

## Igniter-induced hybrids in the 20-I sphere

Taveau, J. R.; Going, J. E.; Hochgreb, S.; Lemkowitz, S. M.; Roekaerts, D. J.E.M.

**DOI**

[10.1016/j.jlp.2017.07.014](https://doi.org/10.1016/j.jlp.2017.07.014)

**Publication date**

2017

**Document Version**

Accepted author manuscript

**Published in**

Journal of Loss Prevention in the Process Industries: the international journal of chemical and process plant safety

**Citation (APA)**

Taveau, J. R., Going, J. E., Hochgreb, S., Lemkowitz, S. M., & Roekaerts, D. J. E. M. (2017). Igniter-induced hybrids in the 20-I sphere. *Journal of Loss Prevention in the Process Industries: the international journal of chemical and process plant safety*, 49(Part B), 348-356. <https://doi.org/10.1016/j.jlp.2017.07.014>

**Important note**

To cite this publication, please use the final published version (if applicable). Please check the document version above.

**Copyright**

Other than for strictly personal use, it is not permitted to download, forward or distribute the text or part of it, without the consent of the author(s) and/or copyright holder(s), unless the work is under an open content license such as Creative Commons.

**Takedown policy**

Please contact us and provide details if you believe this document breaches copyrights. We will remove access to the work immediately and investigate your claim.

## Igniter-Induced Hybrids in the 20-l Sphere

J. R. Taveau <sup>a,b</sup>

J. E. Going <sup>a</sup>

S. Hochgreb <sup>c</sup>

S. M. Lemkowitz <sup>d</sup>

D. J. E. M. Roekaerts <sup>b,e</sup>

<sup>a</sup> Fike Corporation, Blue Springs, Missouri, United States of America

<sup>b</sup> Section Fluid Mechanics, Department of Process and Energy, Delft University of Technology, Delft, The Netherlands

<sup>c</sup> Department of Engineering, University of Cambridge, England, United Kingdom

<sup>d</sup> Department of Chemical Engineering, Delft University of Technology, Delft, The Netherlands

<sup>e</sup> Section Multiphase & Reactive Flows, Department of Mechanical Engineering, Eindhoven University of Technology, Eindhoven, The Netherlands

### Abstract

Dust explosibility is traditionally described by two parameters, namely the maximum explosion pressure,  $P_{\max}$ , and the deflagration index,  $K_{St}$ , usually determined through testing in a closed, pressure-resistant spherical vessel, either 20 liter or 1 m<sup>3</sup> in volume. These parameters constitute key variables in the design of explosion protection systems, such as venting, suppression or isolation systems.

The potential for overdriving dust combustion with pyrotechnical igniters in the 20-l sphere has been recognized, discussed and analyzed for many years, notably in the determination of the minimum explosible and limiting oxygen concentrations, which has led to specific guidelines regarding the ignition source strength in ASTM standards.

The current paper presents new experimental evidence that the energy provided by pyrotechnical igniters may, in some instances, physically alter the dust being tested in the 20-l sphere.  $K_{St}$  values can be several times greater in the small vessel compared to those measured in the 1-m<sup>3</sup> chamber. Further visual evidence is provided to show that high energy ignition can produce a turbulent flame region, possibly consisting of a hybrid mixture of flammable gas (or vapor) and dust, which can propagate faster than the corresponding pure dust. The experiments suggest that  $K_{St}$  values measured in the 20-l sphere may no longer be representative of a dust deflagration in a real process environment. We recommend additional tests in a 1-m<sup>3</sup> chamber when a dust exhibits a low flash point, or when its  $K_{St}$  is above 300 bar.m/s in the 20-l sphere.

**Keywords:** dust; deflagration; igniter; 20-l sphere; overdriving; hybrid

**Email:** [jerome.taveau@fike.com](mailto:jerome.taveau@fike.com)

**Nomenclature:**

ASTM	American Society of Testing and Materials
$K_G$	Deflagration Index for Gases (bar.m/s)
$K_{St}$	Deflagration Index for Dusts (bar.m/s)
LOC	Limiting Oxygen Concentration (% O <sub>2</sub> )
MAIT	Minimum Auto Ignition Temperature (°C)
MAP	Mono Ammonium Phosphate
MEC	Minimum Explosible Concentration (g/m <sup>3</sup> )
MIC	Minimum Inerting Concentration (g/m <sup>3</sup> )
$P_{max}$	Maximum Explosion Pressure (barg)
PPC	Pulverized Pittsburgh Coal
SBC	Sodium Bicarbonate

## 1. Introduction

A dust explosion occurs when an airborne combustible dust cloud encounters an effective ignition source. The resulting pressure and temperature increase can severely injure people and damage surrounding equipment and buildings, and therefore needs to be prevented or controlled.

The severity of a dust explosion is described by two parameters, the maximum explosion pressure  $P_{\max}$  and the deflagration index  $K_{St}$ , where the latter is the product of the maximum rate of pressure rise and the cube root of the vessel volume.  $P_{\max}$  and  $K_{St}$  are determined through testing in a closed, pressure-resistant spherical vessel: a known quantity of dust is dispersed in the vessel and the resulting dust cloud is ignited after a certain delay by pyrotechnical igniter(s) placed at the center of the vessel.  $P_{\max}$  is determined based on the maximum pressure reached during the deflagration test, while  $K_{St}$  is calculated using the slope of the steepest part of the pressure-versus-time curve recorded during the deflagration.

A 20-l sphere apparatus, as well as a modified testing protocol, have been developed by Siwek (1977) as an alternative for the 1-m<sup>3</sup> chamber introduced by Bartknecht (1981) in order to achieve cheaper and faster tests. Several modifications (volume of the dust container, ignition delay time, dispersion systems) were made so the results found in the 20-l sphere would match the results of the 1-m<sup>3</sup> chamber (Figure 1). However, the same pyrotechnical igniters were used to perform explosion tests.



Figure 1: Photo of 20-l sphere (left) and 1-m<sup>3</sup> chamber (right) operated by Fike Corporation

The potential for overdriving dust combustion with pyrotechnical igniters in the 20-l sphere has been recognized, discussed and analyzed for many years (Cashdollar and Chatrathi, 1992; Mintz, 1995; Cashdollar, 2000; Going et al., 2000; Cloney et al., 2013; Gao et al., 2013). The current paper presents new experimental evidence that the strong pyrotechnical igniters employed for dust explosibility testing may physically alter some dusts being tested in a 20-l sphere in such a way, that a flammable gas (or vapor) and dust hybrid mixture is formed prior to the actual arrival of the flame front.

Section 2 of the present article summarizes previous studies relative to the effect of ignition energy on dust explosibility. The effects of pyrotechnical igniters on the initial pressures and temperatures in the 20-l sphere and in the 1-m<sup>3</sup> chamber are reviewed in

section 3. Section 4 presents new experiments carried out in the two vessels for the same dusts, showing large discrepancies in  $K_{St}$  values. Finally, section 5 discusses the experimental evidence obtained and proposes three alternative ignition/combustion mechanisms for the dusts tested.

## 2. Effect of ignition energy on dust explosive properties: previous experimental investigations

### 2.1 Effect of ignition energy on deflagration index

Zhen and Leuckel (1997) were among the first to recognize, describe and study the effects of pyrotechnical igniters on dust explosions. They conducted dust explosion tests in a 1-m<sup>3</sup> chamber with cornstarch using 10-kJ and 75-J pyrotechnical igniters. Values of  $K_{St}$  are consistently higher for a 10-kJ ignition energy. The authors proposed that pyrotechnical igniters may accelerate the burning rate during an explosion due to volumetric and/or multipoint ignition effects. The extent of this overdriving is related not only to the energy of the igniters, but also to the reactivity of the mixture.

Proust et al. (2007) measured the  $K_{St}$  of different dusts in both a 20-l sphere and a 1-m<sup>3</sup> cylindrical chamber using a 10-kJ ignition energy in each case. While the correlation in the results between the two vessels was reasonable, four of the tested dusts had low  $K_{St}$  values in the 20-l sphere (sodium monochloroacetate, Lixivalt, Metco, and solid sewing residues), but were found to be non-explosible when tested in the 1-m<sup>3</sup> chamber. The authors suggested that a dust with a  $K_{St}$  below 45 bar.m/s as measured in the 20-l sphere test would likely be shown to be non-explosible when tested in a 1-m<sup>3</sup> chamber (Figure 2, dashed region).

