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New Experimental Findings of Temperature and Turbulence Influence on the Safety Characteristics of Dusts

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The year 1967 can be regarded as the founding year of the "Flammable Dust" working committee of the chemical industry with representatives of BASF, BAYER, Hoechst and Ciba-Geigy AG. Even today, many of the regulations and directives in force [...] are still based on the findings and research results of the above-mentioned working committee in the 1970s - 1980s. These include also many postulates and interpretations for the evaluation or calculation of the ignition and explosion behaviour of substances depended on the ambient conditions, which are actively applied nowadays by experts in industrial practice. However W. Bartknecht and G. Pellmont already pointed out in that the extrapolations of these findings are often of limited value. The paper contains research results and thus serves as an example of those unknown explosion parameters that could not be taken into account. In the first part, the authors report on G. Pellmont's investigations of the temperature dependence of the minimum ignition energy of pyridine-3-carboxylic acid (niacin), which increases with increasing temperature, and not - as the extrapolations of T. Glarner, as well as the interpretations in regulations state - decreases. The second part summarise the experimental investigations of Leksin about the influence of turbulence on the burning behaviour of the dust in the quasi-stationary state in comparison to the sedimented dust, where also for certain dusts the above-mentioned parameter prevents the burning and does not, as expected, influence it more strongly. Both examples from the laboratory investigations shows, that there are a lot of other influencing variables that complicate the approximate predeterminations of explosion processes, not only temperature, admission pressure, humidity (for dusts), turbulence, geometry of the explosion chamber, location of the ignition site, but also the size of the explosion and combustion chamber, effectiveness of "operational" ignition sources, homogeneity, chemical and physical substance properties and many others.

1. Introduction

The drying of pyridine-3-carboxylic acid (further: niacin) was carried out in a plate dryer under specified operating conditions, which are presented in Table 1:

Table 1. Plate drying: Operating conditions for pyridine-3-carboxylic acid (niacin)

Drying air	Supply air temperature [°C]	100 – 110		
	Plate temperature [°C]	140 – 150		
	Exhaust air temperature [°C]	~ 90		
pyridine-3-carboxylic acid	Inlet temperature [°C]	~ 50	Moisture [wt.%]	~ 12
	Outlet temperature [°C]	~ 120	Moisture [wt.%]	< 0.1

Niacin is combustible and therefore in dust form (median value M < 63 µm) explosive. According to the information provided by the manufacturer, the following safety characteristics describing the explosion and decomposition behaviour were known (Table 2):

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Table 2. Safety characteristics of pyridine-3-carboxylic acid (niacin) with median value M = 24.5 µm

Safety characteristics		Valuation
Burning class number		BZ 5
Dust explosion class $^{\text{modified Hartmann apparatus}}$		ST 2
Minimum ignition energy$^{\text{according manufacturer}}$	with inductance	5 mJ < MIE < 10 mJ
	without inductance	5 mJ < MIE < 10 mJ
Minimum ignition temperature	dust layer	T_Z > 365 °C
	dust cloud	T_Z > 440 °C
Exothermic decomposition		None until 230 °C

Based on these temperature values for the niacin-dust/air-mixture (and niacin-dust layer) in the plate dryer, there is neither an ignition hazard due to hot surfaces nor a hazard due to spontaneous combustion. However, once the deposited product is ignited, burning takes place with the appearance of flames. Also, the minimum ignition energy of niacin is in the range below 10 mJ. Based on experience, such products are considered to be very sensitive to ignition from electrostatic ignition sources, e.g. brush discharges. This must be taken into account especially if, according to the current state of knowledge (TRGS 721 2020, VDI 2263 Part 1 2019), the minimum ignition energy of dusts falls with increasing temperature (Bartknecht 1993, Glarner 1984, Bartknecht & Zwahlen 1992). This relationship is shown in Figure 1:

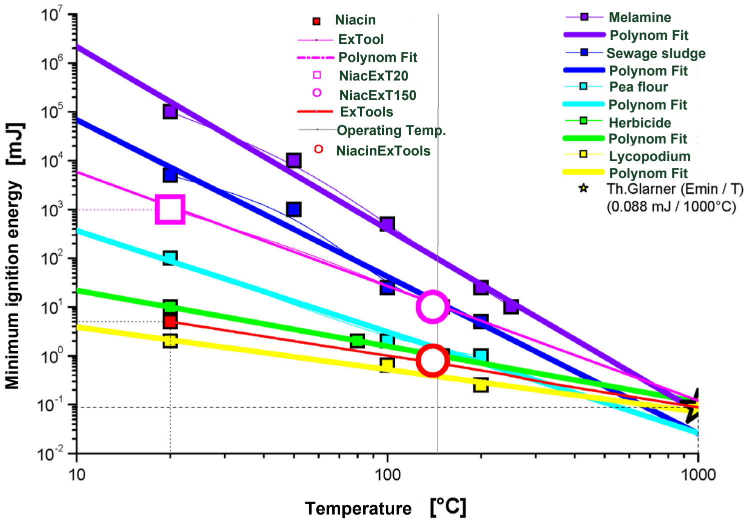

Figure 1. Expected influence of temperature on the minimum ignition energy of niacin.

