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Influence of the Pyrotechnic Igniter Composition Aging on Explosion Parameters of Dispersed Dusts

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Abstract: A commercially available pyrotechnic igniter was used according to the EN 14034 and ASTM E1226a Standards to study the explosiveness of dispersed dusts. Its pyrotechnic composition consists of 1.2 g of zirconium (40% wt.), barium peroxide (30% wt.) and barium nitrate (30% wt.). The energy released during the combustion of that amount of composition is 5 kJ. The article investigates the influence of aging of the pyrotechnic composition in the igniter on its initiation parameters. In the study, igniters of different years from date of manufacture were used: Igniter 1, manufactured in 2021 (less than 1 year from date of manufacture), and Igniter 2 (more than 2 years from date of manufacture). The study was performed in the KV 150M2 explosion chamber with a volume of 365 L and the 20 L sphere chamber with a volume of 20 L. A standard sample of *Lycopodium clavatum* was used in the KV 150M2 explosion chamber. Magnesium and benzoic acid were used as the samples in the 20 L sphere explosion chamber. The experiment showed that the explosion pressure P_{max} of the igniter with more than 2 years from date of manufacture decreased by up to 10%, while the value of the explosion constant K_{st} decreased by up to 40%. The attained results proved that aging of igniters affects their explosion parameters and measurement accuracy.

Keywords: dust explosion; pyrotechnic igniter; dispersed dust; magnesium powder; *Lycopodium clavatum* powder; benzoic acid

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1. Introduction

Explosions of dispersed dusts are characterized by certain parameters. According to the EN 14034 Standard, those are: P_{max}—the maximum explosion pressure of the dust sample, $(dP/dt)_{max}$—the maximum rate of the dust sample pressure increase, LEL—the lower explosive limit and MOC—the minimum oxygen concentration [1–3].

Determination of those parameters is determined by the ASTM E1226a and EN 14034 (1–4) Standards. The Standards specify the procedure and technical equipment necessary for the correct performance of the determination of explosion parameters. Non-compliance with the procedure, incorrect technical equipment and sample properties may have unfavourable effects on the attained values. This concerns mainly the values of Pmax and $(dP/dt)_{max}$. The explosion parameters can be affected by the shape and size of vessel, type of the igniter used, location of the igniter in the chamber, and the type and parameters of the disperser. The effect of the disperser on the samples was investigated by Sanrichico [4]. The influence of the disperser on the dust dispersion process and explosion parameters was investigated by Sarli et al. [5] and Portarapillo [6].

Explosion parameters are also affected by the sample properties, such as particle size and ratio, humidity, impurities, etc. Their influence has been studied especially by

Bartknecht [7], but one can also find the latest scientific publications that examine the issue of interest. For example, the effect of moisture was investigated by [8–11], while the effect of particle size was investigated by [10,12,13], etc.

The ASTM E1226a [2] and EN 14034 [1] Standards specify the igniter parameters determining the explosion characteristics of dispersed dusts. ASTM E1226A specifies the amount and composition of the pyrotechnic charge used (1.2 g, 40% wt. zirconium metal powder, 30% wt. barium nitrate, 30% wt. barium peroxide). When using an igniter according to the EN 14034 Standard, the main parameter is the calorimetric value of the released energy 2×5 kJ. Additional parameters of the igniter are the fuse head activation speed (10 ms) and the location of the igniter in a confined chamber [1,2].

To ignite a combustible dust–air mixture and achieve explosion, it requires an ignition source with adequate initiating energy. Initiating energy for dust clouds is generally stronger than that for gases. The type and strength of an ignition source have a significant impact on the initiation and progress of explosion [14,15].

The impact of the amount of initiation energy released from the igniter was discussed by Bartknecht [7], Field [16] and Eckhoff [17].

The energy of a pyrotechnic igniter could strongly affect certain explosion parameters, such as the K_{St} values and the lower explosion limits of dust explosions in the standard 1 m^3 vessel and the 20 L chamber. [14,15]

Some parameters that have an impact on the measured explosion characteristics are discussed by Ogle [18]. An igniter in the environment of hybrid mixtures has a specific effect on the course and results of the explosion process. The impact of different types of igniters on explosion characteristics was investigated by Janovský et al. [19], Xu et al. [20] and Spitzer et al. [21] The authors found that various types of igniters (a spark, pyrotechnic, exploding wire or resistive) have an impact on the $(dP/dt)_{max}$ value. The measurement results of hybrid mixtures seem to have been affected by particular measurement conditions; the attained values of explosion parameters of dispersed dust may be both higher or lower.

