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Explosibility and Flammability Characteristics of Nicotinic Acid-Lycopodium/Air Mixtures

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Powder products are frequently meet as dust mixtures in industrial processes. However the effect of mixing combustible dusts on the flammability and explosion behaviour of their mixture is still unclear. This work dealing with the explosibility and flammability of dust mixtures made of Nicotinic acid and Lycopodium. Explosion tests on the 20-L explosion vessel at different dust concentrations and relative amounts were performed. The two dusts showed the same minimum explosion concentration (MEC), but different explosibility parameters (K_{St} and P_{max}). The predominant influence of the most reactive compound (Nicotinic acid) on the explosibility of the dust mixture was found. This effect was not encountered for the minimum explosion concentration.

1. Introduction

Mixing of ingredients, or dispersion of one phase into another, is an operation widely used in industry. Powder products are frequently encountered as dust mixtures in food, pharmaceutical, wood, polymer, pigment and dyes industrial processes. Dust particles of such mixtures suspended in the air and exposed to an ignition source may cause a dust cloud explosion.

In the literature, there is a good knowledge of explosion and ignition behaviour of dust mixture when a combustible dust is added with a solid inertant (Janès and Carson, 2013).. Solid inerting is a known way of reducing the risk of dust explosions. This method is either used to generate a resulting mixture that is rendered non-explosible or the admixture is done with the purpose of mitigating the consequences of a dust explosion.

On the contrary, in the case of mixtures made of two or more combustible dusts, the flammability and the explosion features of the mixture have been scarcely studied.

Only few works on dusts mixtures were performed many years ago (Nagy et al., 1968). However, from these data, it is not possible to derive the reactivity and the flammability of the mixtures with respect to the single dust.

More recently, Denkevits and Dorofeev (2006) studied the explosion behaviour of fine graphite and tungsten dusts and their mixtures. They found that maximum rate of pressure rise had a pronounced peak for the mixture with a molar ratio 1:1, this mixture burned faster than either pure graphite or pure tungsten dust. Conversely, they found that the maximum pressure of the mixtures is a good average of the maximum pressure of the pure compounds.

Finally, Dufaud et al. (2012) investigated the flammability and the explosion behaviour of various solid/solid mixtures, observing different behaviours: they can react together more or less directly to modify their explosion features, the properties of the mixture uniformly ranged between those of the pure compounds or the explosion behaviour of the mixture decreased with respect to the components.

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In this work, the effect of mixing combustible dusts on the flammability and explosion behaviour of dust mixture is studied. We tested mixtures made of dusts with different explosion properties. In particular we tested mixtures of Nicotinic acid and Lycopodium. We performed explosion tests on the 20-L explosion vessel at different dust concentrations and relative amounts.

2. Experimental

Experiments were performed using the standard 20 L apparatus manufactured by Adolf Kühner AG (CH).
The equipment is made of a stainless steel spherical bomb surrounded by a water jacket for the control of the internal wall temperature. A flange on the top of combustion chamber allows for the insertion of two electric rods, in order to reach the center of the sphere for spark electrodes. At the bottom and right side of the bomb, an outlet valve and two piezoelectric transducer are installed. The outlet valve is connected to a rebound nozzle placed at the bottom of the bomb for the dispersion of the dust/air mixture. The input section of the outlet valve is connected to the sample container (0.6 L). All the timing sequences and the acquisition of the pressure signals were performed by means the electronic module KSEP 332, which is interfaced by a desktop computer for the remote control of the system. The system is also connected to vacuum pump and to a re-circulating crio-thermostat for the temperature control (Julabo CF31). In all runs it was used water as cooling fluid, at 25 °C.
The measurements of explosion parameters: minimum explosion concentration (MEC), maximum pressure (P_{max}) and deflagration index (K_{St}), were done following the procedures described in the standard test methods (ASTM E1515, 2007; ASTM E1226, 2010) except for the ignition source. With respect to the standard test method where the use of an ignition source of 10 kJ (two 5-kJ igniters) is recommended, in this work a permanent spark generator capable to supply 15 kV, 30 mA as produced also by Kühner (KSEP 320) was used. The spark electrodes were two rounded tungsten rod (diameter 2 mm), the tips of which were spaced at the standard distance of 6 mm. The ignition delay time i.e. the time between the onset of dust dispersion and the ignition of the dust/air mixture was set at 60 ms, as standard test method.
The choice of this ignition source, the same we used in the case of hybrid (gas-dust) mixtures (Di Benedetto et al., 2012; Sanchirico et al., 2012), was done in order to avoid that the test may overdriven. In fact there are indications that the use of energetic ignition sources in the 20-L vessel may overdrive the system and alter measured explosion characteristics (Cloney et al., 2012; Juai et al., 2013).
As testing materials we chose dusts used worldwide as standard for explosion tests: Lycopodium and Nicotinic acid.
Explosion tests were performed for Lycopodium/air, Nicotinic acid/air and for the dust mixtures Nicotinic acid-Lycopodium/air. The dust concentration was varied in the range 50 – 1,000 g /m^3. In the case of the Nicotinic acid-Lycopodium/air mixtures, explosion tests were carried by varying the total dust concentration in the mixture concentration and maintaining constant the weight fraction of Lycopodium in the dust mixture (0.25, 0.50 and 0.75). Each run was performed three times.
Dusts, used as received, were supplied by Sigma Aldrich. Nicotinic acid or Niacin ($C_6H_5NO_2$) was previously characterized by laser diffraction granulometry using di-ethyl ether as disperdant solvent (Malvern Instruments Mastersizer 2000); scanning electron microscopy (Philips mod. XL30); simultaneous TG/DSC analysis (TA Instruments SDTQ600). Results (i.e., mean particle size = 32 ❍m; humidity = 0 %) were presented in a previous paper (Garcia-Agreda et al., 2011). The particle density of nicotinic acid is 1.473 g/cm^3.
The chemical formula of Lycopodium is $C_1H_{1.68}O_{0.22}N_{0.017}S_{0.00044}$ as reported by Skjold (2003). Lycopodium particles are substantially monosized, with an arithmetic mean diameter of about 30 ❍m (Eckhoff 2003). The particle density is about 1.18 g/cm^3.
Results of laser diffraction granulometry tests of dust samples were reported in Table 1.