The expected minimum ignition energy of niacin approaches the energy range valid for the fuel gases (e.g., propane) at the specified plate temperature of the dryer of T = 140 - 150 °C. Even though the ignition capability of brush discharges with a similarly low equivalent energy has not yet been experimentally proven, ignition hazards towards very easily flammable products cannot be excluded. For this purpose, according to the information in (figure X oben), it is necessary to add such quantities of nitrogen to the combustion air that the niacin/dust/nitrogen/oxygen mixture will have a minimum ignition energy of MIE = 10^3 mJ at room temperature. Due to the influence of the temperature, a minimum ignition energy in the order of 10 mJ can be expected - in this case at T = 140 °C i.e., an ignition hazard due to electrostatic brush discharges can be excluded.

2. Influence of temperature on the ignition- and explosion-behaviour of niacin

A 20-l laboratory apparatus (20-l sphere) (Bartknecht 1993, VDI 2263 Part 1 1990) for investigations at elevated temperature was converted and the ignition spark device of the modified Hartmann apparatus (protracted capacitor discharges) (Berthold 1987) was adopted. During the first ignition tests, which were carried out to test the experimental equipment at a product and apparatus temperature of T = 140 °C and a maximum possible capacitor discharge energy of E = 12.5 J, there was no ignition of the niacin-dust/air mixture (by low turbulence) over a wide dust concentration range, despite clearly visible sparks. Only when pyrotechnic igniters with energy of 500 J were used as the ignition source, the niacin-dust/air mixture has ignited under specified temperature conditions. This observation led to the conclusion that, at least in the case of pyridine-3-carboxylic acid, the minimum ignition energy does not fall with increasing temperature, as actually expected, but rises, because the minimum ignition energy of this product can be expected in the range of 5 - 10 mJ at room temperature according to the data in table 2. Therefore, the intended tests to determine the limiting oxygen concentration related to an ignition energy of E = 1000 mJ (fig. 1) were dispensed and the influence of temperature on the minimum ignition energy of niacin was systematically investigated. The test results can be seen in figure 2:

