

EXPLOSION PROPERTIES OF ADMIXTURES[†]

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Many dusts used within an industrial environment are explosible when dispersed in air. However, when such dusts are mixed with an “inert” non-explosible dust, the explosible properties may be suppressed, or at sufficiently high concentrations even eliminated completely. This principle is well known and is used as a means of suppressing dust explosions, explosive gases and vapours.

In certain industrial situations combinations of an explosible dust and an inert dust (admixtures) exist and the explosion behaviour is reduced but not completely suppressed. There is little guidance on this situation, in terms of best practice in industrial plant, or on the details of the best way to approach the laboratory testing. The main issue of concern is the size of the ignition source for such tests and whether the admixture will be over-driven e.g. low concentrations of a combustible dust mixed with an inert dust, may show some explosion behaviour if a sufficiently powerful ignition source is used.

In the standard 20 litre sphere test, a very powerful 10 kJ ignition source is used. Industry is actively investigating the use of smaller ignition energies in conjunction with tests on admixtures using the 20 litre sphere test. The objective is to show that lower ignition energy is more representative of ignition strengths likely to be found on real plant and the resulting explosion characteristics are not as severe.

This paper reports on a research project that explores the situation where admixtures are such that the explosion behaviour is reduced but not completely suppressed. The research shows that the use of a low ignition energy source in the standard 20 litre sphere test results in explosion characteristics that are not as severe as those obtained using the 10 kJ igniter. The results from admixtures of wood dust/calcium carbonate, sulphur/calcium carbonate and zinc stearate/calcium carbonate are presented.

INTRODUCTION

Many dusts used within an industrial environment are explosible when dispersed in air. However, when such dusts are mixed with an “inert” non-explosible dust, the explosible properties may be suppressed, or at sufficiently high concentrations even eliminated completely. This principle is well known and is used as a means of suppressing dust explosions, explosive gases and vapours (BS EN 14373:2005 Explosion suppression systems). Several authors have reported explosion protection by the addition of inert powders such as calcium carbonate, clay or stone dust to combustible dusts to form admixtures that are no longer capable of explosion.

The Centre for Chemical Process Safety (CCPS) book “Guidelines for Safe Handling of Bulk Solids” (2005) reports that although complete inerting with a diluent dust is seldom done in the surface based industries, many particulate solid products contain inert additives as part of their formulation. These additives reduce the risk of explosion. For example, in cement production rock powder has been added to a coal grinding mill under conditions where the mill has to be stopped without emptying the coal from the system. This procedure prevents smouldering of the coal and reduces the risk of an explosion when the mill restarts.

Palmer (1973) has described several solid materials used as inerting powders including sodium carbonate, potassium carbonate, sodium bicarbonate, fullers earth and calcium carbonate (limestone). Palmer points out that the type

of inert powder and degree of fineness will influence the efficiency of the mixture. A well dispersed admixture requires less inert material than a poorly dispersed admixture. Palmer noted that other factors such as particle size and moisture content of the combustible dust and the inert dust influence the dispersability of the mixture.

In a review of stone dusting in mines, Lunn (1995) pointed out that the fineness of the inerting dust is an important aspect of mine safety in that fine limestone dust is more effective at inerting explosions than coarse limestone dust. The use of inert dust to suppress explosions in coal mines is well known. The UK regulations that were drawn up many years ago and specify that inert stone dust is distributed along specific roadways in UK mines to avoid coal dust explosions. The stone dust is normally limestone and is intimately mixed with coal dust deposits so that, in the event of an explosion, the dust lifted into the air ahead of the flame is incombustible. Research has suggested that the proportion of stone dust should increase as coal particle size decreases.

Gummer and Chung (1998), reported on tests in the 20 litre sphere apparatus. The apparatus produces dust explosions by dispersing the dust homogeneously in the enclosed explosion test chamber and applying a strong 10 kJ (2×5 kJ) ignition source. The tests clearly demonstrated trends in the explosibility of coal dust/stone dust mixtures. The results showed that the K_{St} values and maximum explosion

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pressures of coal samples progressively decreased with increasing incombustible content. At 72% incombustible content, a weak explosion was generated in the sphere with K_{St} values of 27–42 $\text{bar} \cdot \text{m} \cdot \text{s}^{-1}$ measured. With 92% incombustible content in the K_{St} values were reduced to 11–17 $\text{bar} \cdot \text{s}^{-1}$. The finer coal sample required a larger quantity of incombustible material to give an equivalent inerting effect on the sample. With regard to limestone inerting, limestone used in UK coal mines is water-proofed to prevent caking when wet. Palmer (1973) has noted that water-proofed limestone is less efficient in suppressing coal dust explosions due to the influence of the coating.