Table 1: Granulometric distributions of dusts.

Powder	D(0,1), µm	D(0,5) µm	D(0,9), µm
Nicotinic acid	6	32	93
Lycopodium	25	31	36

3. Results

The results of explosion tests in terms of maximum pressure (P_{max}) as function of dust concentration for the Lycopodium/air mixtures, Nicotinic acid/air mixtures and Nicotinic acid-Lycopodium/air mixtures are shown in Figure 1. The values of Nicotinic acid-Lycopodium/air mixtures refers to total dust (Nicotinic acid + Lycopodium) concentration.

The value of maximum pressure (7.7 barg) of the Nicotinic acid/air mixture was slightly higher than that (6.9 barg) obtained for the Lycopodium/air mixture. The P_{max} for the Nicotinic acid-Lycopodium/air mixture is to that of the Nicotinic acid/air mixture and higher than that found for the Lycopodium/air mixture.

It appears that the mixtures has an enhanced value of the maximum pressure with repsect to the single compound. For both single dust/air mixtures the minimum explosive concentration (MEC) was equal to 125 g/m^3. The same value was measured also in the case of Nicotinic acid-Lycopodium/air mixture.

In Figure 2, the deflagration index (K_{St}) is plotted versus the dust concentration for the Lycopodium/air, the Nicotinic acid/air and the Nicotinic acid-Lycopodium/air mixtures.

For the Nicotinic acid/air mixture the maximum value of the deflagration index was 140 bar m/s at concentration of 600 g/m^3, while for the Lycopodium/air mixture was 55 bar m/s at concentration of 750 g/m^3. The higher deflagration index of the Nicotinic acid/air mixture suggests that the mixture is more reactive than the lycopodium/air mixture.

For Nicotinic acid-Lycopodium/air mixture K_{St} was about 120 bar m/s at concentration of 600 g/m^3. We observed, quite surprisingly, that the behaviour of the two-dust mixture was closer to the behaviour of the more reactive dust (Nicotinic acid).