Although several researchers have reported the effects of ignition sources on determining certain explosion parameters of dust, little attention has been paid to the igniting behaviour of a pyrotechnic igniter itself or to its effect on the explosion dynamics, especially on flame propagation immediately after the ignition. [14,15]

Correct functionality of an igniter can be affected by:

1. Igniter shell
   - shell material (plastics/metal)
   - cover material
   - method of sealing the cover
2. Fuse head used in the igniter
   - type and composition of the fuse head pill
   - fuse head response speed
   - ignition ability of the fuse head
3. Pyrotechnic composition inside the igniter
   - components of the pyrotechnic composition
   - total energy
   - rate of energy release
   - humidity and ambient temperature (storage, handling)
   - manipulation with the igniter (vibrations, shocks, falls, etc.)
   - instability of pyrotechnic composition (instability of components, reactivity of individual components).

Influence of the igniter shell material on the explosion parameters was investigated by Zhen et al. [14] The authors studied the igniting behaviour of pyrotechnic igniters. Other authors have dealt with the influence of various igniters on explosion parameters, e.g., Hailin et al. [22], Gao et al. [23] and Benedetto [24].

The presented paper deals with the impact of the igniter charge aging on the explosion parameters of selected dust samples. The charge of a commercially available pyrotechnic igniter consists of a chemically resistant metal (Zr), chemically stable barium nitrate ($Ba(NO_3)_2$) and barium peroxide (BaO_2), which is chemically unstable in relation to the other two components. The unstable barium peroxide decomposes (for example, in the presence of humidity) over time according to the equation

$$BaO_2 \leftrightarrow BaO + \frac{1}{2}O_2 \tag{1}$$

The decomposition of barium peroxide and the presence of oxygen can cause a reaction with zirconium powder (reaction surface of powder is larger), thus causing a partial degradation of the pyrotechnic composition in the igniter. This can initiate changes in the igniter properties (reducing the amount of metallic zirconium, reducing the energy released by the igniter), and also affect the results of the explosion parameters measurement.

2. Materials and Methods

The measurement of explosion characteristics was performed in the KV 150 M2 explosion chamber with internal volume of 365 L (OZM Research, Figure 1) and the 20 L sphere (Kühner AG, Figure 2) explosion chamber with internal volume of 20 L. The two chambers were chosen to exclude the dependence of explosion parameters on the type of the equipment used.