More recently, Thomas et al. (2013) conducted screening explosibility tests per ASTM E1226 with urea dust in both a 20-l sphere (with either 1 or 2 x 5 kJ igniters) and Fike 1-m<sup>3</sup> chamber (with either 1 or 2 x 10 kJ igniters). They determined that the urea dust was explosible in the small vessel, but not explosible in the large vessel (Table 2). They concluded that the “false positive” result obtained in the 20-l sphere was the result of overdriving the combustion process, while testing in the 1-m<sup>3</sup> chamber allowed the urea dust to be properly characterized. They recommended testing low- $K_{St}$  dusts in a vessel larger than 20-l, in which the flame must propagate over a certain distance in order to develop a maximum explosion pressure  $P_{max}$  value sufficiently high to classify the dust as explosible.

Vessel volume (m <sup>3</sup> )	Igniter energy (kJ)	Result
0.020	5	No ignition
0.020	10	Ignition $P_{max} = 5.4$ bar $K_{max} = 21$ bar.m/s
1	10	No ignition
1	20	No ignition

Table 2. Results of screening tests with urea in the 20-l sphere and Fike 1-m<sup>3</sup> chamber at varying ignition energies (Thomas et al., 2013)

Gao et al. (2013) conducted tests in a 20-l sphere to examine the effect of four different igniters on the explosibility of 1-Octadecanol (C<sub>18</sub>H<sub>38</sub>O) powder, which melting, flashing and boiling points are respectively 60, 195 and 345 °C. They observed that varying ignition energy influenced P<sub>max</sub>, and more significantly K<sub>St</sub> (Figure 3). The maximum reactivity is reached at a dust concentration of 500 g/m<sup>3</sup>, with K<sub>St</sub> varying from 49 bar.m/s (2.5-kJ electrostatic ignition) to 167 bar.m/s (10-kJ pyrotechnical ignition).

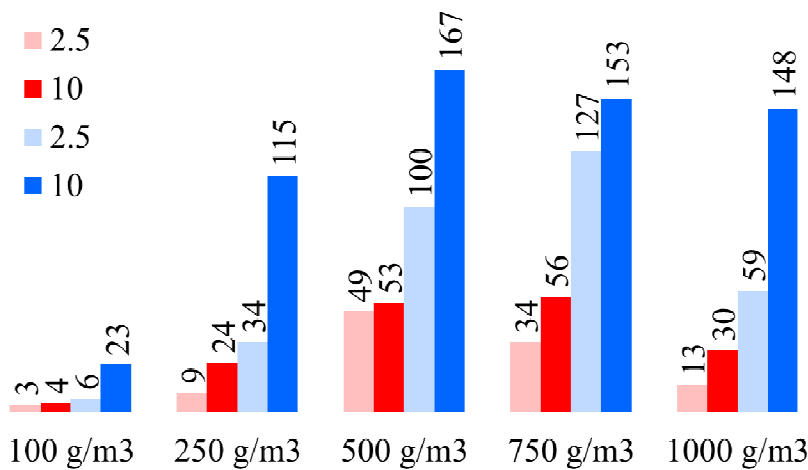


Figure 3: K<sub>St</sub> values (bar.m/s) as a function of dust concentration (g/m<sup>3</sup>) in the 20-l sphere for 1-Octadecanol using electrostatic igniters (red) and pyrotechnical igniters (blue)

## 2.2 Effect of ignition energy on minimum explosible and limiting oxygen concentrations

Going et al. (2000) present a comparison of minimum explosible concentrations (MEC) and limiting oxygen concentrations (LOC) determined in a 20-l sphere and in a 1-m<sup>3</sup> chamber. All tested dusts had similar low moisture content (below 3%) and comparable median particle size (between 20 and 44 μm), but with a wide range of chemistry and volatile content (Table 3). Results of their investigation are summarized on Figures 4 and 5.

Dust	Median particle size (μm)	Volatile Content (%)
Tetramethylpiperidine (RoRo93)	29	100
Pulverized Pittsburgh Coal (PPC)	44	37
Gilsonite	28	84

Lycopodium	28	92
Aluminum	20	0
Iron	23	0

Table 3. Physical and chemical properties of the tested dusts (Going et al., 2000)

The measured MECs from the 1-m<sup>3</sup> chamber are essentially independent of ignition energy over the range studied, unlike tests in the 20-l sphere: the apparent MEC decreases with increasing ignition energy, and is notably lower than the 1-m<sup>3</sup> chamber results in the case of carbonaceous dusts (Figure 4). The authors attributed this behavior to overdriving of the dust combustion by the strong pyrotechnical igniters. As expected, the closest comparisons between the MEC in the larger and smaller vessels appear for low ignition energies. However, the data also show that the more difficult-to-ignite dusts, such as iron, with higher MEC values require a greater ignition energy of 5-kJ.

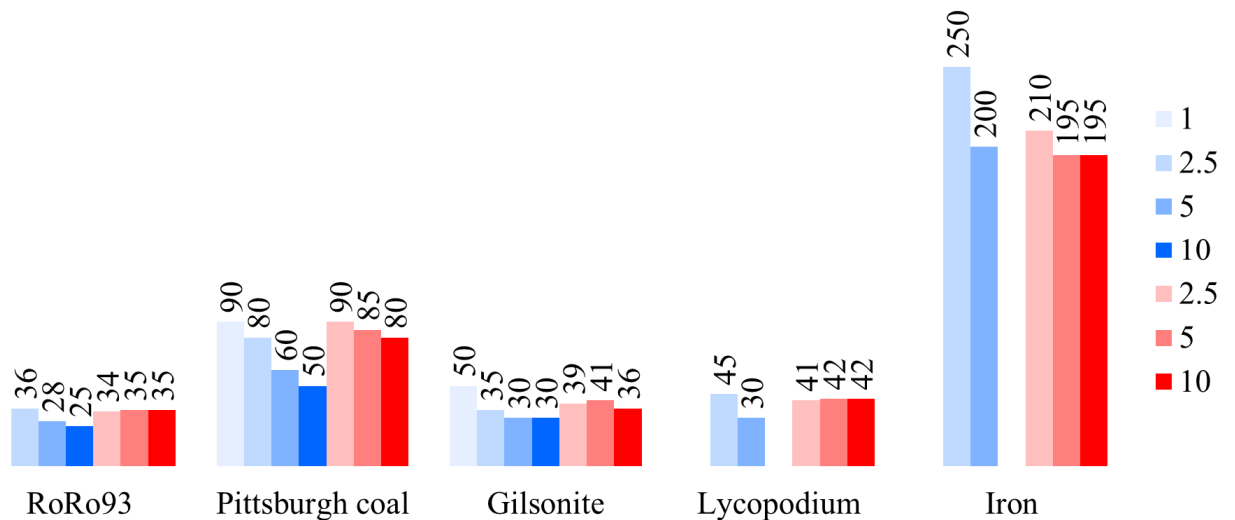


Figure 4. Results of MEC testing (g/m<sup>3</sup>) for five dusts measured in the Fike 20-l sphere (blue) and in the Fike 1-m<sup>3</sup> chamber (red) at varying ignition energies (kJ) (Going et al., 2000)

Similar behavior can be observed with LOC measurements (Figure 5). The experiments in the smaller vessel are sensitive to the igniter energy, the apparent LOC decreasing with increasing ignition energy. Again, this suggests that the igniter energy provides more than just ignition for the initial propagation, affecting the overall mixture explosibility. The authors therefore recommended the 1-m<sup>3</sup> chamber for measuring LOC values below 10%.

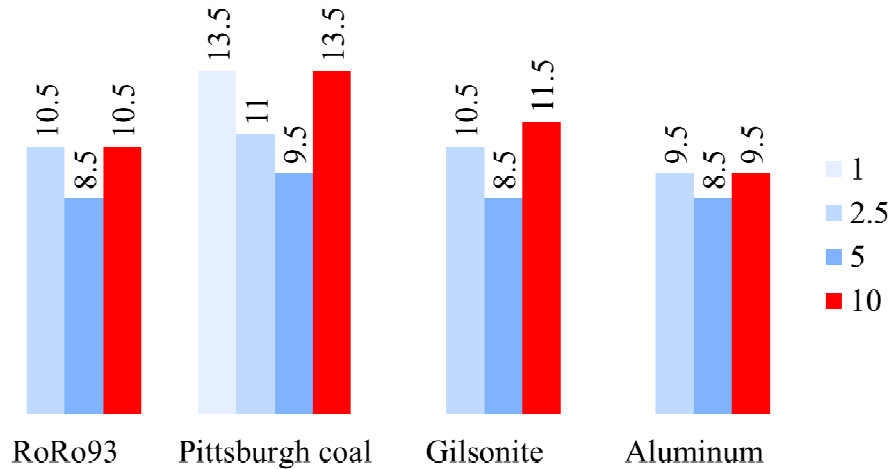


Figure 5. Results of LOC testing (% O<sub>2</sub>) for four dusts measured in the Fike 20-l sphere (blue) and in the Fike 1-m<sup>3</sup> chamber (red) at varying ignition energies (kJ) (Going et al., 2000)

Results of these two testing campaigns show that the best agreement between the 20-l sphere and the 1-m<sup>3</sup> chamber is obtained for an ignition energy of 2.5-kJ. This energy level is currently recommended in ASTM standards related to MEC (E1515) and LOC (E2931) determination (Table 4).

