

Volatile point of dust mixtures and hybrid mixtures

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ABSTRACT

A novel flammability parameter (Volatile Point, VP) of pure dusts, dust mixtures and hybrid mixtures is here measured and proposed as a key parameter to assess flammability.

This parameter is defined as the temperature at which volatiles are produced generating a flammable vapor mixture, similarly to the Flash Point defined for flammable liquid substances and mixtures.

Measurements of VP were performed for niacin, lycopodium, anthraquinone, and their mixtures. The VP values of suspensions of these dusts in glycerol were measured as well. Results show that the VP of the mixtures may be lower than the VP of the pure compounds, suggesting that a synergistic behavior takes place which should be carefully considered when storing and handling dust mixtures and hybrid mixtures.

1. Introduction

In many industrial chemical processes, flammable dusts as well as flammable dust and hybrid (dust-gas) mixtures are used. Prevention and mitigation of accidents coming from their explosions require the knowledge of flammability and explosion parameters. In order to classify the flammability of dusts (or dust and hybrid mixtures), the evaluation of the minimum explosive concentration (MEC), the limiting oxygen concentration (LOC), and the minimum ignition temperature (MIT) is required.

The minimum ignition temperature (MIT) is defined as the temperature at which a dust cloud ignites. MIT is used to evaluate the maximum temperature of a hot surface in the proximity of dust, dust mixture and hybrid mixture. In addition to MIT, the smolder temperature (or layer ignition temperature, LIT) is also measured. The smolder temperature is the temperature at which a flat 5 mm thick dust layer starts reacting. The smolder temperature is very important since the dust in the deposit that has not yet burnt, may be entrained by the gas phase leading to ignition of the dust cloud, as it happened in the Biella accident (Salatino et al., 2012).

In a previous paper, we have shown that the path for dust explosion/flammability may be both heterogeneous through direct combustion of the dust, and/or homogeneous through combustion of the volatiles produced by pyrolysis (Di Benedetto and Russo, 2007). In the presence of the homogeneous path, the flammability and explosion behavior of the dust is highly influenced by the production of volatiles

(Di Benedetto and Russo, 2007; Eckhoff, 2003; Continillo, 1989; Di Benedetto et al., 2010a, 2010b). The generation of flammable volatiles may cause the formation of a flammable atmosphere in air thus activating flame propagation. This phenomenon is similar to the formation of a flammable cloud by liquid evaporation which is quantified by the Flash Point (FP). The only difference is that, in the case of liquids, vapors are produced by evaporation whereas, in the case of dusts, they are formed by pyrolysis.

FP is a widely recognized key parameter for evaluating the liquid mixture flammability. It is defined as the minimum temperature at which vapors given off by a liquid pool form an ignitable mixture with air. This temperature is significantly lower than the MIT (hundreds of degrees Celsius). It helps to characterize flammability of liquids thus providing the conditions for safe packaging, shipping, storage and handling. In some cases, a similar parameter is calculated and reported in the material safety data sheet (MSDS) of the pure dust. However, it is not recognized as a key parameter for characterizing the flammability of dust and/or hybrid mixtures.

In this work, we aim at quantifying the values of the temperature at which pure dusts, dust mixtures and hybrid mixtures are able to produce volatiles which form a flammable vapor mixture. The proposed parameter is here named Volatile Point (VP).

2. Materials and methods

Volatile Point was measured by using the same apparatus used for

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Fig. 1. Setaflash Series 3 Plus closed cup flash point tester.

Flash Point (FP), the Setaflash Serie 3 Plus (Fig. 1). The device is a closed cup instrument that allows the measurement of FP/VP for liquid/solid samples following different international standards (see, e.g. (ASTM E1491-97, 1997; ASTM E2021-15, 2015),) over the range ambient to 300 °C.

The solid sample was put into a container which is heated at a speed equal to 20 °C/min. Samples (analytical grade reagents) were made by mixtures of niacin (i.e., nicotinic acid) (CAS number: 59-67-6), anthraquinone (CAS number: 84-65-1) and lycopodium (CAS number: 8023-70-9) provided by Sigma Aldrich.

Solid mixtures were prepared by intimately mixing the suitable amount of substances and charging the resulting systems into the oven cup by means of a particular sample holder accessory (part n. 13747-0).