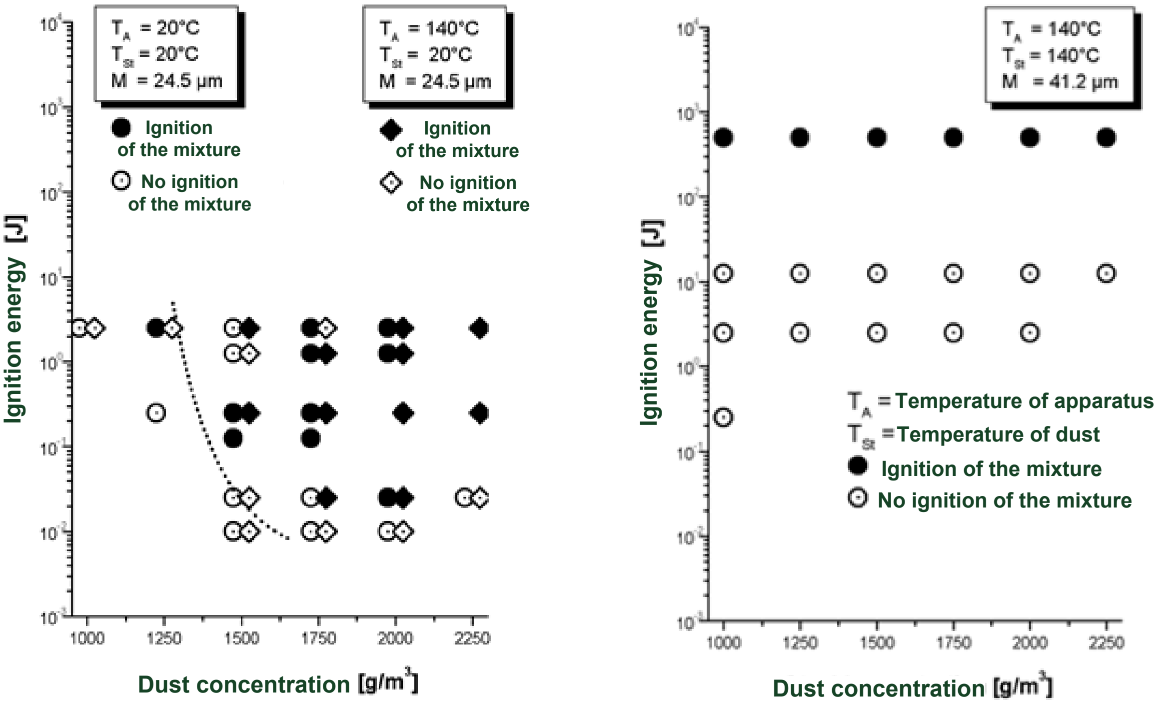

Figure 2. Influence of the product temperature T_{St} and the apparatus temperature T_A on the explosion range - 20-l sphere, protracted capacitor discharges

Thus, niacin-dust/air mixtures have anomalous ignition behavior when the temperature is raised. This anomaly is also present when the correlation between the minimum ignition energy and the oxygen concentration on the one hand and the median value on the other is considered. Table 3 summarises the test result with additional specification of the oxygen concentration in nitrogen.

Table 3. Influence of temperature on the median value M and the minimum ignition energy of pyridine-3-carboxylic acid - 20-l sphere, tv=120ms, protracted capacitor discharges

Temperature of dust T_{St} [°C]	Temperature of apparatus T_A [°C]	Median value M [µm]	Oxygen conc. $c(O_2)$ [$mol \cdot l^{-1}$]	Minimum ignition energy [mJ]
20	20 140	24.5	0.00877 0.0060	10
80	80	27.2	0.00730	125
140	140	41.8	0.0060	≥12500

Pyridine-3-carboxylic acid behaves fundamentally differently from all other combustible dusts investigated in this regard. G. Pellmont found that the reason for this was that niacin sublimates with increasing temperature. Taking into account the measurement results, the safety characteristics of niacin fine dust are summarised in Table 4:

Table 4. Safety characteristics of pyridine-3-carboxylic acid (niacin)

Safety characteristics		Valuation
Maximum explosion pressure p_{max}		8.6 bar
K_{St} - value		190 bar·m·s^{-1}
Dust explosion class		St1
Minimum explosion energy MIE	without inductance: T=20°C	5 - 10 mJ
	with inductance: T=20°C	5 - 10 mJ
	with inductance: T=80°C	125 mJ
	with inductance: T=140°C	≥12500 mJ
Minimum ignition temperature	dust cloud	>440°C
	dust layer	>365°C
Burning class number		BZ 5
Exothermic decomposition		Non until 230°C

Thus, neither ignition-effective electrostatic discharges nor mixture ignition by hot surfaces or spontaneous combustion are to be expected in the plate dryer, because the maximum temperature values given in Table 1 are clearly below the measured ignition temperatures, or below the temperatures required for decomposition. The determination of the permissible oxygen concentration was dispensed with and the assumption that the minimum ignition energy increases with temperature was confirmed by the results of systematic ignition tests (Figure 2). With this result, the danger of the ignition of niacin-dust/air mixture in the dryer by electrostatic brush discharges could be excluded, because the product, with additional consideration of the residual moisture (of 12 wt.% water), has a minimum ignition energy in the entry state that is already clearly above the specified equivalent energy for brush discharges.

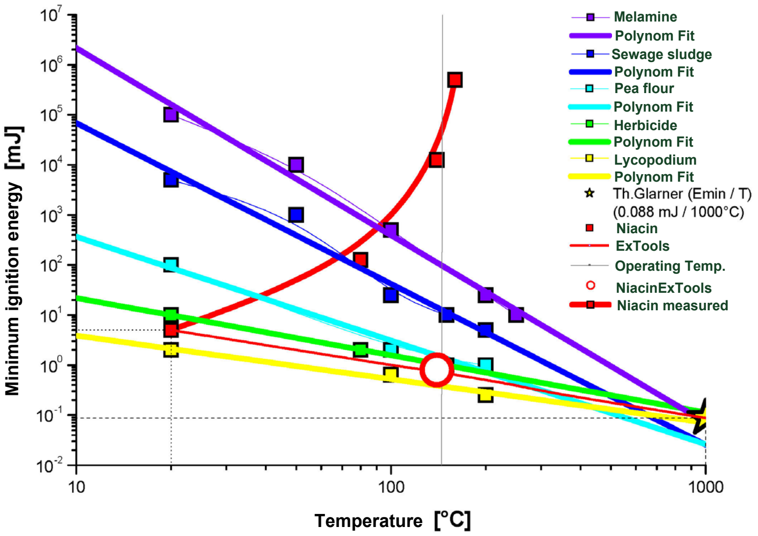

Figure 3. Influence of increasing temperature on the minimum ignition energy of pyridine-3-carboxylic acid

The results of the tests carried out with pyridine-3-carboxylic acid show that the information on safety characteristics, including their dependence on operational parameters, in tables of publications or regulations can only be indicative and are not necessarily generally valid. The results of the ignition tests in the 20-l laboratory apparatus also provided information on the explosion behaviour of niacin. Explosion pressure and rate of explosion pressure rise are falling with increasing temperature in comparison to other dusts (e.g., melamine polymer). The absolute values of both parameters depend on the type of ignition source (point source: capacitor discharge; flame jet: pyrotechnical igniter) and the turbulence. In this respect, too, the behaviour is different from what has been observed so far.