	Bureau of Mines 20-l vessel		Fike 1-m <sup>3</sup> chamber
	2.5-kJ	5-kJ	10-kJ
Bituminous coal, Pocahontas seam	120	85	...
Bituminous coal, Pittsburgh seam	80	60	80
Lycopodium	45	30	42
Gilsonite	35	30	36
Polyethylene	32	28	...

Table 4: Comparison of MECs (g/m<sup>3</sup>) determined in the 20-l US Bureau of Mines vessel and 1-m<sup>3</sup> Fike chamber at varying ignition energies (kJ) (ASTM E1515, 2014)

More recently, Kuai et al. (2013) studied the effect of ignition energy (1, 2, 5 and 10-kJ pyrotechnical igniters) on the maximum explosion pressures and the MECs of sweet potato, magnesium, and bituminous coal dusts in a 20-l sphere. They concluded that MEC is significantly affected by ignition energy (Figure 6) and suggested using different ignition energies, depending on the nature of the dust (5 to 7-kJ for carbonaceous dusts, 2 to 5-kJ for light metals), thus corroborating the earlier conclusions of Going et al.



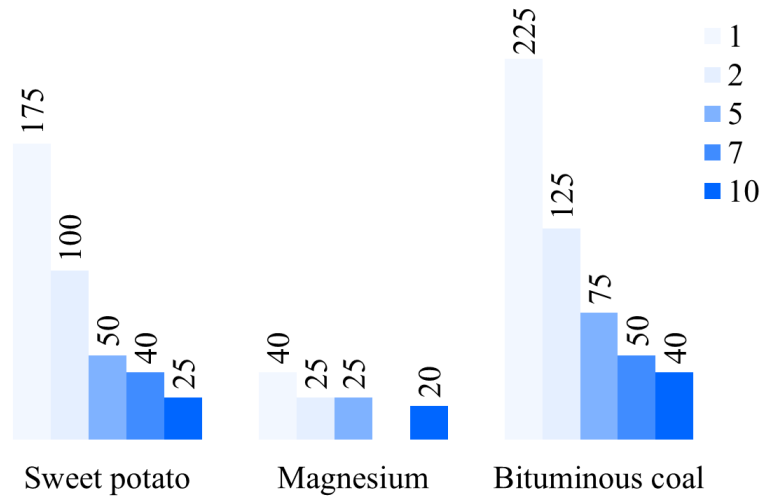


Figure 6: Results of MEC testing ( $\text{g/m}^3$ ) for sweet potato, magnesium and bituminous coal powders measured in the 20-l sphere at varying ignition energies (kJ) (Kuai et al., 2013)

### 2.3 Effect of ignition energy on minimum inerting concentrations

Dastidar and Amyotte (2002) compared minimum inerting concentrations (MIC) measurements in the 20-l sphere to those in the Fike 1- $\text{m}^3$  chamber. Figure 6 shows an example of the different minimum inerting concentrations envelopes obtained for PPC dust and MAP as an inerting agent. The results in the larger chamber show that the maximum reactivity is reached for around  $750 \text{ g/m}^3$ , which requires the largest amount of MAP. For the 20-l sphere, the results approximate those of the larger chamber for the lowest ignition energy, and depart to higher required MAP inerting concentrations for increasing ignition energies. This clearly indicates that the larger energies used for ignition influence the explosibility measure. A similar behavior was also observed for cornstarch dust explosibility, for which sodium bicarbonate is used as an inerting agent (Figure 7).

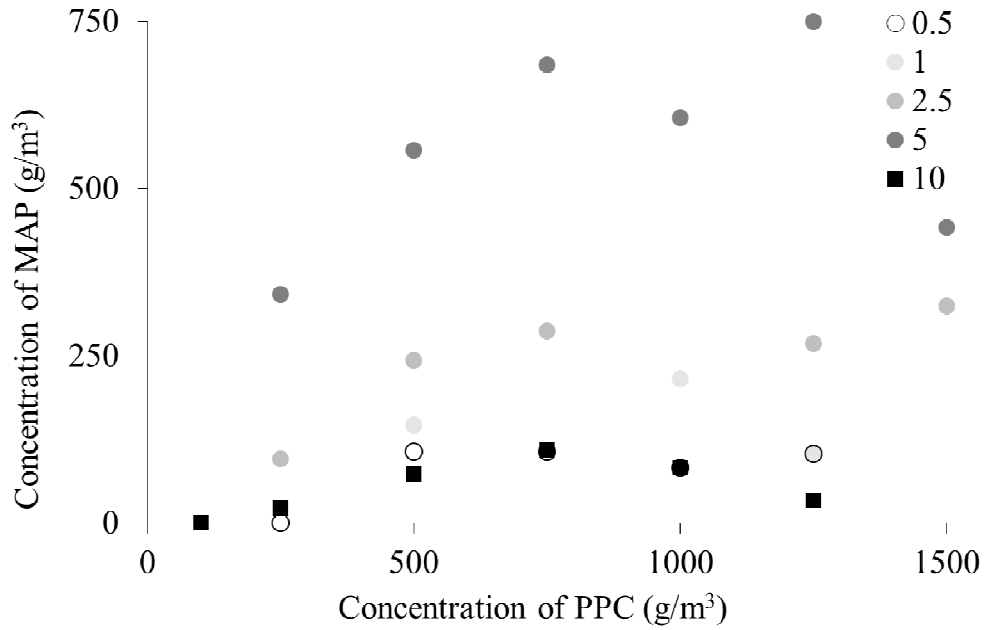


Figure 6: Comparison of 20-l sphere (circles) and Fike 1-m<sup>3</sup> chamber (filled squares) inerting curves at varying ignition energies (kJ) for PPC using MAP as inerting agent (Dastidar and Amyotte, 2002)

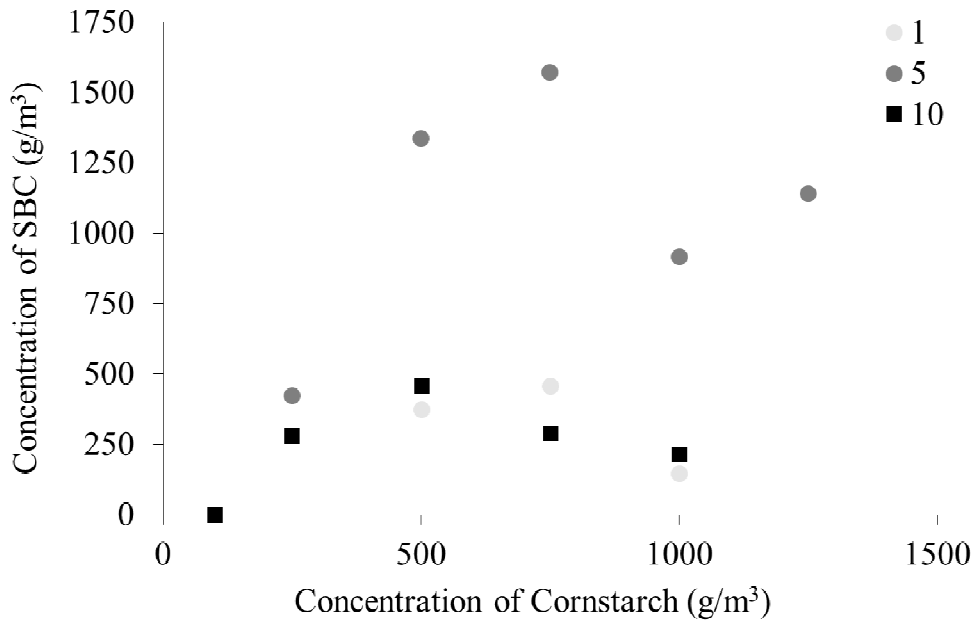


Figure 7: Comparison of 20-l sphere (circles) and Fike 1-m<sup>3</sup> chamber (filled squares) inerting curves at varying ignition energies (kJ) for cornstarch using SBC as inerting agent (Dastidar and Amyotte, 2002)

## 2.4 Conclusions

The investigations summarized in this section show that the high energy delivered by pyrotechnical igniters introduces bias in dust explosibility measurements performed in the 20-l sphere. However, the underlying mechanisms are still not clear.