Amyotte *et al.* (2007) has discussed the principle of moderation where the possibility of dust explosion prevention or mitigation is achieved by a range of means including processing an explosive dust in a less hazardous form such as in admixtures with solid inertants or by increasing the explosible dust particle size. An example is given in which tests were done in the 20 litre sphere apparatus on a sample of Datong coal dust. The tests demonstrated that the inerting level was achieved with the addition of >80% dolomite for a coal concentration of 500 g/m^3 . The 5 kJ ignition source used in the tests was less than that used in the standard explosion indices determination where a 10 kJ (2×5 kJ) igniter is specified; BS EN 14034-1 (2004) and BS EN 14034-2 (2006).

The influence of ignition energy on the inerting level has been demonstrated by Dastidar and Amyotte (2002). Corn starch was inerted using sodium bicarbonate in the 20 litre sphere. Decreasing the ignition energy from 5 kJ to 1 kJ in the sphere resulted in a lowering of the minimum inerting concentration (MIC) from 1500 g/m^3 to approximately 500 g/m^3 . The MIC is described as the least amount of inertant required to prevent an explosion regardless of fuel concentration. It is interesting to note that the authors report that inerting data in a larger-scale 1 m^3 test vessel (10 kJ igniter) is approximated by the 20 litre sphere only when the ignition energy in the 20 litre sphere is reduced to 1 kJ. Amyotte indicates that the 1 kJ ignition source would not be expected to overdrive the admixture deflagration and similarly the 10 kJ igniter in the 1 m^3 test vessel would not be expected to overdrive the deflagration. On this basis, the inerting level of the admixture of cornstarch and sodium bicarbonate measured using a 5 kJ igniter in the 20 litre sphere would be unrealistically high if applied to an enclosure volume of 1 m^3 .

Overdriving the deflagration has also been discussed by Sapko *et al.* (2000). The minimum explosible concentration (MEC) values from the 20 litre sphere using a 5 kJ igniter were compared with results from the 1 m^3 chamber using a 10 kJ igniter. The conclusion was that the 5 kJ igniter in the 20 litre sphere overdrives the system for the dusts tested; gilsonite had a MEC of 36 g/m^3 and 30 g/m^3 in the 1 m^3 and 20 litre chambers respectively, and Pittsburgh coal had a MEC 80 g/m^3 and 60 g/m^3 in the 1 m^3 and 20 litre chambers respectively.

Eckhoff (1997) has expressed some concern over the use of mixtures of combustible and inert dust in that there is

the possibility of segregation of the two components during dust dispersion due to the differences in particle properties. Should segregation occur in the region of the ignition source, the admixture may be below or above the assumed nominal average mixture and this could lead to unpredictable combustion.

In certain industrial situations where admixtures exist the explosion behaviour is reduced but not completely suppressed. There is little guidance on this situation, in terms of best practice in industrial plant, or on the details of the best way to approach the laboratory testing. The main issue of concern is the size of the ignition source for such tests and whether the admixture will be over-driven e.g. low concentrations of a combustible dust mixed with an inert dust, may show some explosion behaviour if a sufficiently powerful ignition source is used.

In the standard 20 litre sphere test, a powerful 10 kJ (2×5 kJ) ignition source is normally used in the tests to assess the explosion severity. However, the use of smaller ignition energies may be more representative of ignition strengths found in industry. The objective of the work reported in this paper is to assess the explosion severity of different admixtures in the 20 litre sphere using a range of ignition energies. The project explores the situation where admixtures are such that the explosion behaviour is reduced but not completely suppressed. The results from admixtures of wood dust/calcium carbonate, sulphur/calcium carbonate and zinc stearate/calcium carbonate are presented.

DUSTS

Zinc stearate, sulphur and wood dust were selected as the combustible dusts and represent a range of explosion characteristics. Calcium carbonate was used as the inert component for the admixtures. The materials are presented in Table 1.

TEST PROGRAMME

The admixture samples were prepared for testing by mixing the non-combustible dust and combustible dust in proportions of 70/30, 80/20 and 90/10 (% by weight) using a proprietary tumbling mixing chamber. All the materials were prepared and tested as 100% sub-63micron samples.

