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Influence of pre-ignition pressure rise on safety characteristics of dusts and hybrid mixtures

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ABSTRACT

For the determination of the safety characteristics of dusts it is necessary to disperse the dust in the oxidating atmosphere (usually air). In the standard procedures for dusts this is realized by a partially evacuated explosion vessel (20L-sphere) in which the dust gets injected from a dust chamber pressurized with air. Shortly after that injection (60 ms) the dust cloud gets ignited under turbulent conditions, that are otherwise seen as almost ambient with 20 °C and about 1 bar (abs). While there has been a lot of research about the influence of the ignition delay time and the level of turbulence in the recent years little attention was paid to the pre–ignition pressure rise and the allowed variations in the standards. In the following work we showed that the allowed ranges for the pressures in the different dust standards influence the safety characteristics of dust alone severely.

Even though hybrid mixtures are an emerging risk problem in an interconnected industry there is no standard for the determination of their safety characteristics. In this work it is shown that especially for the preparation of hybrid mixtures of flammable dust and gas the pressures after injection of the dust and the mixing procedure have a large influence on the composition of the tested mixtures and therefore on the safety characteristics. Considering both effects, wrong concentration of gas and wrong initial pressure, the discrepancy of safety characteristics from different facilities will be too big to applicable. The methods to overcome these weaknesses are also presented.

1. Introduction

For the determination of safety characteristics of flammable dusts there are typically two standardized test-autoclaves: one is almost spherical and has an inner volume of 1 m^3 and the other one is spherical and has an inner volume of 20 L (the so called Siwek-chamber or 20L-sphere, European standard [7], US standard [1]). While the first one used to be the reference chamber the latter one is more popular today, because more research institutions and test facilities use it due to its smaller size and easier handling. It is necessary to disperse the dust in the oxidating atmosphere (usually air). In the test procedures for the 20L–sphere this is realized by injecting a dust from a dust container that is pressurized with air into the 20L-sphere that is previously evacuated to a pressure of 0.4 bar (abs). In the 1 m^3-chamber this first pressure rise is smaller, because the chamber is not evacuated before injection. This first pressure rise for dispersing the dust is called the **Pre-Ignition Pressure Rise** (**PIPR**, in the manufacturer's software called Pd [17]). 60 ms after the injection the dust cloud gets ignited in the 20L-sphere and after 600 ms in the 1 m^3-chamber.

While the focus of research regarding the test procedure has so far been on the ignition delay time (Li et al. [15], Chen and Zhang [2], Cao et al. [27]), the level of turbulence (Wheeler [23], Harris [11], Kundu et. al. [14]) or both (Garcia-Agreda et al. [9], Di Benedetto and Russo [4], Zhang and Zhang [26], Dahoe et al. [3], Zhen and Leuckel [24], Rodriguez [20], Skjold [22], Di Benedetto et al. [5]) little attention was paid to the PIPR, the allowed variations in the standards and the mixing procedure.

Especially for hybrid mixtures containing a flammable dust as well as a flammable gas the pressure compensation and the mixing procedure can have a great influence on the test result, because they affect the gas

Abbreviations: PIPR, Pre-Ignition pressure rise; PIPD, Post-injection pressure drop.

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composition significantly. The flammable gases are filled either in the 20L-sphere or in the dust container and usually the flammable gas concentration is calculated from the partial pressure fraction of the gas. Even though it has been known for a long time, that the explosion pressure is dependent on the initial pressure (Pilao et al. [28], Hertzberg et. al. [12], Glarner [10], SAFEKINEX Del. Nr. 8 [29], Lazaro ant Torrent [6], Pascaud and Gillard [19]) the allowed pressure ranges and the requirements for the pressure measurement systems in the standards for the determination of the safety characteristics of dusts can lead to a wide variation of initial pressures in the tests.

The pressure inside the 20L-sphere before injection of the dust, the PIPR and the subsequent pressure drop, that occurs due to thermodynamic equilibration by dissipation of the heat induced previously by adiabatic compression inside the sphere have to be taken into account for calculating the amount of flammable gas correctly in case of hybrid mixtures and to obtain robust and reproducible results for the determination of safety characteristics.