As an attempt to fill this knowledge gap, the following sections 3 and 4 investigate the effect of pyrotechnical igniters on initial testing conditions and present new experimental evidence about the effect of ignition energy on the deflagration indexes of some organic dusts.

### 3. Anatomy of the effects of pyrotechnical igniters

Dusts can require a significantly higher energy than gases to be ignited: as such, pyrotechnical igniters (made of 40% of zirconium, 30% of barium nitrate and 30% of barium peroxide) used for dust testing provide energies of several kilojoules (Figure 8), while fuse wires employed to ignite gaseous mixtures have energies of only several joules.



Figure 8: Pyrotechnical igniters (Sobbe GmbH)

Figure 9 shows a 5-kJ pyrotechnical igniter fired in an open 20-l sphere. The hot gas and particles fill the entire visible volume, potentially acting like a multiple ignition source.



Figure 9: Visualization of the fireball and hot particles generated by a 5-kJ pyrotechnical igniter in the open Fike 20-l sphere (0.34 m in diameter)

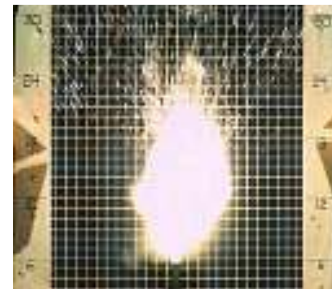
This is in contrast with electrical sparks, or even fuse wires and gel caps shown in Figure 10, which can be qualified as non-invasive ignition sources.



Fuse wire



Gel cap



5-kJ  
pyrotechnical igniter

Figure 10: Visualization of the fireball generated by different ignition sources

For comparison purposes, high-speed recordings displayed on Figure 11 show the progression of the flame induced by 2 x 5-kJ pyrotechnical igniters in the open Fike 1-m<sup>3</sup> chamber.

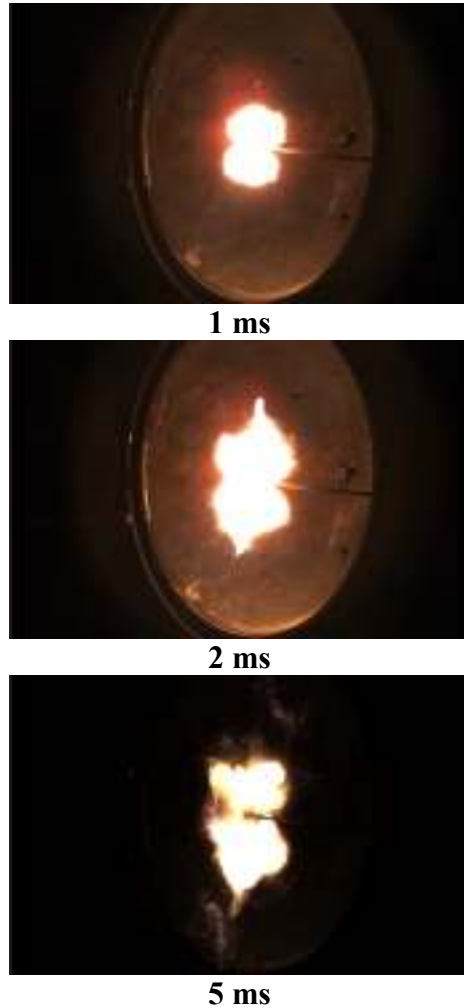


Figure 11: Visualization of the fireball generated by 2 x 5-kJ pyrotechnical igniter in the open Fike 1-m<sup>3</sup> chamber (1.22 m in diameter)

### 3.1 Pressure increase due to pyrotechnical igniters

Figure 12 compares the pressure developed by different pyrotechnical igniters alone as measured by the present authors in a 20-l sphere and 1-m<sup>3</sup> chamber. One single test has been conducted per energy level with the vessel closed and free of any combustible dust.

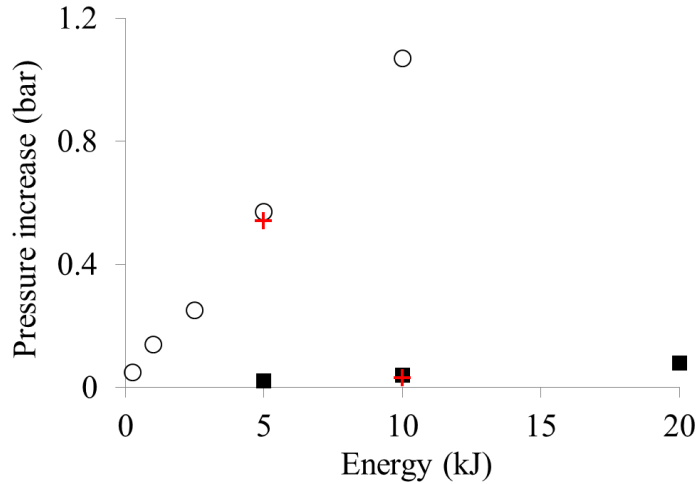


Figure 12: Pressure increase (bar) due to pyrotechnical igniters in a 20-l sphere (open circles) and 1-m<sup>3</sup> chamber (filled squares), and data from Cashdollar and Chatrathi (1992; red crosses)

Clearly the overpressure is negligible in the 1-m<sup>3</sup> chamber, but linearly increases with the total ignition energy up to significant values in the case of the 20-l sphere. Similarly, Cashdollar and Chatrathi (1992) reported a pressure rise of about 0.54 bar for a 5-kJ igniter in the 20-l sphere, compared to 0.03 bar with a 10-kJ igniter in a 1-m<sup>3</sup> chamber, which agrees with the present authors' data.

A 10-kJ pyrotechnical igniter creates, on its own, an overpressure of more than 1 bar. This is known to significantly increase the deflagration index. Dahoe (2000) reports experiments from Wiemann (1987), Bartknecht (1989), and Siwek et al. (1992) showing a proportional relationship between  $K_{St}$  (and also  $P_{max}$ ) and initial pressure (Figure 13).

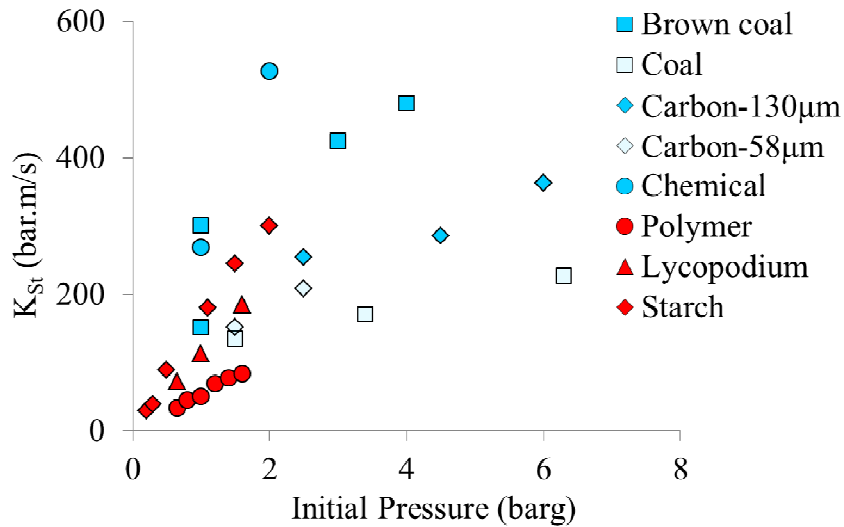


Figure 13:  $K_{St}$  values (bar.m/s) as a function of initial pressure (barg) for different powders according to Bartknecht (1989; in red) and Siwek et al. (1992; in blue)

In a similar fashion, Figure 14 presents the volume-normalized pressure rate of rise created by the pyrotechnical igniters alone (without combustible dust present in the vessel). Again, the effect of pyrotechnical igniters is much more pronounced in the smaller vessel.