Table 1. Test dusts

Dust	HSL Sample Reference	K_{St} ($\text{bar} \cdot \text{m} \cdot \text{s}^{-1}$)	P_{max} (bar)	Moisture content (% w/w)
Zinc Stearate	EC/118/08	293	7.6	1.56
Sulphur	EC/067/09	226	6.2	1.12
Wood dust	EC/074/09	136	7.8	1.44
Calcium carbonate	EC/078/09	0	0	0.32

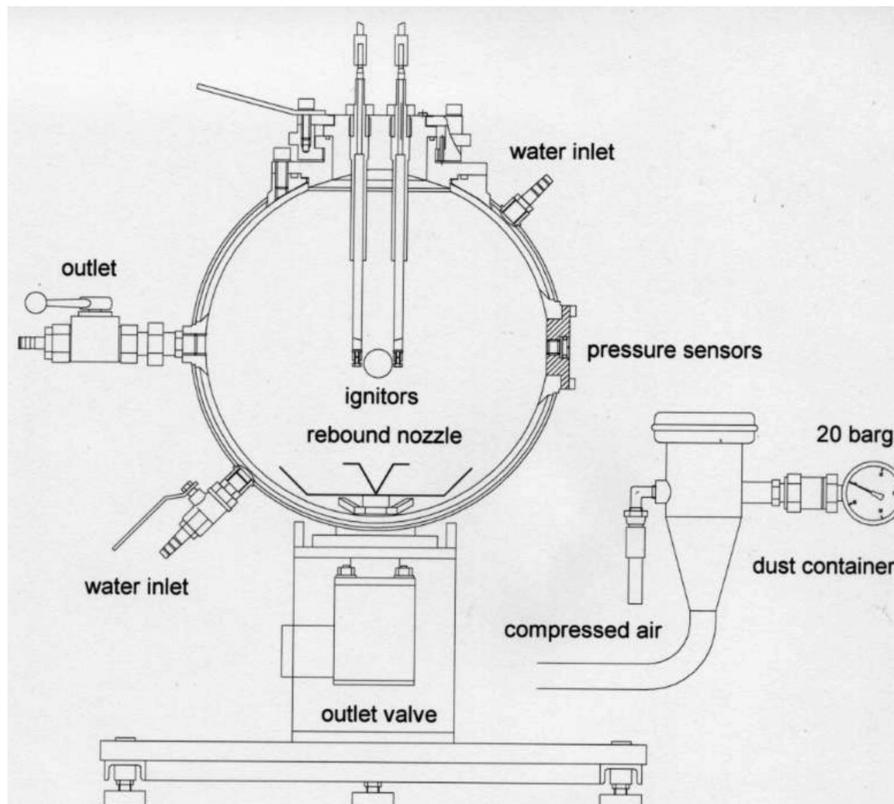


Figure 1. 20 litre sphere apparatus (Used with permission from Kuhner AG)

The explosion characteristics of the admixtures were measured in the 20 litre sphere apparatus (Figure 1).

The maximum rate of pressure rise and the maximum explosion pressure was measured in each test. The apparatus consists of a stainless steel spherical test chamber having an internal volume of 20 litres, a water jacket to dissipate the heat of explosions, an external dust chamber, ignition system and instrumentation/control system. During testing, the dust is dispersed into the sphere from a pressurised storage container via an outlet valve and a rebound nozzle. The increase in pressure caused by injecting the dust is balanced by evacuating the sphere to a known pressure prior to injection. The explosion is thus ignited when the pressure inside the sphere is at 0 bar g.

The peak maximum explosion pressure, P_{max} , and the peak maximum rate of pressure rise, $(dP/dt)_{max}$, are measured in this standard test procedure. The P_{max} and $(dP/dt)_{max}$ are the highest values generated in an enclosed dust explosion. These characteristics are measured in a standard test at the optimum dust concentration and are obtained by testing the dust over a wide range of dust concentrations. The two peaks normally occur at different dust concentrations. The $(dP/dt)_{max}$ is used to calculate a dust specific explosibility characteristic called the K_{St} or K_{max} . The K_{St} is given by:

$$K_{St} = (dP/dt)_{max} V^{1/3}$$

where $(dP/dt)_{max}$ is the peak maximum rate of pressure rise (bar/s) and V is the total internal volume of the test vessel (m^3). The units of K_{St} are $bar \cdot m \cdot s^{-1}$.