According to [1] a pressure before injection of 0.4 bar (abs) and a pressure at the ignition moment between 0.94 bar (abs) and 1.06 bar (abs) are stated but no accuracy of the pressure measuring system. According to the European standard EN 14,034 series a pressure before injection of 0.4 bar (abs) and an accuracy of the pressure measuring system of ± 0.1 bar is suitable resulting in a pressure before injection of the dust of 0.3 bar (abs) to 0.5 bar (abs).

1.1. Pre-ignition pressure rise (PIPR)

Table 1 shows the pressures, that are relevant for the mixing procedure and their allowed ranges in different standards.

The PIPR is only defined directly in the manufacturer's software, which shows an error, if the PIPR is lower than 0.55 bar or higher than 0.7 bar. The American standard defines the allowed PIPR indirectly with the pressure before injection and the pressure at ignition resulting in an allowed range between 0.54 bar and 0.66 bar. However, the required accuracy of the pressure measuring system is not specified. The European standard EN 14,034 series have a theoretical PIPR of 0.613 bar. Considering the defined accuracy the allowed range is between 0.513 bar and 0.713 bar. The maximum measuring uncertainty of the pressure measuring system is not mentioned in any of these standards.

1.2. Post-injection pressure drop

After the fast injection of the dust via pressurized air from the dust container the pressure is recorded and the whole mixture should have atmospheric pressure. Normally the ignition takes place shortly after the opening of the valve and this pressure shortly before ignition is stated as the initial pressure p_i in the software of the manufacturer. However, the heat rise due to the compression is not taken into account in the standard procedures. Especially, in case of hybrid mixtures, if this initial pressure is used for calculating the amount of flammable gas according to the partial pressures, this leads to wrong results. The effect of adiabatic compression is not mentioned in any standard and has not been found in research articles so far. Cashdollar observed the temperature increase while and decrease after the injection process, but does not mention the pressure rise [30].

Table 1
Pressure specifications stated in the standards for determination of safety characteristics of dusts and in the standard software of a manufacturer of the 20L-sphere.

Standard/ Procedure Source	Pressure before injection [bar (abs)]	Pressure at ignition [bar (abs)]	Accuracy of the pressure measuring system	PIPR [bar]
EN 14034	0.4	1.013	± 0.1 bar (or better)	**0.513–0.713***
ASTM 1226 - 2019	0.4	0.94–1.06	Not defined	**0.54–0.66***
Manufacturer's Software (Cesana (2020))	0.4	1	Not defined	***<u>0.55–0.7</u>***

*Not defined explicitly, but calculated from the other specifications.

1.3. Mixing procedures

The mixing procedure for the determination of the safety characteristics of hybrid mixtures in the 20L-sphere can be conducted in three different ways:

- Method I: A premixed gas-air mixture is used for both, the 20L-sphere and the dust container before ignition (see Fig. 1, left).
- Method II: The flammable gas is added in the 20L–sphere and dust is injected by pressurized air from the dust container (see Fig. 1, middle)
- Method III: The 20L-sphere is filled only with air and the dust container is pressurized by a mixture of flammable gas and air injecting the dust from the dust container (see Fig. 1, right)

The three different methods all have their benefits and disadvantages:

Method I: The most precise and homogeneous gas mixtures are obtained, if the explosion vessel and the dust container are both filled with the same premixture before injection of the dust. This method is the most complicated one, but has the benefit, that the following sources of error are avoided:

- 1: Discrepancy between the calculated gas concentrations, derived from measured pressures with the pressure sensors installed in the sphere and the real concentration of the gas
- 2: Local concentration variations because of incomplete mixing at the ignition moment after 60 ms of ignition delay time

The other two mixing procedures assume, that the gas-phase is homogenous at the ignition moment.

Method II has the benefit, that the flammable gas-air mixture is not under high pressure which makes this procedure the safest one. This procedure is more prone to the tolerances regarding the PIPR given in the standards. This method is recommended in the manufacturer's handbook [17] and applied by most of the research facilities.