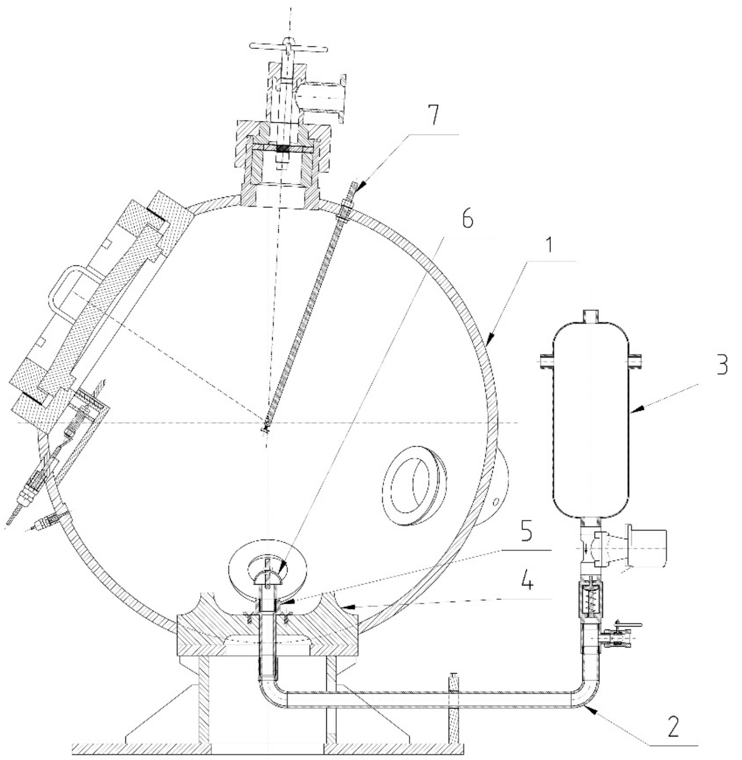


Figure 1. Cross section of 365 L explosion chamber (1—explosion spherical chamber, 2—disperser tube, 3—pressure vessel, 4—dispersing plate, 5, 6—disperser, 7—igniter rod) (Reprinted Ref. [25]).

Lycopodium clavatum was used as a standard sample, Figure 3. Explosion parameters of *Lycopodium clavatum* were measured in the KV 150M2 chamber. Explosion parameters of the sample of magnesium powder (−325 mesh, up to 45 μm) and benzoic acid were tested in the 20 L sphere explosion chamber, Figure 1. The parameters to be compared were measured at the concentrations of 250 and 500 $g \cdot m^{-3}$, and also at the concentrations of 750 and 1000 $g \cdot m^{-3}$ in the case of benzoic acid.

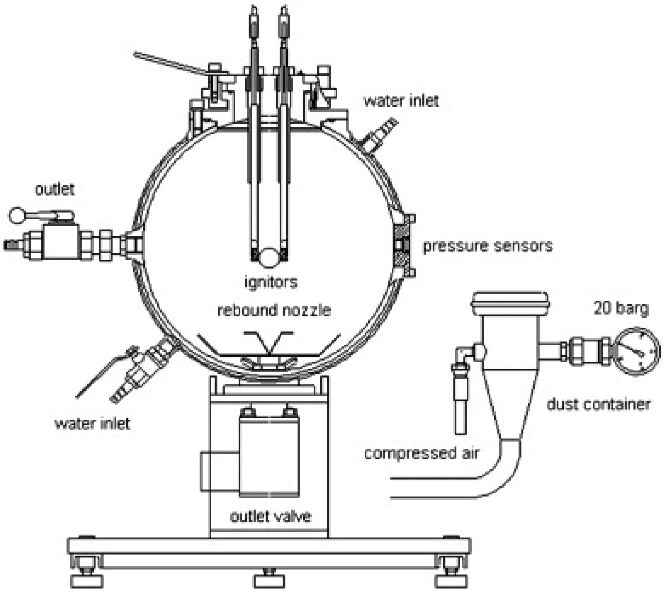
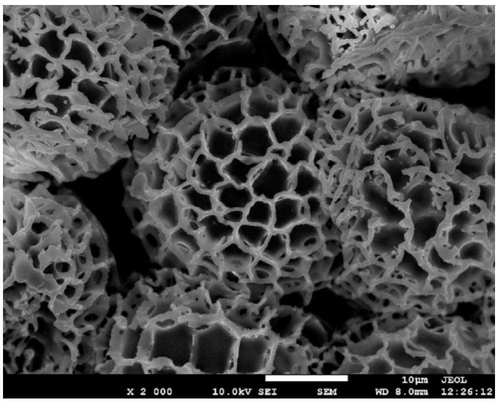
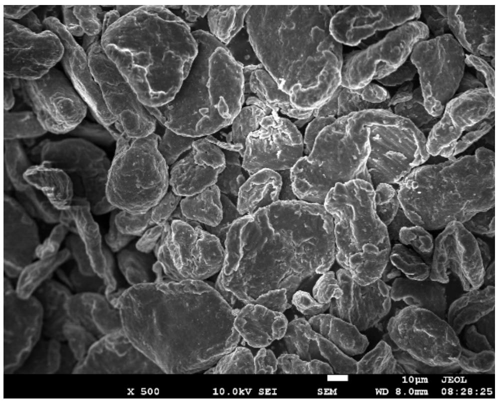


Figure 2. 20 L apparatus (Kühner AG) (Reprinted Ref. [26]).