3. Influence of turbulence of combustibility of dusts in quasi-stationary state

A description about the determination of the new safety characteristic, the so-called fire behaviour number in quasi-stationary state (BVZ_{qs}), with the modified test apparatus according to Barth and Leksin (VDI 2263 Part 1 2021) was presented (Leksin & Barth 2019). The reason for this was that the combustion behaviour in the modified burning test instrument for fluid beds cannot be described with the standardized burning class number (combustibility index BZ for sedimented dusts) definitions.

Table 5. Definition of the fire behaviour number in quasi-stationary state (BVZ_{qs})

BVZ_{qs}	Type of reaction	
I	No fire behaviour	No spreading of fire
II	Local fire behaviour of the fluidized bed or jet flame in the presence of an ignition source	
III	Continuous fire behaviour with an open flame (torch fire)	Fire spreads
IV	Continuous fire behaviour over the entire length or rapid combustion with flame propagation	

Investigations with more as 40 different dusts had shown that the fire behaviour number in quasi-stationary state can't be estimated and thus the influence of the turbulence on the combustion behavior remains an unassessable factor. This is underlined by experimental results (table 6): the fire behaviour number in quasi-stationary state is not always increasing in comparison to the burning class number (for dust in sedimented form) but also for certain dusts the above-mentioned parameter (BVZ_{qs}) decreases or prevents the burning completely.

Table 6. Comparison of burning class number (BZ) and fire behaviour number in quasi-stationary state (BVZ_{qs}) of different dusts

Dust	BZ	BVZ_{qs}
Aluminium grinding powder	1	IV
Keton-resin	1	IV
Melamin-resin	1	I
Copolymer	1	II
Coffee creamer	2	IV
Sweet whey	2	III
Whey powder	2	II
Baby milk (NANPro)	3	I
Fish flavour	3	I
Protein	2	II

The investigations the fire behaviour number of different dusts in a quasi-stationary state had shown, as mentioned above, that the influence of turbulence cannot be estimated or calculated. The latest laboratory tests from 2020/2021 had also shown that certain dusts, which were determined with a BVZ_{qs} III in the modified test apparatus (according to Barth and Leksin), can have a fire behaviour number of BVZ_{qs} IV in larger fluidised bed systems or apparatus. Thus, this observation suggests that the factor of the geometry of the chamber or the size of the plant can also have a strong influence on the safety characteristic of substances. Probably it can be traced back to different heat transfer processes.

Experience of the author shows that explosion suppression systems are generally installed on fluid beds, as a constructional explosion protective measure. But real scale investigations of the effectiveness of such measures for different dusts in quasi-stationary state are missing. Such mistakes can be found still today in the industries e.g., on installations of explosion suppression systems on plants in which dusts with explosion class ST3 are handling. This means that companies, which produce constructive explosion protection equipment, have a lack in competence. This is due to the fact that a university education of engineers in the field of explosion technology is missed and the number of real-scale investigations is limited to a small number.

4. Conclusions

Both examples from the laboratory investigations shows, that there are a lot of other influencing variables that complicate the approximate predeterminations of explosion processes which are described in (Bartknecht & Pellmont 2018):

- temperature
- admission pressure
- humidity (for dusts)
- turbulence
- geometry of the explosion chamber
- location of the ignition site
- size of the explosion and combustion chamber
- effectiveness of "operational" ignition sources
- homogeneity
- chemical and physical substance properties and many others

Furthermore, the example of the influence of the turbulence on the burning behaviour of the dust in the quasi-stationary state had shown how extremely difficult the estimation can be, consequently the influence of turbulence on an explosion is much complicated. As can be concluded from this, postulates and interpretations for the evaluation or calculation of the ignition and explosion behaviour of combustible dusts, gases or vapours depending on the ambient conditions can only be used for those substances that have also been investigated or have been determined on a real scale and under real conditions.

Another important point is that the error rate resp. deviations are not taken into account when determining the safety characteristics. This could expand the field of research. Thus, the current tendency to simulate or model explosions will generate even more errors and misinterpretations, as there are too many influencing parameters that cannot be taken into account.

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