Method III has the disadvantage, that flammable gas-air mixtures are pressurized to 21 bar (abs), so if an explosion occurs an overpressure of up to 200 bar (abs) might occur inside the dust container which is not

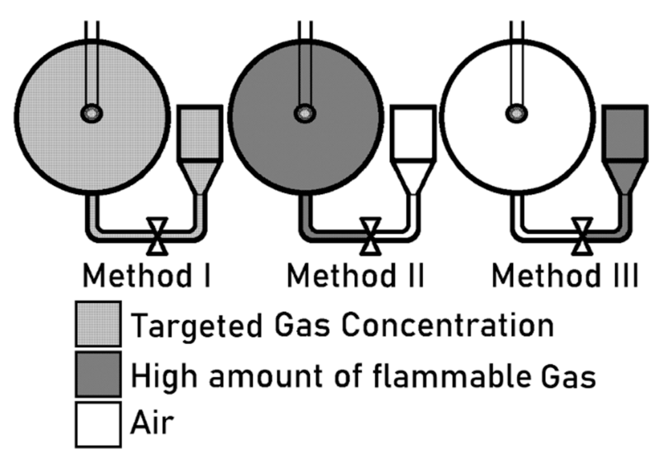

Fig. 1. Three different ways of mixing; Gas concentration before opening the fast-acting valve between 20L-sphere and dust container.

designed for these high pressures. The benefits of this method are that there are no modifications needed on the standard dust sphere and the test procedure is comparably short. This method is applied by Dalian University (China [16]) and Sichuan University (China, [25]).

A comparison between the three mixing procedures has not taken place so far and with that the two sources of error mentioned above have not been investigated as well.

2. Experimental methods

For all the experiments a standard 20L-sphere was used and a test procedure following the European standard EN 14034 was applied. The sphere was evacuated to 400 mbar (abs) ± 2 mbar in each test before dust-injection. The pressure development was recorded with the two piezoelectric pressure sensors that are installed in the 20L-sphere in the default configuration of the manufacturer. Two additional piezoresistive pressure sensors (company: Keller, type: PA-10, linearity: better than 0.5 % full scale) were installed for the tests, one with a resolution of 0.1 mbar and a range of 1 bar (abs), the other with a resolution of 1 mbar and a range of 10 bar (abs). The first one was just used for measuring the pressure before injection of the dust and had to be closed before initiating the ignition, the latter one for detecting leakages and determining the post-injection pressure drop by recording the pressure for three minutes after injection of the dust. As igniters two 1 000 J pyrotechnical igniters or optionally two exploding wires with a net energy of 1 000 J each were used, pointing in opposite direction placed in the center of the sphere according to the standard for the determination of explosion characteristics of dust clouds (EN 14034 series, [1]).

2.1. Determination of the pre-ignition pressure rise (PIPR)

For determining the PIPR in the 20L-sphere the allowed range of up to ± 100 mbar was tested. According to the standards the dust container is pressurized with air up to 21 bar (abs), which is theoretically necessary to achieve a pressure rise up to 1 bar (abs). But this does not take into account that clogging or friction of the dust might slow the injection, so that the pressure in the dust container at the ignition moment is still elevated and in the 20L-sphere the pressure is lower than atmospheric pressure. For the tests the air pressure in the dust container was adjusted with a pressure reducer between 16 bar (abs) and 27 bar (abs) to achieve the variety of PIPRs on purpose and investigate their influence on the safety characteristics of dust clouds. Except for the adjustment of the air-pressure, the tests were conducted with corn starch according to the European standard EN 14034 with corn starch.

In addition to the investigations in the 20L-sphere the PIPR in the 1 m^3 chamber also determined with one test without igniters and without dust and one dust chamber, that was pressurized to 21 bar (abs). The chamber was partially evacuated to 0.905 bar (abs) before dust-injection, so that the end pressure was about 1 bar (abs). The pressures were recorded with two piezoelectric pressure sensors at 4 kHz beginning 46 ms before the injection.

2.2. Determination of the post-injection pressure drop

For determining the post-injection pressure drop in the 20L-sphere several tests without triggering the ignition source and without dust were carried out. The PIPR was varied from 0.5 bar to 0.7 bar. In additional tests the pressure in the 20L-sphere before injecting the dust was varied from 0.3 bar (abs) to 0.5 bar (abs). In each test, it was waited for at least two minutes before injection of air to be sure, that the sphere and the air inside were fully thermally equilibrated. After the injection the pressure was recorded for three minutes with the 10–bar (abs) pressure sensor and additionally checked with the precise 1-bar (abs) pressure sensor. The recorded pressures were compared to the initial pressure before dust injection plus the PIPR the manufacturer's software stated.