(**a**)



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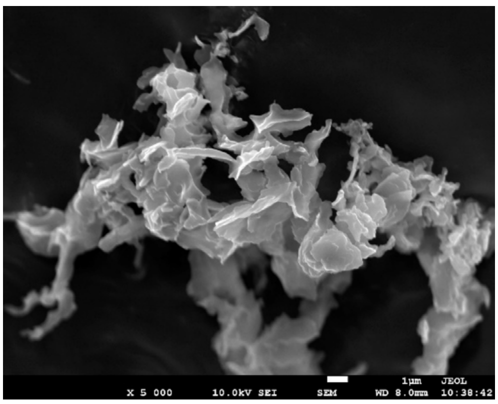

(**c**)

Figure 3. Microstructure of the samples (SEM). (**a**) *Lycopodium clavatum* powder particles, (**b**) Magnesium powder particles, (**c**) Benzoic acid sample.

Measurements of explosion parameters were performed in accordance with the EN 14034 Standard. Two pyrotechnic igniters (by Fr. Sobbe manufacturer) with the nominal energy value of 5 kJ/pcs were used for each measurement. The delay time in the KV 150M2 explosion chamber was 350 ms, and 60 ms in the 20 L sphere explosion chamber.

The Sobbe pyrotechnic igniters are charged with a pyrotechnic composition containing 1.2 g of the mixture (40%wt. zirconium metal, 30%wt. barium nitrate, 30%wt. barium peroxide). The igniter is activated by the fuse head within 10 ms. The cross section of the Sobbe lighter is shown in Figure 4.

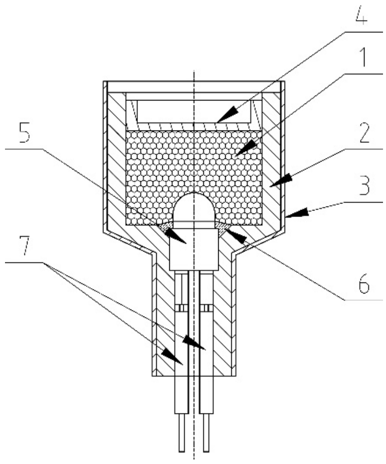

Figure 4. Cross section of commercially available igniter (redrawn according to patent application Chemical detonator with electric trigger—US 2016/0102957 A1 [27]) 1—pyrotechnic composition filling, 2—plastic case, 3—metal case, 4—stopper, 5—fuse head, 6—fuse head sealing, 7—priming wires.

In the KV 150M2 explosion chamber, the measurements were conducted at a speed of 50,000/s, using a Kulite pressure transducer. In the 20 L sphere explosion chamber, the measurements were conducted at a speed of 5000/s, using a piezoelectric pressure transducer.

3. Results

The experiment described in the article studied the possible influence of storage period under common conditions (15–30 °C, humidity 40–60% RH) on degradation of the pyrotechnic charge of the Sobbe igniter. Igniters manufactured in 2021 and in 2018 were used for the measurement. The storage period of the igniter manufactured in 2021 was less than 1 year. The storage period of the igniter manufactured in 2018 was more than 2 years (up to 3 years). Igniter 1 and Igniter 2 were selected based on the data provided by the manufacturer, who recommends using the igniter within two years from the date of manufacture, see Figure 5. The current paper does not study the aging process of the pyrotechnic composition in the SOBBE igniter. The aging process of a pyrotechnic composition can be determined, e.g., according to the Pyrotechnic materials—Directive 2013/29/EU–EC Other pyrotechnic articles (category P1 and P2)—EN 16263. However, the pyrotechnic composition used in the igniter is extremely dangerous (handling, friction, sparks, fire, etc.), and its properties and parameters can be determined only in a workplace equipped with specific technology for the given purpose, material support and authorised staff.

The measurement was performed with three dust samples at the concentrations of 250 and 500 g·m^{-3} in the KV 150M2 explosion chamber and in the 20 L sphere explosion chamber.

Pressure records to compare the course of the explosion of overpressure P and rate of pressure rise dP/dt of the *Lycopodium clavatum* sample in the KV 150M2 chamber are shown in Figures 6 and 7 (for the concentration of 250 g·m^{-3}), and in Figures 8 and 9 (for the concentration of 500 g·m^{-3}).