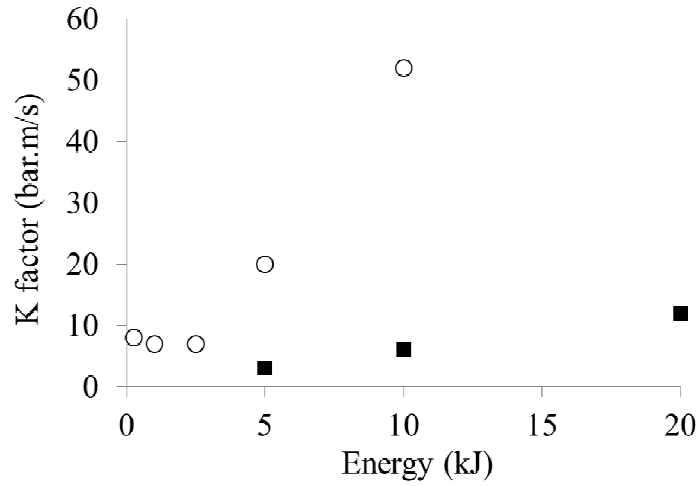


Figure 14: Volume-normalised pressure rate of rise (bar.m/s) due to pyrotechnical igniters in a 20-l sphere (open circles) and 1-m<sup>3</sup> chamber (filled squares)

### 3.2 Temperature increase due to pyrotechnical igniters

The mean overpressure in the 20-l sphere from the energy added by a 10-kJ pyrotechnical igniter being around 1.07 bar, a mean temperature rise greater than 350 K can be expected inside the vessel. However, temperatures may locally greatly exceed this mean value, especially near the center of the vessel (i.e. close to the pyrotechnical igniters).

Scheid et al. (2013) reported high-speed images during the firing of fuse wires and pyrotechnical igniters in an 11-l windowed autoclave. A fast IR camera was used to estimate the maximum temperature and volume in the generated flame/arc. Figure 15 shows four sequences of the flame/arc propagation of exploding wire (top and third row) and pyrotechnical igniter (second and fourth row) recorded with the IR camera. The sequences show a period of approximately 5 ms between images within the first 15 ms after triggering the igniter. The temperature range in the first two rows was adjusted such that temperatures between 200 °C and 650 °C can be observed, while temperatures between 650 °C and 2000 °C can be visualized on rows three and four. The volume of hot gases is clearly much larger in the case of the pyrotechnical igniter compared to the exploding wire, especially shortly after (i.e. 5 ms) after ignition. It can be concluded that temperatures in excess of 650 °C can be reached within a significant volume in the 20-l sphere when using pyrotechnical igniters.

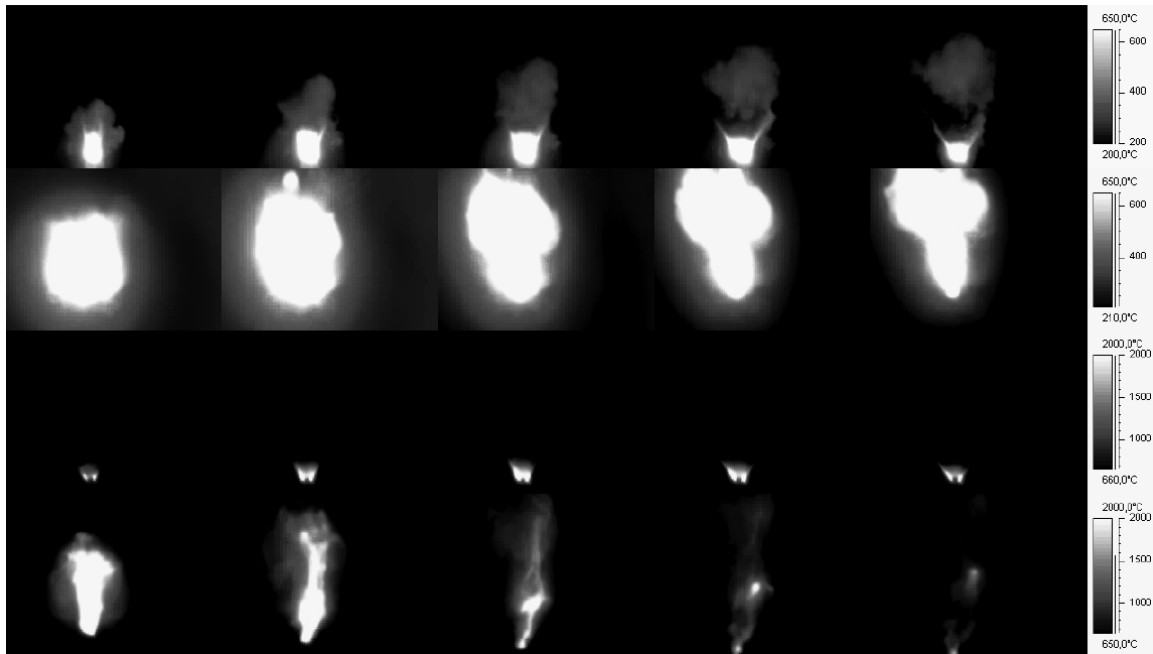


Figure 15: Visualization of the maximum flame/arc volume for an exploding wire (rows 1 and 3) and a pyrotechnical igniter (rows 2 and 4), from (Scheid et al, 2013)

### 3.3 Conclusions

Clearly the strong pyrotechnical igniters used for dust explosibility testing affect the initial testing conditions in the 20-l sphere by increasing both the initial pressure and



temperature inside the vessel. A pressure increase of more than 1 bar and a temperature increase of more than 350 K can be reached when using a 10-kJ pyrotechnical igniter. On the other hand, the effect of pyrotechnical igniters seems negligible in the larger 1-m<sup>3</sup> chamber.

#### 4. Effect of ignition energy on the deflagration indexes of some dusts: new experimental evidence

The previous paragraphs suggest that pyrotechnical igniters can notably affect dust explosibility measurements performed in the 20-l sphere, by increasing both the initial pressure and temperature inside the vessel.

The strong preheating confirmed by Scheid et al. (2013), in particular, may lead to the partial reaction of the dust and the formation of a more reactive hybrid mixture, consisting of a turbulent flammable gas (or vapor) and dust. We propose to call this phenomenon an “igniter-induced hybrid”.

As such, the explosibility parameters  $P_{\max}$  and  $K_{St}$  can be expected to be close to those of a turbulent gas deflagration.

To study this behavior, experiments were performed in Fike’s 20-l sphere and 1-m<sup>3</sup> chamber with four different dusts. The 1-m<sup>3</sup> chamber is spherical, with an internal diameter of 1.22 m and a wall thickness of 9.5 mm (Going et al., 2000), rated to a maximum pressure of 21 bar gauge. The two halves of the sphere are connected by 12 bolts of 51 mm diameter. Two pressure transducers (OMEGA PX459 with > 0.1% accuracy) are used to measure the explosion pressure. Data from the transducers are collected by a 16-bit NI-OCI-6221 DAQ running at 1 kHz. The dust injection system for the 1-m<sup>3</sup> chamber consists of a 5.4-l dispersion reservoir and a standard rebound nozzle. In order to create a dust cloud, a sample of dust weighed by a microscale (OHAUS VALOR 5000 with > 0.1 % accuracy) is placed in the dispersion reservoir. The reservoir is pressurized using dry air to 20 bar absolute and the chamber is partially evacuated. Activation of a ball valve by a solenoid disperses the dust and air into the 1-m<sup>3</sup> chamber through the rebound nozzle and raises the chamber pressure to about 1 bar absolute. Pyrotechnical igniters are fired after a fixed 0.6 s delay after activation of the ball valve. The 20-l spherical vessel used for these experiments, on the other hand, has an internal diameter of approximately 0.34 m, and consists of two halves connected by 8 bolts. The dust injection system of the 20-l sphere consists of a 0.6-l dispersion reservoir and a standard rebound nozzle.

The first experiments were conducted with anthraquinone (C<sub>14</sub>H<sub>8</sub>O<sub>2</sub>). Test results (Figure 16) show a  $K_{St}$  increase of more than 200% when comparing the 20-l sphere to the 1-m<sup>3</sup> chamber. Anthraquinone has reported flash, melting and boiling points of 185, 286, and 380 °C respectively, i.e. all well below the maximum temperature produced by the pyrotechnical igniters. A possible explanation is that the igniter is able to induce sufficient sublimation and vapor formation to change the combustion mechanism scenario from combustion of solid particles (i.e. dust) to combustion of a mixture of particles and a flammable gas (or vapor).