For determining the post-injection pressure drop in the 1 m^3 chamber one test without igniters and without dust was conducted with one dust chamber, that was pressurized to 21 bar (abs). The chamber was partially evacuated to 0.905 bar (abs) before dust-injection, so that the end pressure was ~ 1 bar (abs). The pressures were additionally to the two piezoelectric pressure sensors recorded with one piezoresistive pressure sensor at 1 Hz for three minutes before and three minutes after the injection.

3. Results and discussion

3.1. Influence of the PIPR on the explosion overpressure

In Fig. 2 the explosion overpressure measured for 500 g/m^3 cornstarch is presented against the PIPR. The maximum explosion overpressure for the cornstarch sample was measured at 750 g/m^3, so the oxygen was available in excess in each test. Thus, the result was not influenced by the stoichiometric ratio, an effect that could be observed on the oxygen-lean side at higher amounts of cornstarch. All tests were performed with 2 000 J of ignition energy. Pyrotechnical igniters and exploding wires were used but no difference was observed between them, so they are presented with the same symbols.

One can clearly see the increasing explosion overpressure with increasing PIPR, which is mainly due to the higher initial pressure in the 20L-sphere. If a test laboratory conducts the tests at the lowest allowed PIPR with 0.5 bar it will obtain an averaged p_{ex} of 6.5 bar overpressure, another laboratory that conducts the tests with a PIPR of 0.7 bar, will determine a p_{ex} of 8.5 bar overpressure that is higher by 2 bar (or 30 percent). A similar tendency could be observed in tests with aluminum dusts and in literature this correlation is stated as well, though in wider ranges from 10 mbar (abs) to 2 bar (abs) [10]. To correct the error caused by different PIPRs, a simple formula can be used, derived from the standards for the maximum explosion pressure of gases [8]. One should keep in mind, that pressures in the standards for gases are always stated as absolute pressures, in the ones for dusts as overpressures. The original formula in the gas standard is just a ratio of absolute pressure

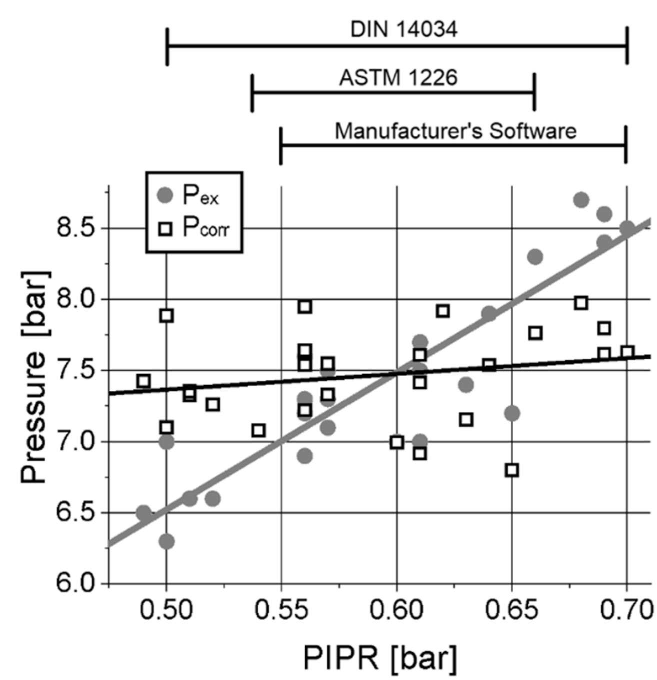

Fig. 2. Explosion overpressure of 500 g/m^3 cornstarch and calculated values acc. to formula (1) as a function of the PIPR with allowed ranges acc. to different standards, linear fit of Pex has an R^2 of 0.7874 and Pcorr of R^2 = 0.0467.

with the pressure at the ignition moment as p_0 and the highest explosion pressure as p_{ex}, in the following formula this is corrected by the ones.

$$P_{corr.} = \frac{P_{Ex} + 1}{P_0} - 1 \tag{1}$$

With this correction, the maximum variation from the mean value of p_{ex} decreases from ±18% to ±8% for PIPRs between 0.5 and 0.7. Considering the corrected p_{ex}-values, the linear correlation between the PIPR and p_{ex} characterized by the R-value is with ±0, 1 bar neglectable. So, it can be concluded that p_{ex} is hardly influenced by the PIPR, with an averaged pressure increase of only 3 % if the effect caused by different initial pressures is compensated, still the allowed range should be narrowed in future standards. For hybrid mixtures variations in the PIPR result in different gas concentrations, if the mixing is conducted by Method II or III (according to Fig. 1), what is usually the case so that the allowed variations should be narrowed and the PIPR should be stated with all test results.