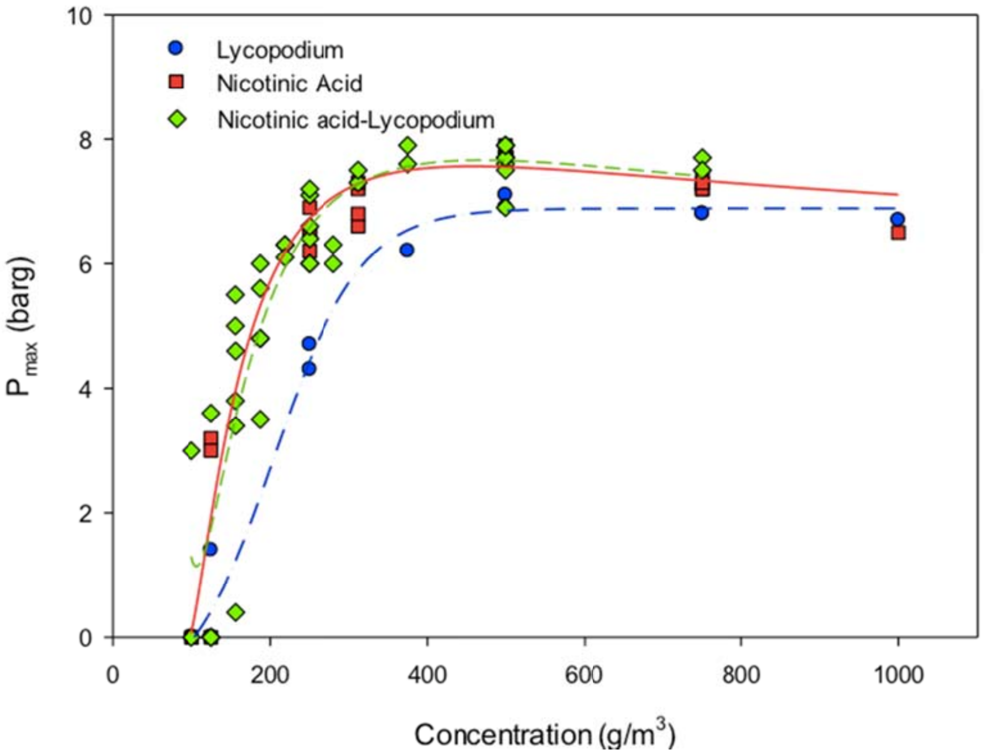

Figure 1: Maximum pressure vs. dust concentration for Lycopodium/air, Nicotinic acid/air and Nicotinic acid-Lycopodium/air mixtures.

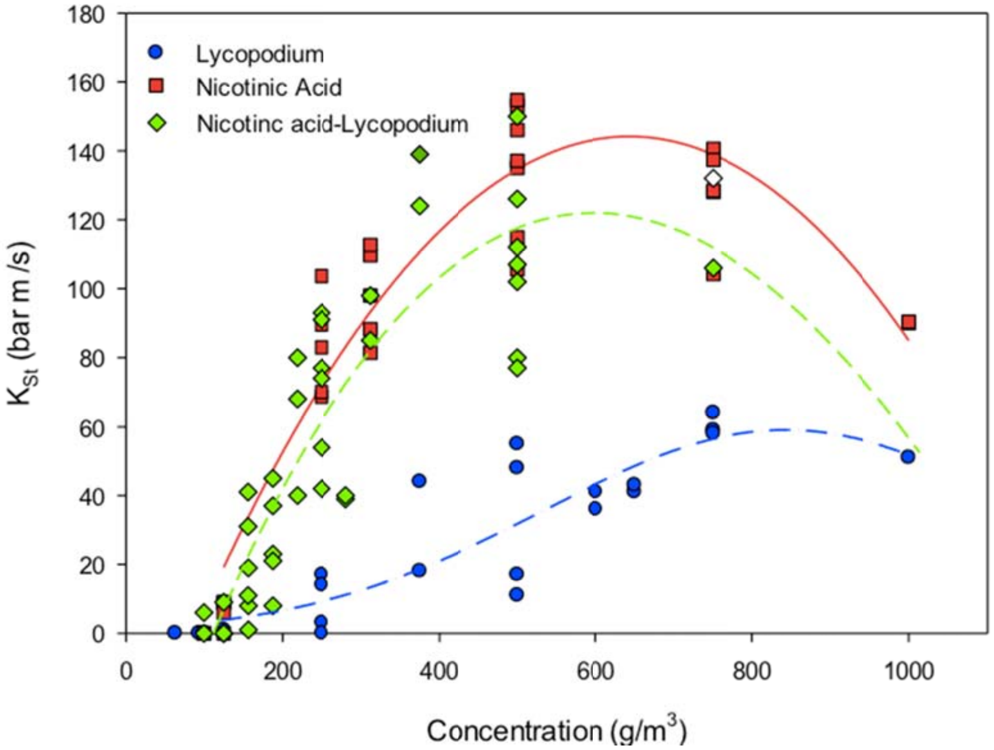

Figure 2: Deflagration index vs. dust concentration for Lycopodium/air, Nicotinic acid/air and Nicotinic acid-Lycopodium/air mixtures.

