

The inerting attitude depends on several factors, i.e.

- Diameter of the inert particle
- chemical properties
- physical properties

The results demonstrate that the lower the diameter of the inert, the stronger the inerting effect with respect to all the combustible dusts studied.

The dusts capable of endothermic decomposition, such as CaCO₃, were much more efficient in increasing the MIE than inert dusts like silica sand.

The MIE of a mixture depends on the dust weight ratio, according to two mechanisms: at a low weight ratio, thermal effects are more important, while at a high weight

ratio, optical effects become more important. The critical weight ratio, ϕ_c , depends on the chemical and physical characteristics of the combustible and inert dust, and it is much lower in the case of strongly reactive solids such as CaCO_3 .

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