The PIPR in the 1 m^3 chamber with one pressurized dust container was with 115 mbar lower than the PIPRs in the 20L-sphere (see Fig. 3). In cases with higher dust amounts two dust containers can be used so theoretically the PIPR may be around 200 mbar. For the reported values of p_{max} this means, the explosion pressures may be 10 % to 20 % higher than if they were measured under ambient pressure. In future standards, especially for hybrid mixtures this should be taken into account and investigated further, since usually the tests are conducted with initial pressures of 1.1 bar (abs) or 1.2 bar (abs) in the 1 m^3 chamber. Only few test facilities conduct the tests in the 1 m^3 chamber (against the standard but) with initial pressures of 1 bar (abs) [21,13].

3.2. Determination of the post-injection pressure drop

In Fig. 4 the pressure development in the 20L-sphere during and after injection of air is shown without the activation of the ignition source. The pressure increases directly up to the peak pressure. Afterwards the pressure decreases slightly and slowly because of dissipation of the heat that was induced previously by the fast compression. The end pressure after equilibration was measured after at least 180 s, even though after about 14 to 30 s the value did not change anymore.

Normally the ignition takes place at the peak pressure. Due to the explosion this pressure after equilibration and without igniter cannot be recorded (see Fig. 5).

The post-injection pressure drop is dependent on the PIPR and is slightly higher with higher initial pressures, even though this effect is

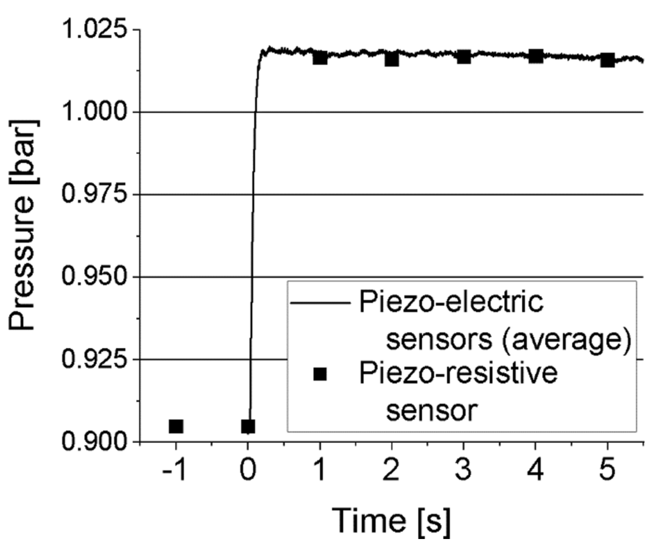

Fig. 3. Pressure against time measured for 1 m^3 chamber with two piezo-electric pressure sensors (average shown) and piezo-resistive pressure sensor.

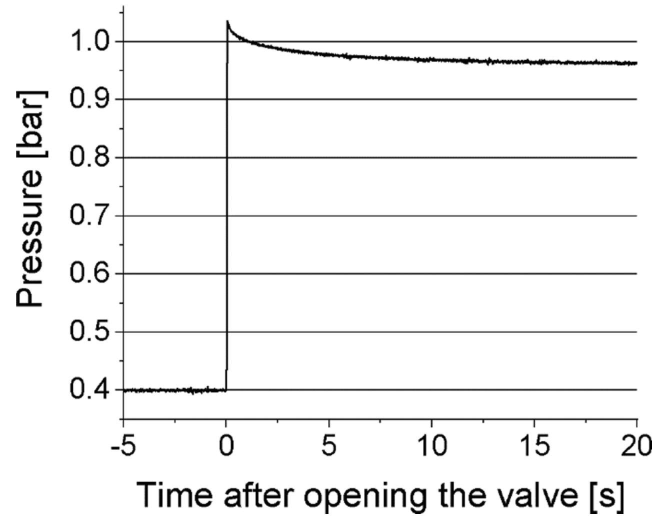

Fig. 4. Recorded pressure against time after opening the fast-acting valve without igniter and without dust (No explosion).