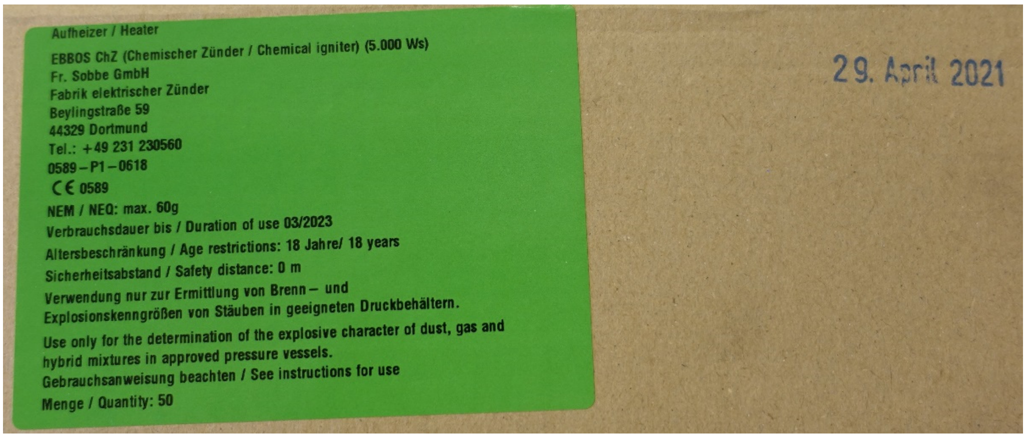

Figure 5. Sobbe igniter label.

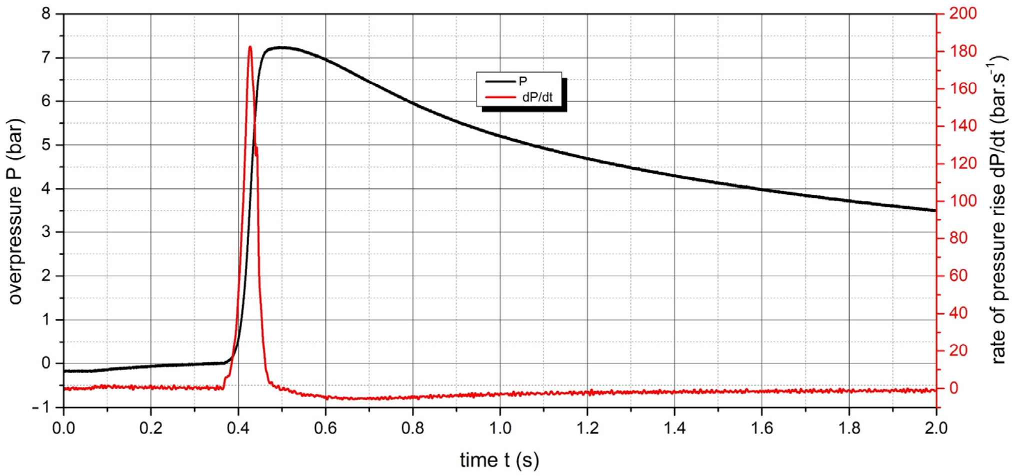

Figure 6. Overpressure P and rate of pressure rise dP/dt of 250 g·m^{-3} *Lycopodium clavatum* powder ignited by Igniter 1 (shelf life less than 1 year).