Hybrid mixtures were prepared by mixing solid samples with glycerol (Sigma Aldrich, CAS number: 85-81-5). The resulting mixtures were fed into the instrument cup by means of a syringe. A suitable amount of solid powders (niacine/antraquinone) was added to 10 ml of glycerol so as to make the suspensions fluid enough to be injected into the instrument cup. The sample amount was found to be suitable to carry out the necessary runs for the VP determination.

Depending on the size of the sample being tested, the standard procedure involves the tentative of triggering combustion of the gaseous layer by means of the use of a calibrated pilot flame activated after a predetermined interval of time. The instrument, in the case of positive outcome, records automatically the “flash” event at the temperature level considered. The flash delay time at a given temperature was measured as the lowest time at which the flash is recorded for the first

Table 1
Granulometric distribution of niacin.

Percentile diameter (µm)	
D (0,1)	5.56
D (0,5)	32.00
D (0,9)	93.06
Surface weighted mean diameter (µm)	
D (3, 2)	14.37
Volume weighted mean diameter (µm)	
D (4, 3)	41.43

time after the heating of the sample from ambient to the desired isothermal temperature.

The temperature increase we used is equal to 0.5 °C (which is the minimum temperature increase allowed by the equipment used). The standard deviation for each measurement is then equal to 0.5 °C.

2.1. Materials characterization

We characterized the behavior of niacin (i.e., nicotinic acid), anthraquinone and lycopodium. Niacin ($C_6H_5NO_2$) is an organic compound classified as a B-vitamin. It is used mainly in pharmaceutical industries and is also a reference dust for testing dust explosion. Anthraquinone (9,10-dioxoanthracene, $C_{14}H_8O_2$) is an aromatic organic compound. It is used in the production of dyes and natural pigments, as catalyst in the production of wood pulp in pulp and paper industry, and as laxative in pharmaceutical industry. Lycopodium is composed of the dry spores of clubmoss plants. It is used in standard explosion and flammability tests, and also in fireworks and explosives. All samples were characterized as received by Sigma Aldrich.

For characterization, scanning electron microscopy (SEM) (Philips mod. XL30) was used. We also used laser diffraction granulometry (Malvern Instruments Mastersizer, 2000) to characterize the granulometric distribution of niacin (di-ethyl ether was the disperdant solvent) and anthraquinone (water was the disperdant solvent).

SEM images of niacin (Fig. 2) show that the sample is composed of smooth-faced prismatic particles.

Details of the granulometric distribution of niacin are given in Table 1.

SEM images of anthraquinone (Fig. 3) show that the sample is composed of smooth-faced prismatic particles.

Data of the granulometric distribution of anthraquinone are given in Table 2.

SEM images of lycopodium are shown in Fig. 4. The diameter of the particles is equal to 34 µm.

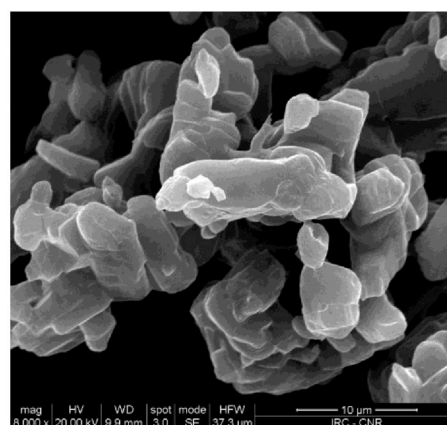
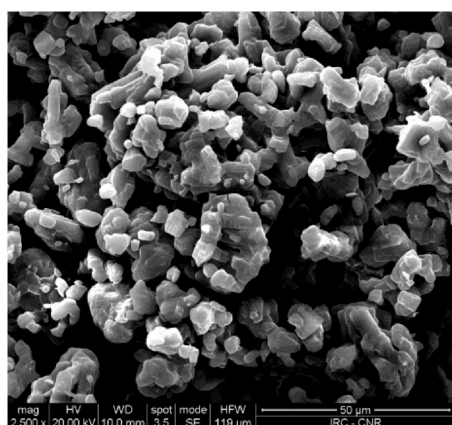


Fig. 2. SEM images of niacin at 2500 × (left) and 8000 × (right) magnification.

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