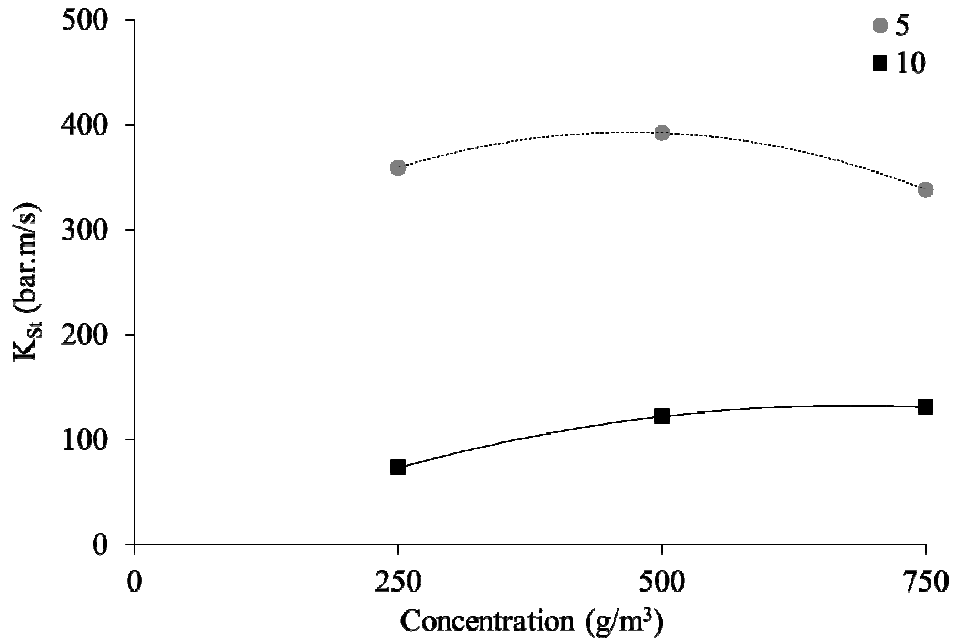


Figure 16:  $K_{St}$  values (bar.m/s) as a function of dust concentration ( $g/m^3$ ) in the 20-l sphere (circles) and the 1- $m^3$  chamber (squares) for anthraquinone using varying ignition energies (kJ)

The same behavior was observed when testing a pigment of low flash point. The  $K_{St}$  in the 20-l sphere was increased by more than 50% at 250  $g/m^3$  (Figure 17).

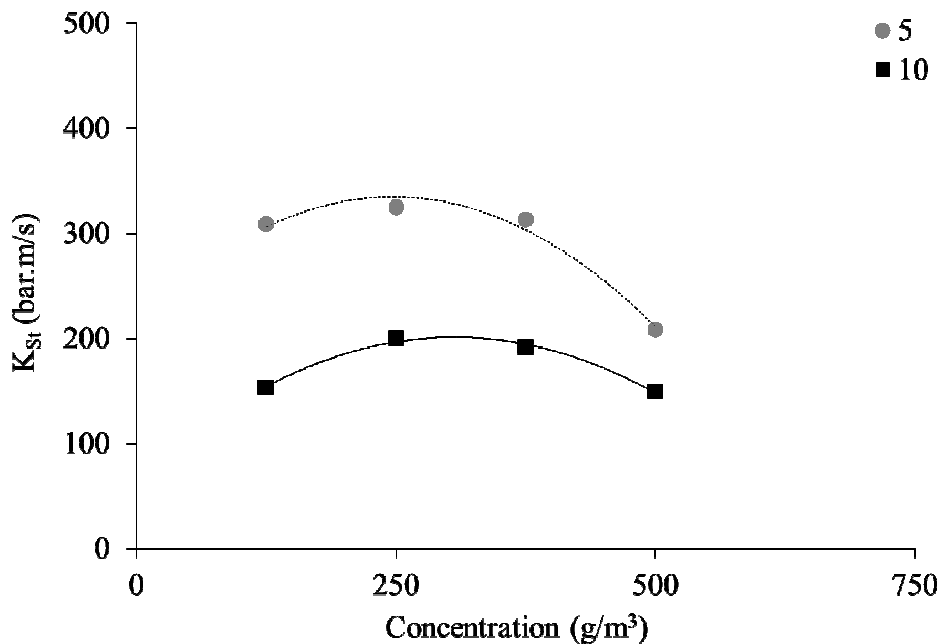


Figure 17:  $K_{St}$  values (bar.m/s) as a function of dust concentration ( $g/m^3$ ) in the 20-l sphere (circles) and the 1- $m^3$  chamber (squares) for a pigment using varying ignition energies (kJ)

To investigate this phenomenon further, additional tests were performed with an oil-encapsulated powder. Given the high  $K_{St}$  values obtained, it is likely that the ignition was sufficient to fracture the microcapsule and vaporize some of the oil. The test results in Figure 18 indicate a  $K_{St}$  increase of 40% at 500  $g/m^3$  when comparing 20-l sphere data to 1- $m^3$  chamber data. It is likely that if a 10-kJ pyrotechnical igniter had been used in the 20-l sphere, the  $K_{St}$  might have been even higher. Results with a lower 1-kJ igniter in the 20-l sphere gave a significant reduction at 250  $g/m^3$ .

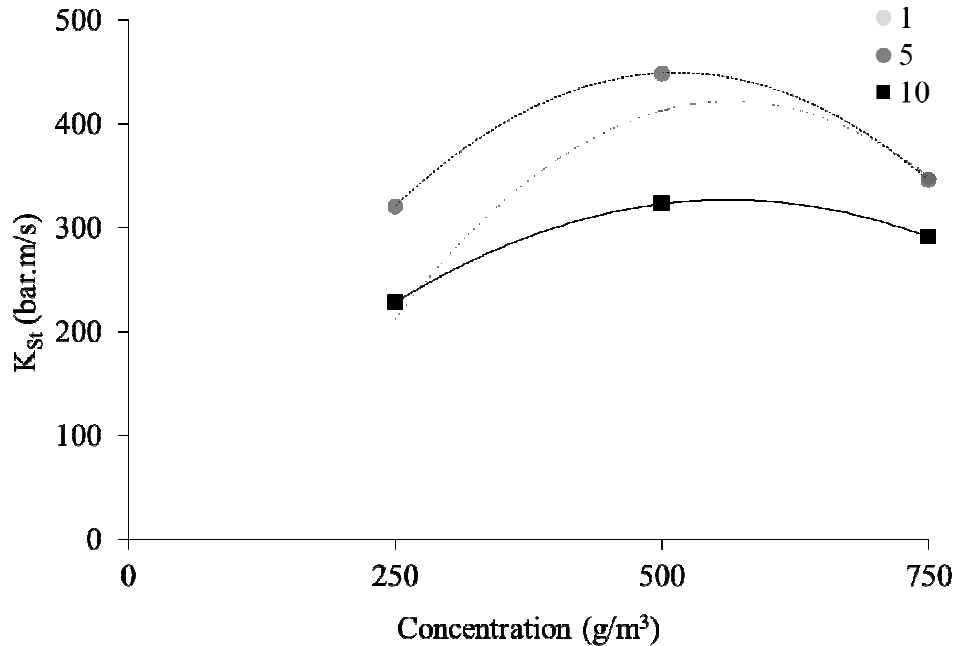


Figure 18:  $K_{St}$  values (bar.m/s) as a function of dust concentration ( $g/m^3$ ) in the 20-l sphere (circles) and the 1- $m^3$  chamber (squares) for an oil-encapsulated powder using varying ignition energies (kJ)

The last tested sample was a wax-coated powder. The wax was reported to have a low melting point, though not established. It is believed that the temperature rise from the 5-kJ igniter was sufficient to vaporize the wax coating, resulting in the formation of a hybrid mixture. In this case, the  $K_{St}$  in the 20-l sphere was increased by more than 100% when using a 5-kJ igniter (Figure 19) at 250  $g/m^3$ . This sample, tested by another laboratory in a 20-l sphere, exhibited a  $K_{St} > 400$  bar.m/s as well.

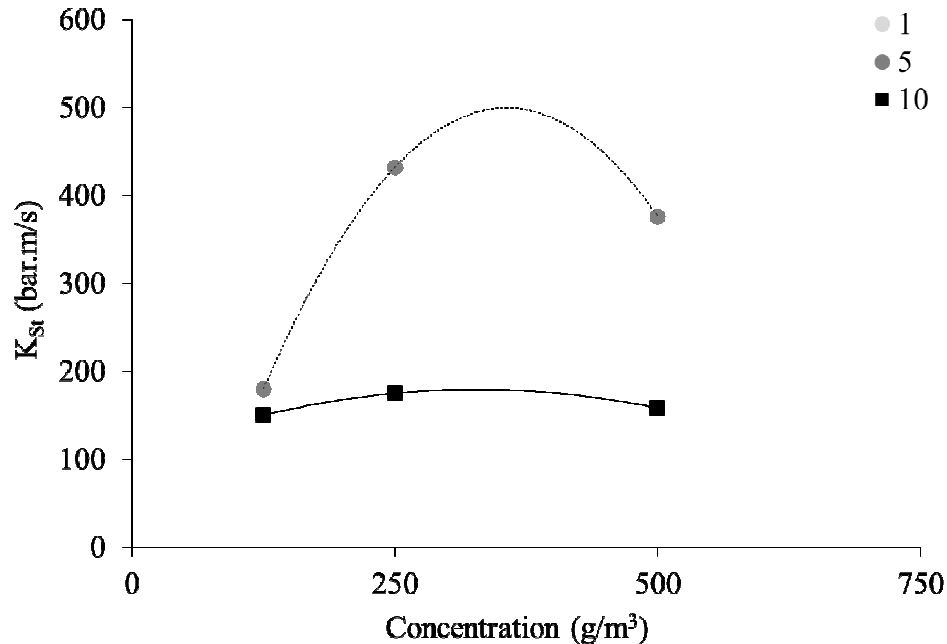


Figure 19:  $K_{St}$  values (bar.m/s) as a function of dust concentration ( $\text{g/m}^3$ ) in the 20-l sphere (circles) and the 1- $\text{m}^3$  chamber (squares) for a wax-coated powder using varying ignition energies (kJ)