The equation is well known as the ‘‘cubic law’’ or ‘‘cube root law’’ (Barton (2002)). The K_{St} is considered to be a constant for any dust, independent of vessel size and the equation acts as a simple scaling law. The K_{St} value is derived only from measurements in either a 20 litre sphere vessel or a $1 m^3$ vessel and if any other vessel is used to measure the K_{St} it must be calibrated against the $1 m^3$ or 20 litre standard test vessels.

For the standard explosion severity test an ignition energy of 10 kJ, comprising two 5 kJ chemical igniters, is normally used, and is located at the centre of the sphere. To explore the effect of different ignition strengths, the ignition energy was varied and energies of either 10 kJ (2×5 kJ), 5 kJ or 2 kJ were used. The igniters were manufactured by Sobbe GmbH.

Two Kistler piezo-electric pressure transducers are used to measure the pressure-time history in the sphere. These are mounted on the side the sphere and are connected to a control unit that is also used to initiate the test. Equipment is calibrated and is traceable to national standards.

All tests were conducted in accordance with the principles of BS EN 14034-1:2004 and BS EN 14034-2:2006, although the ignition energy was varied.

The test conditions were as follows:

Dust-dispersion system	Rebound Nozzle
Ignition Energy	10 kJ, 5 kJ or 2 kJ
Ignition Delay	60 ms
Dust Injection Pressure	20 bar g
Pressure before dispersion	-0.6 bar g
Sphere pressure before ignition	0 bar g
Ambient temperature	20°C ± 5°C

RESULTS

The test results are presented in Tables 2–4.

DISCUSSION

ZINC STEARATE

Zinc stearate was the most reactive of the three combustible dusts tested. With increasing proportions of calcium carbonate there is a significant decrease in the K_{St} value of the dust. For example with a 10 kJ igniter and without the calcium carbonate, zinc stearate had a K_{St} of 293 bar · m · s⁻¹. With 70% calcium carbonate this was reduced to 146 bar · m · s⁻¹; with 80% calcium carbonate this was reduced further to

99 bar · m · s⁻¹ and reduced to 20 bar · m · s⁻¹ with 90% calcium carbonate.

There is also a trend for lower K_{St} values with decreasing ignition energy. This is clearly shown by the considering the results of the 70/30 admixture. At 10 kJ, 5 kJ and 2 kJ, the K_{St} was 146 bar · m · s⁻¹, 136 bar · m · s⁻¹ and 120 bar · m · s⁻¹ respectively. Similarly, a progressive reduction was seen with the 80/20 admixtures; at 10 kJ, 5 kJ and 2 kJ, the K_{St} was 99 bar · m · s⁻¹, 77 bar · m · s⁻¹ and 65 bar · m · s⁻¹ respectively.

Non-ignition of 90/10 admixtures using the 5 kJ igniter suggests that this admixture will be protected from ignition by low level ignition sources in an industrial situation. However, these admixtures will still be potentially ignitable with higher strength ignition sources, demonstrated by the 10 kJ test, but this will result in low explosion violence. The 90/10 admixture with a 2 kJ igniter was not tested.

The P_{max} followed similar trends with a reduction in P_{max} with increasing proportions of calcium carbonate and with progressively lower ignition energies. With an ignition energy of 10 kJ the 100% zinc stearate P_{max} was 7.6 bar and this progressively reduced: with 70/30 admixture the P_{max} was 6.5 bar, with 80/20 admixture the P_{red} was 5.8 bar and at 90%/10% admixture the P_{max} reduced to 2.9 bar.

Table 2. Calcium carbonate/Zinc Stearate

Admixture proportion CaCO ₃ /zinc stearate (%w/w)	Ignition energy [kJ]	P_{max} [barg]	$(dP/dt)_{max}$ [bar/s]	K_{St} [bar · m · s ⁻¹]
70/30	10	6.5	540	146
70/30	5	6.3	502	136
70/30	2	6.1	442	120
80/20	10	5.8	364	99
80/20	5	5.4	282	77
80/20	2	5.3	238	65
90/10	10	2.9	74	20
90/10	5	0.0	0	0
90/10	2	–	–	–

Table 3. Calcium carbonate/Sulphur

Admixture proportion CaCO ₃ /sulphur (%w/w)	Ignition energy [kJ]	P_{max} [barg]	$(dP/dt)_{max}$ [bar/s]	K_{St} [bar · m/s]
70/30	10	3.2	126	34
70/30	5	3.4	87	24
70/30	2	3.6	98	27
80/20	10	2.1	104	28
80/20	5	2.5	66	18
80/20	2	2.7	66	18
90/10	10	0.5	27	7
90/10	5	0.8	13	4
90/10	2	0.7	6	2