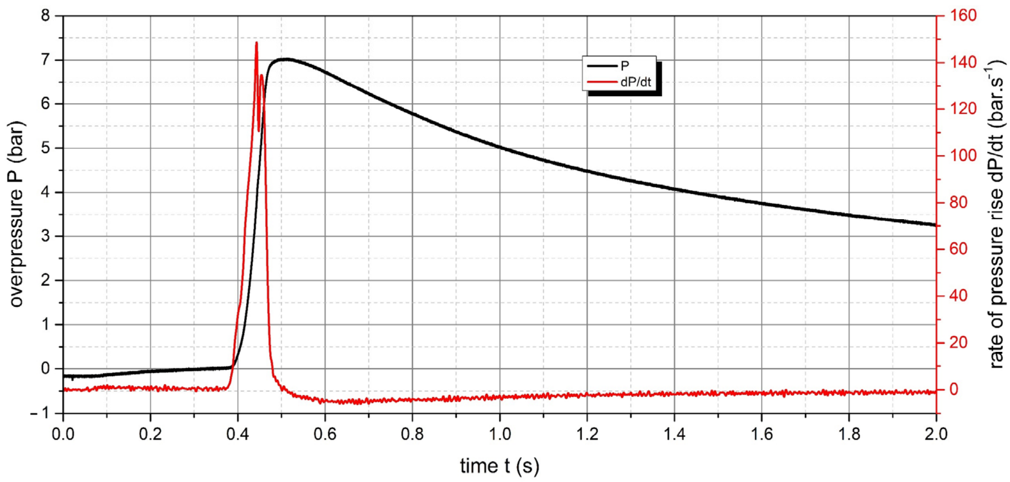

Figure 7. Overpressure P and rate of pressure rise dP/dt of 250 g·m^{-3} *Lycopodium clavatum* powder ignited by Igniter 2 (shelf life more than 2 years).

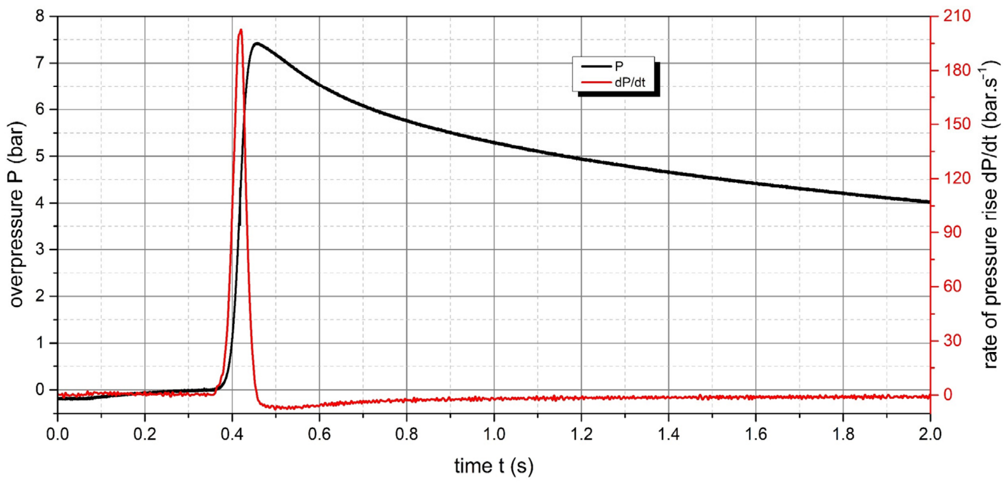

Figure 8. Overpressure P and rate of pressure rise dP/dt of 500 $g \cdot m^{-3}$ *Lycopodium clavatum* powder ignited by Igniter 1 (shelf life less than 1 year).

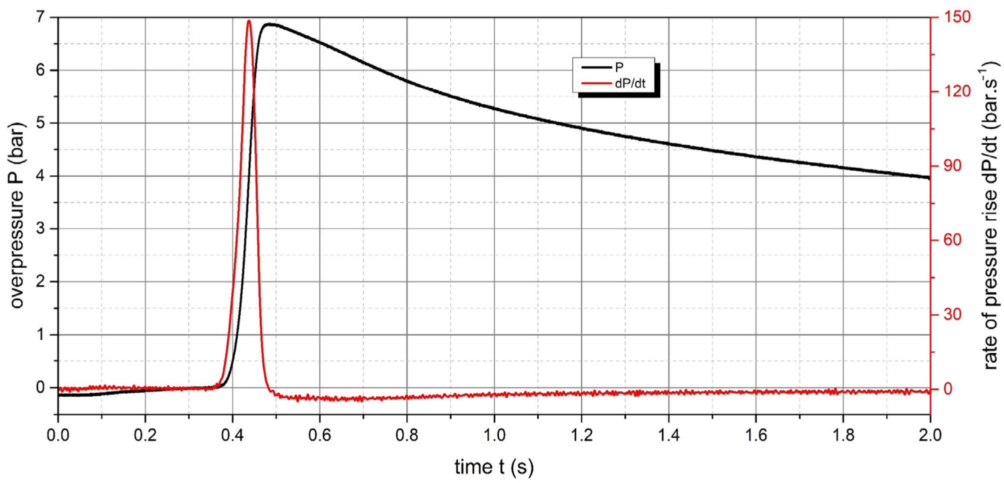

Figure 9. Overpressure P and rate of pressure rise dP/dt of 500 $g \cdot m^{-3}$ *Lycopodium clavatum* powder ignited by Igniter 2 (shelf life 2 years).