Figure 20 reports the maximum  $K_{St}$  (or  $K_{max}$ ) values found for these four dusts in the 20-l sphere and 1- $\text{m}^3$  chamber. Values measured in the 20-l sphere are surprisingly high for dusts, and close to  $K_G$  values reported by Britton and Chippett (1989) for methane and propane, respectively 510 bar.m/s and 635 bar.m/s. These results tend to demonstrate that the experiments carried out in the 20-l sphere with the four dust samples described above actually involved turbulent gas (or vapor)-dust mixtures. These high  $K_{St}$  values found in the 20-l sphere also suggest a dust hazard much more difficult to protect effectively by venting, suppression, or isolation techniques.

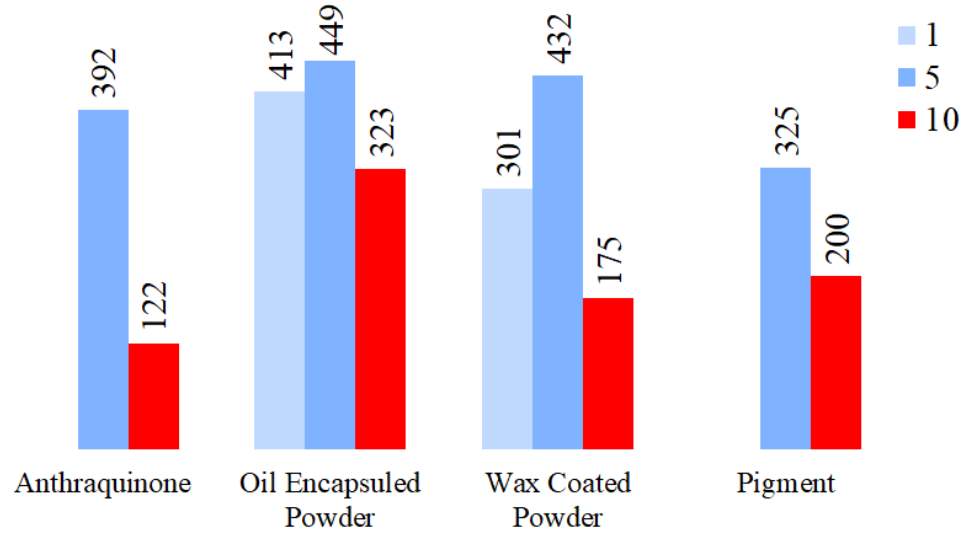


Figure 20:  $K_{St}$  values for the four tested samples in the 20-l sphere (blue) and 1- $m^3$  chamber (red) using igniters of varying energy

## 5. Discussion

The data shown in the previous section clearly suggest that pyrotechnical igniters can have a significant effect on the rate of combustion when used in a small test vessel (e.g. a 20-l sphere). The conjectured mechanism is that the high energies associated with ignition may physically alter the dust sample being tested. Whereas in a large vessel the effect disappears after the initial kernel propagates, in a small vessel these effects persist throughout the flame propagation.

As an example, Table 4 shows the percent of the total energy released by the igniters for the tests involving anthraquinone ( $\Delta H_C = 31 \text{ kJ/kg}$ ) at a concentration of  $500 \text{ g/m}^3$ . Although this is still a small fraction, the energy due to ignition is clearly a couple of orders of magnitude higher in the case of the small vessel. One must remember that the measurements of  $K_{St}$  are acquired at a point where about half of the total energy is released. This means that as much as 5-10 % of the total energy released is due to the original igniter in the case of the small vessel.

	20-l sphere	1-m <sup>3</sup> chamber
Combustion energy (MJ)	0.3	15.5
Ignition energy (kJ)	10	10
Fraction (%)	3.2	0.06

Table 4. Fractional energy due to ignition for anthraquinone in the 20-l sphere and 1-m<sup>3</sup> chamber

Synthetic materials with low flash points, such as polymers or certain chemicals, are believed to quickly generate combustible vapors by sublimation (i.e. a physical process) due to the thermal activation of the pyrotechnical igniters.

This first postulated mechanism shown in Figure 21, describing the combustion of anthraquinone and a pigment, is comparable to droplet combustion.

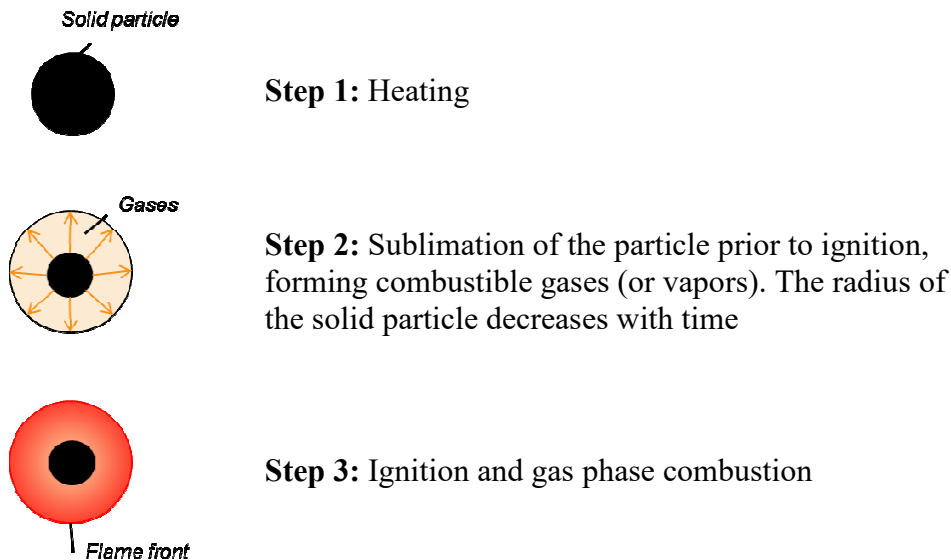


Figure 21. Proposed ignition/combustion mechanism (here for a homogeneous material, e.g. anthraquinone and pigment)

The combustion of the oil-encapsulated powder is illustrated on Figure 22. The oil is released upon heating and break-up of the particles. Liquid droplets are released, probably vaporized and burned around the particle.

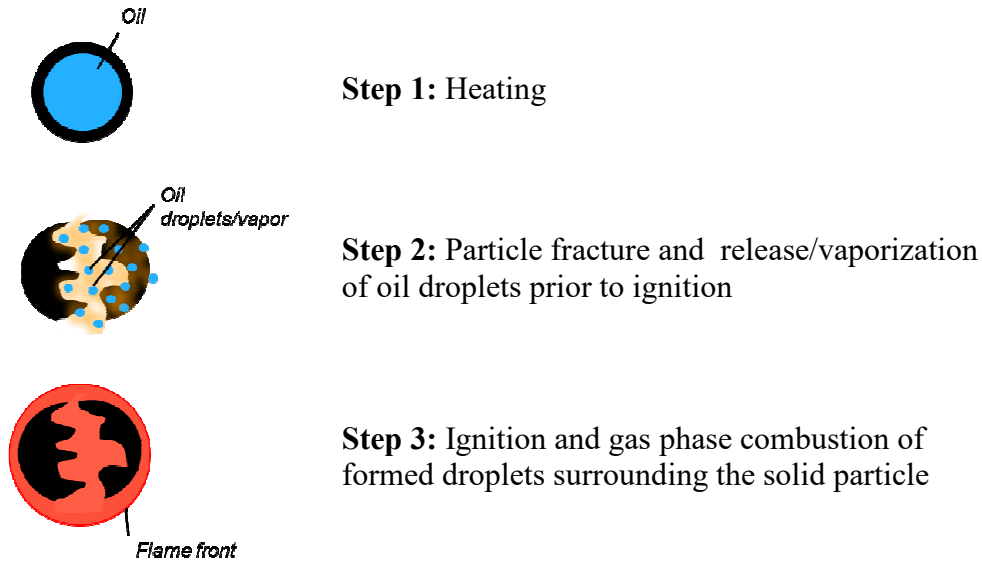


Figure 22. Proposed ignition/combustion mechanism (here for a heterogeneous material, e.g. an oil-encapsulated powder)

In Figure 23 we propose a combustion mechanism for the wax-coated powder: prior to ignition, the wax layer vaporizes, creating combustible vapors that surround the solid particle.