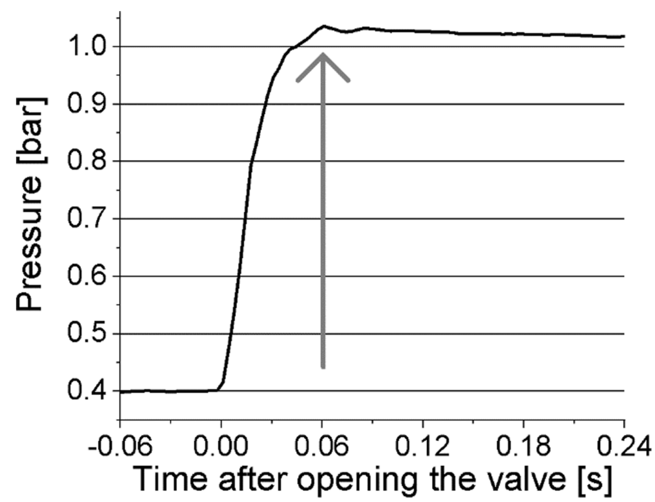

Fig. 5. Recorded pressure against time after opening the fast-acting valve without igniter and dust (No explosion), a close-up with the usual ignition moment pointed out.

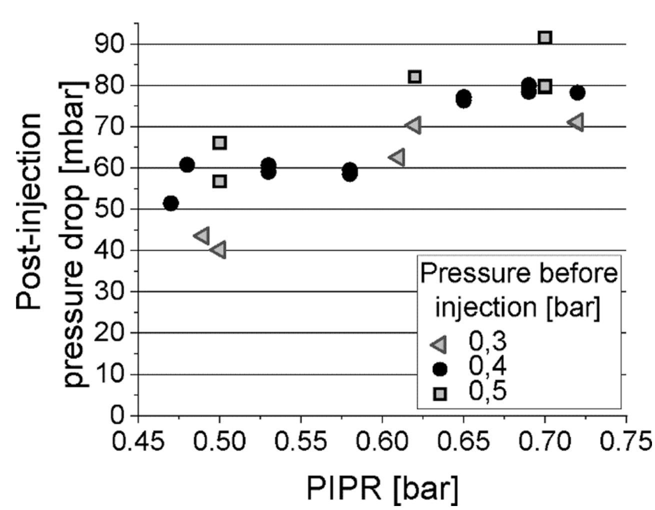

Fig. 6. Post-injection pressure drop against PIPR (acc. to the software) with 3 different initial pressures.

less obvious (see Fig. 6).

With a constant pressure of 400 mbar (abs) before injection and a PIPR of 0.64 bar (acc. to the software) an average pressure drop of 80 mbar ± 10 mbar was measured. If this is neglected in the calculation of the gas fraction the 80 mbar lead to a wrong calculated concentration of about 8 % rel. Calculating the fraction of flammable gas by partial pressures considering the PIPR and the post-injection pressure drop the remaining variation of the final pressure causes a variation (within ± 10 mbar) for the calculation of the gas component of about 1 % rel. For determining the safety characteristics of hybrid mixtures variations in the concentration of the flammable gas have a large influence on the test result, so it is necessary to reduce this variation to an acceptable level.

The initial temperature at the ignition moment can be estimated by the ideal gas law assuming homogeneous mixtures:

$$p^*V = n^*R^*T \tag{2}$$

Because the number of molecules (n), the gas constant (R) and the volume (V) don't change after closing the fast-acting valve the formula can be converted into

$$T_2 = T_1{}^*\frac{p_2}{p_1} \tag{3}$$

With T_1 = 293,15 K (water jacket temperature is set to 20 °C), p_2 = 1,013 bar (abs) = 1013 mbar (abs) and p_1 = 1013 mbar (abs) − 80 mbar = 933 mbar (abs), the temperature at the ignition moment is about 318,3 K or 45,1 °C. This should be considered because it could lead to an error in the determination of the safety characteristics. While most of the dusts react faster under higher temperatures others (for example the often-used niacin) are harder to ignite [18].