A sample of benzoic acid and magnesium powder was measured in a 20 L sphere blast chamber. The comparison of the measured explosion parameters with the deviation is shown in Table 1.

The value of the explosion constant K_{st} is calculated by the formula

$$K_{st} = \sqrt[3]{V} \times \left(\frac{dP}{dT}\right)_{max} \tag{2}$$

The explosion constant for a standard sample of *Lycopodium clavatum* (Igniter 1) at a concentration of 250 and 500 $g \cdot m^{-3}$ is calculated as

$$K_{st,250,\ igniter\ 1} = \sqrt[3]{V} \times \left(\frac{dP}{dt}\right)_{max} = \sqrt[3]{0.365\ m^3} \times 182.7\ bar \cdot s^{-1} = 130.6\ bar \cdot m \cdot s^{-1} \tag{3}$$

$$K_{st,500,\ igniter\ 1} = \sqrt[3]{V} \times \left(\frac{dP}{dt}\right)_{max} = \sqrt[3]{0.365\ m^3} \times 208.5\ bar \cdot s^{-1} = 149.0\ bar \cdot m \cdot s^{-1} \tag{4}$$

The value of deviation is calculated by the formula

$$\delta = \frac{\text{value}_{\text{igniter2}} - \text{value}_{\text{igniter1}}}{\text{value}_{\text{igniter1}}} \times 100 \quad (5)$$

Table 1. Maximum values of explosion parameters for individual concentrations attained during the Igniter 1 and Igniter 2 measurements.

Concentration	Parameter	Igniter 1 <1 Year from Date of Manufacture	Igniter 2 2–3 Years from Date of Manufacture	Deviation δ %
Lycopodium clavatum, characteristic particle size 30–32 μm (365 L chamber)				
$250\ g{\cdot}m^{-3}$	$P_{max,250}$ (bar)	7.21	7.03	−2.5
	$(dP/dt)_{max,\,250}$	182.7	148.6	−18.7
	$K_{st,\,250}$ $(bar{\cdot}m{\cdot}s^{-1})$	130.6	106.2	
$500\ g{\cdot}m^{-3}$	$P_{max,\,500}$ (bar)	7.43	6.88	−7.4
	$(dP/dt)_{max,\,500}$	208.5	149.4	−28.3
	$K_{st,\,500}$ $(bar{\cdot}m{\cdot}s^{-1})$	149.0	106.8	
Magnesium powder < 45 μm (−325 mesh), median 33.1 μm (20 L chamber)				
$250\ g{\cdot}m^{-3}$	$P_{max,\,250}$ (bar)	6.2	6.0	−3.2
	$(dP/dt)_{max,\,250}$	453	268	−40.7
	$K_{st,\,250}$ $(bar{\cdot}m{\cdot}s^{-1})$	123	73	
$500\ g{\cdot}m^{-3}$	$P_{max,\,500}$ (bar)	8.2	7.7	−6.1
	$(dP/dt)_{max,\,500}$	519	409	−21.3
	$K_{st,\,500}$ $(bar{\cdot}m{\cdot}s^{-1})$	141	111	
Benzoic acid, median 48 μm (20 L chamber)				
$250\ g{\cdot}m^{-3}$	$P_{max,\,250}$ (bar)	7.2	6.8	−5.6
	$(dP/dt)_{max,\,250}$	719	578	−19.5
	$K_{st,\,250}$ $(bar{\cdot}m{\cdot}s^{-1})$	195	157	
$500\ g{\cdot}m^{-3}$	$P_{max,\,500}$ (bar)	7.6	7.4	−2.6
	$(dP/dt)_{max,\,500}$	980	939	−4.1
	$K_{st,\,500}$ $(bar{\cdot}m{\cdot}s^{-1})$	266	255	
$750\ g.m^{-3}$	$P_{max,\,750}$ (bar)	7.2	7.2	0.00
	$(dP/dt)_{max,\,750}$	982	960	−2.2
	$K_{st,\,750}$ $(bar{\cdot}m{\cdot}s^{-1})$	267	261	
$1000\ g{\cdot}m^{-3}$	$P_{max,\,1000}$ (bar)	6.6	6.6	0.00
	$(dP/dt)_{max,\,1000}$	821	789	−3.9
	$K_{st,\,1000}$ $(bar{\cdot}m{\cdot}s^{-1})$	223	214	