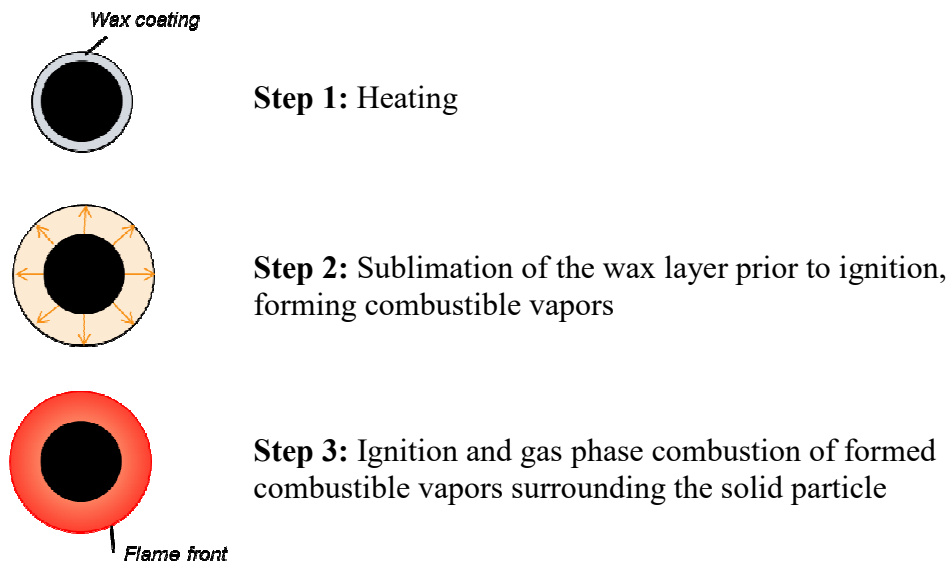


Figure 23. Proposed ignition/combustion mechanism (here for a heterogeneous material, e.g. a wax-coated powder)



## 6. Conclusions

For the first time, experimental evidence is provided supporting the hypothesis that pyrotechnical igniters may, in some instances, physically alter the dust being tested in a 20-l sphere. It is likely that the strong preheating created by the pyrotechnical igniter(s) affects the dust prior to flame arrival, causing partial reaction and the formation of a more reactive hybrid mixture, consisting of a turbulent flammable gas (or vapor) and dust. We proposed to call this phenomenon an “igniter-induced hybrid”.

In our experiments,  $K_{St}$  values obtained in the 20-l sphere were 2 to 4 times greater than those in the 1-m<sup>3</sup> chamber. We therefore believe that the results from the 20-l sphere testing are no longer representative of a dust deflagration in a real process environment.

This behavior depends on the physical properties of the dust. It is expected that the probability of an “igniter-induced hybrid” increases as the flash point and/or the MAIT of the powder in question decreases.

The results provided in the present paper, suggest that when a dust exhibits a low flash point, or when it's  $K_{St}$  is above 300 bar.m/s in the 20-l sphere, the combustion reaction may have been overdriven by the pyrotechnical igniters. In these cases, it is therefore recommended to carry out additional tests in a 1-m<sup>3</sup> chamber, which remains the reference vessel for determining dust explosibility parameters. This recommendation maintains consistency with ASTM E1226 standard.

The consequences of these findings are important, since overestimation of the hazard can result in impractical explosion protection designs, as well as expensive process or equipment modifications. As an example, the high  $K_{St}$  values (> 400 bar.m/s) found in the 20-l sphere for the wax-coated powder suggest a dust hazard much more difficult to protect by either venting, suppression, or isolation, which large-scale testing and industrial experience show not to be true.

## Acknowledgments

The authors gratefully acknowledge the support of Fike Corporation for their permission to publish this work.

## References

- ASTM E1226, 2012. Standard test method for explosibility of dust clouds. ASTM International, West Conshohocken, PA, United States of America.
- ASTM E1515, 2014. Standard test method for Minimum Explosible Concentration of combustible dusts. ASTM International, West Conshohocken, PA, United States of America.
- Bartknecht, W., 1981. Explosions: course, prevention, protection. Springer-Verlag, Berlin.
- Bartknecht, W., 1989. Dust explosions: course, prevention, protection. Springer-Verlag, Berlin.
- Britton, L.G., Chippett, S., 1989. Practical aspects of dust deflagration testing. *Journal of Loss Prevention in the Process Industries*, Volume 2, 161-170.
- Cashdollar, K.L., Chatrathi, K., 1992. Minimum explosible dust concentrations measured in 20-l and 1-m<sup>3</sup> chambers, *Combustion Science and Technology*, Volume 87, 157-171.
- Cashdollar, K.L., 2000. Overview of dust explosibility characteristics. *Journal of Loss Prevention in the Process Industries*, Volume 13, Issues 3-5, 183-199.
- Cloney, C.T., Ripley, R.C., Amyotte, P.R., Khan, F.I., 2013. Quantifying the effect of strong ignition sources on particle preconditioning and distribution in the 20-L chamber. *Journal of Loss Prevention in the Process Industries*, Volume 26, Issue 6, 1574-1582.
- Dahoe, A.E., 2000. Dust explosions - A study of flame propagation. PhD thesis, Delft University of Technology, Delft, The Netherlands.
- Dastidar, A., Amyotte, P., 2002. Determination of minimum inerting concentrations for combustible dusts in a laboratory-scale chamber, *Transactions of IChemE*, Volume 80, Part B, 287-297.
- Gao, W., Zhong, S., Miao, N., Liu, H., 2013. Effect of ignition on the explosion behavior of 1-Octadecanol/air mixtures. *Powder Technology*, Volume 241, 105-114.
- Going, J.E., Chatrathi, K., Cashdollar, K.L., 2000. Flammability limit measurements for dusts in 20-l and 1-m<sup>3</sup> chambers, *Journal of Loss Prevention in the Process Industries*, Volume 13, 209-219.
- Kuai, N., Huang, W., Du, B., Yuan, J., Li, Z., Gan, Y., Tan, J., 2013. Experiment-based investigations on the effect of ignition energy on dust explosion behaviors, *Journal of Loss Prevention in the Process Industries*, Volume 26, 869-877.
- Mintz, K.J., 1995. Problems in experimental measurements of dust explosions. *Journal of Hazardous Materials*, Volume 42, Issue 2, 177-186.
- Proust, C., Accorsi, A., Dupont, L., 2007. Measuring the violence of dust explosions with the “20 l sphere” and with the standard “ISO 1m<sup>3</sup> vessel”. Systematic comparison and analysis of the discrepancies, *Journal of Loss Prevention in the Process Industries*, Volume 20, 599-606.
- Scheid, M., Klippel, A., Tschirschwitz, R., Schröder, V., Zirker, S., Kusche, C., 2013. New ignition source “exploding wire” for the determination of explosion characteristics of combustible dusts in the 20-l sphere. 9<sup>th</sup> Global Congress on Process Safety, American Institute of Chemical Engineers, San Antonio, Texas.

Siwek, R., 1977. 20-l Laborapparatur für die Bestimmung der Explosionskenngrößen brennbarer Stäube. PhD thesis, Technical University of Winterthur, Winterthur, Switzerland.

Siwek, R., Glor, M., Torreggiani, T., 1992. Dust explosion venting at elevated initial pressure. In 7<sup>th</sup> International Symposium on Loss Prevention and Safety Promotion in the Process Industries, Volume 2, pages 57-1-57-15, European Federation of Chemical Engineering (EFCE).

Thomas, J.K., Kirby, D.C., Going, J.E., 2013. Explosibility of a urea dust sample, Process Safety Progress, Volume 32, Issue 2, 189-192.

Wiemann, W., 1987. Influence of temperature and pressure on the explosion characteristics of dust/air and dust/air/inert gas mixtures. In Kenneth L. Cashdollar and Martin Hertzberg, editors, Industrial Dust Explosions, pages 33-44, Philadelphia, American Society of Testing and Materials (ASTM).

Zhen, G., Leuckel, W., Effects of ignitors and turbulence on dust explosions, Journal of Loss Prevention in the Process Industries, Volume 10, Issues 5-6, 317-324, 1997