In order to highlight this behaviour we run a series of explosion tests on the Nicotinic acid-Lycopodium/air mixtures where the total concentration was kept constant and the weight fraction of Lycopodium in the mixture was varied. In Figure 3 the deflagration index of these mixtures is plotted as function of the weight fraction of Lycopodium in the mixture, for two values of total dust concentration: 250 and 500 g/m^3. In the same figure the deflagration index resulting from the weighted average of the deflagration index of the single dust components the mixture was also shown (straight line).
It is found that, at a given value of the total dust concentration, the deflagration index of the mixture is always higher than the average value, whatever the weight fraction of Lycopodium and hence its substitution in the dust mixture. This is the result of synergistic effects which manifest themselves when mixing the two dusts.

4. Conclusions

The effect of mixing combustible dusts (Lycopodium and Nicotinic acid) on the flammability and explosion behaviour of their mixture was investigated. The explosion behaviour of the two-dust mixture was closer to the behaviour of the more reactive dust (Nicotinic acid). Whatever the weight fraction between the two dusts in the mixture the deflagration index was always higher than the average value of the single dust components. On the contrary, synergetic effect was not observed in relation to the MEC.

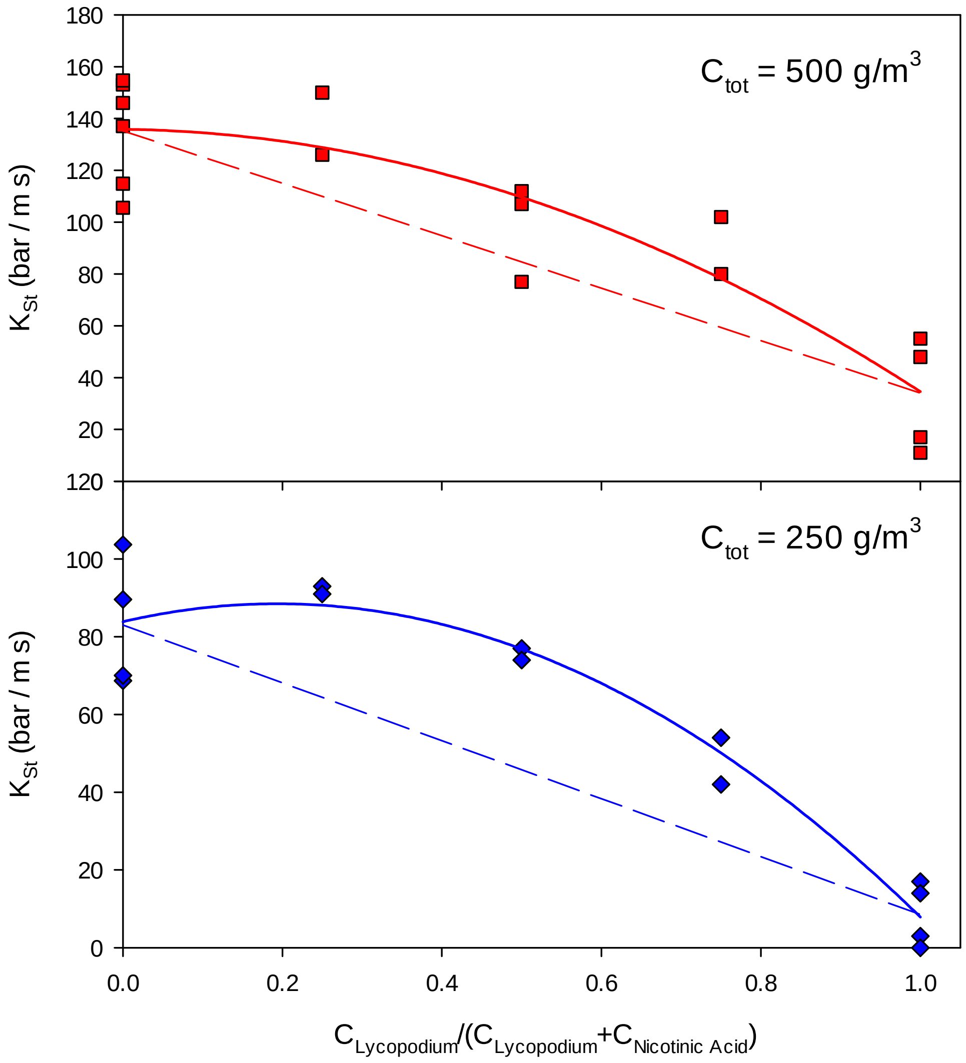

Figure 3: Deflagration index vs. weight fraction of Lycopodium in Nicotinic acid-Lycopodium/air mixture.

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