4. Discussion

The measured explosion parameters of the samples indicate that aging of the igniters may affect their functional parameters. In the KV 150M2 explosion chamber, we detected a 4–7% decrease in the explosion pressure when using Igniter 2 (manufactured in 2018) and a decrease in the pressure increase rate of 17–29% (K_{st} constant) in a standard sample of *Lycopodium clavatum*. For Igniter 1 (manufactured in 2021), the explosion parameters corresponded with the standard characteristics according to the ASTM E1226a Standard.

Measurement of the magnesium sample in the 20 L sphere explosion chamber identified that the value of the explosion pressure P_{max} was reduced by 3–6%, while for the igniters with more than 2 years from the date of manufacture, the decrease in the explosion constant K_{st} was 20–40%.

For igniters with more than 2 years from the date of manufacture, measurement of the sample of benzoic acid in the 20 L sphere explosion chamber recorded a decrease of 2.5–5.5% in the case of explosion pressure P_{max}, as well as a decrease of 4–20% in the case of explosion constant K_{st}.

The manufacturer specifies the consumption period of 2 years for the igniters. Two types of igniters were therefore selected for this study. Igniter 1 was used within 1 year from the date of manufacture. Igniter 2 was used for 2–3 years from the date of manufacture. The EN 14034 Standard determines permissible deviation of the Pmax value of 10% (for each concentration in the experiment described in the current paper). The results in Table 1 show that the Pmax value of the igniters meets the requirement of the maximum tolerance even after expiration of their consumption time.

The tolerance for determining $(dP/dt)_{max}$ depends on its value. Table 2 shows that, in the case of using the igniters after the specified expiration time, the value $(dP/dt)_{max}$ for *Lycopodium clavatum* and magnesium samples decreases below the permissible tolerance. Aging of the igniter does not have such a significant effect in the case of benzoic acid; the permissible deviation (dP/dt) max was exceeded for the sample of benzoic acid only at a concentration of 250 $g \cdot m^{-3}$.

Table 2. Permissible deviations of $(dP/dt)_{max}$ measurement for standard equipment of 1 m^3 (value $(dP/dt)_{max}$ correspond to value K_{st}).

$(dP/dt)_{max}$ $bar \cdot s^{-1}$	Maximal Relative Deviation %
<50	±30
50–100	±20
100–200	±12
>200	±10

5. Conclusions

The measurement results confirmed that the explosion parameters may vary depending on the age of the pyrotechnic igniters used. Igniters with a storage period more than 2 years exhibited the value of maximum explosion pressure at various concentrations being reduced by 10%.

The age of an igniter significantly affects the value of the explosion constant. For the igniters with the storage period more than 2 years, the value of the explosion constant K_{st} can be reduced at various concentrations by up to 40%.

The findings above suggest that the exceeded service life of the igniter does not have a significant effect on the measured values of explosion pressure at different sample concentrations. However, exceeding the service life has a significant effect on the pressure rise rate $(dP/dt)_{max}$ at different sample concentrations.

Based on the above-mentioned results, we recommend to use igniters with a storage period of less than 1 year when measuring explosion parameters. The storage period of an igniter and its use in the explosion chamber can affect the obtained explosion parameters of the samples and, thus, reduce the accuracy of the results.

In the future, research in cooperation with the Institute of Energetic Materials of University of Pardubice will focus on a detailed study of the aging process of a pyrotechnic igniter. The research will focus on the pyrotechnic composition itself and its influence on the explosion parameters of dispersed dusts.

Author Contributions: R.K., Z.S. and M.M. conceived and designed the experiments; R.K., Z.S., M.S., M.M., P.L. and L.K. performed the experiments and analysed the data; R.K. and Z.S. managed all the experimental and writing process as the corresponding authors. All authors discussed the results and commented on the manuscript. All authors have read and agreed to the published version of the manuscript.

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