Table 4. Calcium carbonate/Wood dust

Admixture proportion CaCO ₃ /wood dust (%w/w)	Ignition energy [kJ]	P _{max} [barg]	(dP/dt) _{max} [bar/s]	K _{St} [bar · m/s]
70/30	10	4.7	59	16
70/30	5	3.5	18	5
70/30	2	0	0	0
80/20	10	0	0	0
80/20	–	–	–	–
80/20	–	–	–	–
90/10	–	–	–	–
90/10	–	–	–	–
90/10	–	–	–	–

SULPHUR

In common with the zinc stearate, the sulphur tests showed reductions in the K_{St} with increased proportions of calcium carbonate. With a 10 kJ igniter there was a very significant reduction in the K_{St} from 226 bar · m · s⁻¹ for 100% sulphur to 34 bar · m · s⁻¹ for a 70/30 admixture. Despite this, the sulphur still remained marginally explosible; at 90/10 the K_{St} was 7 bar · m · s⁻¹ and with a 2 kJ ignition source the test resulted in a K_{St} of 2 bar · m · s⁻¹.

WOOD DUST

Wood dust was the least reactive of the three combustible dusts and with a 10 kJ ignition source the plain wood dust had a K_{St} of 136 bar · m · s⁻¹. The low reactivity was reflected in the significant reduction in K_{St} to 16 bar · m · s⁻¹ with an admixture of 70/30. With the lower ignition energy of 5 kJ the 70/30 admixture resulted in lower reactivity and a K_{St} of just 5 bar · m · s⁻¹ was measured. There was no ignition of the 70/30 admixture with a 2 kJ igniter. Ignition was not possible with the 80/20 admixture using a 10 kJ igniter. The 80/20 admixture with a 5 kJ and 2 kJ igniter and 90/10 admixture were not tested.

CONCLUSIONS

- The test programme has shown that it is possible to reduce explosion severity by the addition of inert dust to a combustible dust. With sufficient inert dust the admixture becomes non-explosible. This technique could be employed by industry in the prevention and mitigation of dust explosions when handling potentially explosive dusts.
- Admixtures containing 70%, 80% and 90% calcium carbonate all produced reductions in explosion severity. The three dusts in this test programme had significant reductions in their K_{St} and P_{max} values with the addition of calcium carbonate. For example, using a 10 kJ igniter, an admixture of 70% calcium carbonate and 30% zinc stearate reduced the K_{St} by 50% to 146 bar · m · s⁻¹,

and sulphur was reduced by 85% to 34 bar · m · s⁻¹, and wood dust reduced by 88% to 16 bar · m · s⁻¹.

- In certain tests the admixtures containing the highest levels of calcium carbonate became non-explosible or were reduced to a very low level of reactivity. For example, in the case of zinc stearate containing 90% calcium carbonate and tested with a 5 kJ igniter, the admixture was non-explosible. The sulphur admixture containing 90% calcium carbonate and tested with a 5 kJ igniter was reduced to a very low level of reactivity with a K_{St} of 4 bar · m · s⁻¹.
- The igniter strength had a significant effect on the rate of pressure rise. Although the 10 kJ igniter ensures that hard to ignite dusts are readily ignited and therefore tested as explosible dusts, the high strength of the igniter may overdrive the explosion and produce a result that indicates greater reactivity than is seen with lower strength igniters. For example, considering the results of the 70% calcium carbonate and 30% zinc stearate admixture, at 10 kJ, 5 kJ and 2 kJ, the K_{St} was 146 bar · m · s⁻¹, 136 bar · m · s⁻¹ and 120 bar · m · s⁻¹ respectively. The effect of ignition strength on P_{max} was less clear. With calcium carbonate/zinc stearate admixtures, the P_{max} decreased with decreasing ignition strength. Whereas with calcium carbonate/sulphur admixtures there was a tendency for an increase in P_{max} with decreasing ignition strength.
- In some cases an admixture can be ignited using a 10 kJ ignition source but cannot be ignited using a lower strength igniter. For example, the admixture containing 10% zinc stearate ignited using a 10 kJ igniter but would not ignite using a 2 kJ igniter. Further testing in a larger-scale vessel is needed to check whether or not the 10 kJ igniter overdrives the ignition compared with the lower strength ignition energies. This would help to confirm whether or not the test results can be scaled to larger industrial plant.

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