It shall be considered to use the full correction formula from the germa standard for the determination of maximum explosion pressure and rate of pressure rise of gases and vapours [8] for conditions other than the standard (for gases this is 25 °C and 1 bar (abs)), even though it leads to higher explosion pressures [10,29]. Thus, the comparability with literature data obtained according to the standard procedures for gases or dusts is complicated:

$$P_{max}(T,p) = \frac{P_{max}(T_1,p_1)^*T_1{}^*p}{T^*p_1} \tag{4}$$

The post-injection pressure drop in the 1 m^3 chamber was comparably low with 5 mbar (see Fig. 7). This is caused by the fact, that the pressure ratio after and before injection is highly disparate with 1.018 bar (abs): 0.905 bar (abs) ≈ 1.12 compared to the one in the 20L-sphere with 1 bar (abs): 0.4 bar (abs) ≈ 2.5.

The effect of the post-injection pressure drop for the calculation of the gas amount is comparably lower in the 1 m^3 chamber, and results in an error of only 0.5 % (rel.). The temperature increase in the 1 m^3 chamber is acc. to formula (3) also neglectable with less than 2 K.

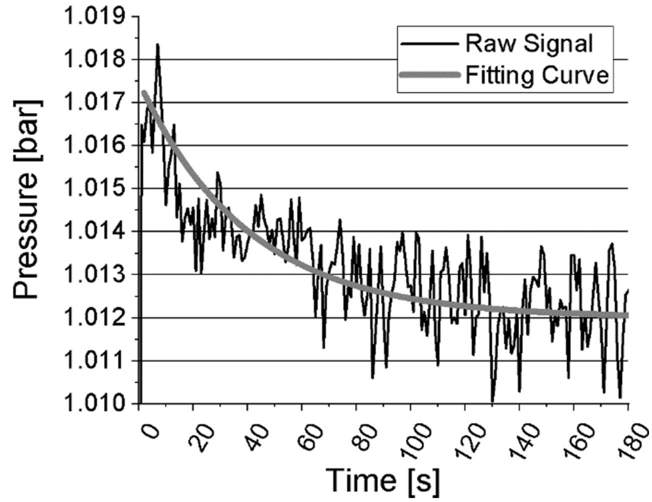

Fig. 7. Pressure against time measured for 1 m^3 chamber with a piezo-resistive pressure sensor, average displayed for better readability.

4. Conclusion

Variation in the PIPR, even within the ranges commonly given in standards, significantly affects the values of the explosion pressure (up to 30%) determined for dusts. Therefore, the PIPR should be fixed at a certain value, here recommended to be 0.64 bar. This value results in an overall pressure of 0.96 bar (abs) in the 20L-sphere after equilibration at 20 °C. Narrowing the range of PIPR values used, would result in more reproducible measurements of the explosion pressure. At a minimum, the PIPR should be stated in future data and reports. An extrapolation to the values recommended in this paper are possible if the PIPR is known.

For testing hybrid mixtures, the preparation of the gas mixtures is much easier directly in the 20L-sphere. So, for the calculation of the composition of hybrid mixtures according to the partial pressures both, the PIPR and the post-injection pressure drop play a big role and it should be investigated before conducting explosion tests. If both effects are disregarded this leads to wrong calculations of the gas amount and by that to highly flawed safety characteristics. Moreover, the accuracy of the pressure sensors that are usually installed in the 20L-sphere for dust explosion testing is too low for preparing gas mixtures. Consequently, for testing hybrid mixtures, the 20L-sphere should be modified by installing two additional pressure sensors, one with a higher accuracy that can be blocked before triggering the igniter to avoid destruction and one for determining the post-injection pressure drop.

For all our future tests the 0,64 bar ± 0,01 bar as PIPR were chosen, because with a pressure drop of 80 mbar it is the best compromise between being close to 1 bar (abs) at ignition and not too far away for the calculations and from comparison with the values from partner institutions.

If the shown effects are all disregarded, the highly flawed safety characteristics may lead either to expensive safety measures or to unsafe operation of processes.

CRediT authorship contribution statement

Stefan H. Spitzer: Conceptualization, Methodology, Software, Writing – original draft, Investigation, Visualization. **Enis Askar:** Writing – original draft, Validation, Conceptualization. **Alexander Benke:** Investigation, Resources. **Bretislav Janovsky:** Investigation, Resources, Data curation. **Ulrich Krause:** Supervision, Funding acquisition. **Arne Krietsch:** Project administration.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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