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PARTIAL INERTING OF FLAMMABLE DUST-AIR MIXTURES

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1. INTRODUCTION

Inert gas purging or inerting of flammable dust atmospheres is a well-known safety measure. Chilworth Technology have recently completed a European Commission funded research project into ignition prevention in partially inerted dust-air atmospheres. The research was carried out in association with TNO Prins Maurits Laboratory in the Netherlands. This paper presents some of the results obtained and discusses the possible application of the data.

2. BACKGROUND

Inerting is frequently used to prevent dust explosions. Provided the oxygen concentration in the atmosphere is below the limiting oxygen concentration (LOC), taking pressure and temperature effects into account, no explosion can be initiated. Inerting is often the preferred measure when ignition sources are expected with a frequency that would be intolerable. It is even used in protected plant when the process interruption for inspection, removal of burned and/or contaminated material, recommissioning of explosion vents or suppression systems etc. would preclude economic operation of the plant.

There are, however, other reasons why inerting is chosen over other measures. Sometimes the process takes place in an inherently (partially) inert atmosphere, e.g. in controlled oxidation processes. In other cases, quality considerations justify the use of inert gas. Occasionally inert gas is available at the plant and its application just seems to be an easy answer to any dust explosion hazards. In these cases, all that is apparently required is to determine the maximum allowable oxygen concentration and install monitoring equipment.

In all cases inerting involves the expense of the nitrogen consumption and brings asphyxiation hazards. The nitrogen consumption could be reduced and the asphyxiation hazards minimized if it were possible to accept slightly reduced oxygen concentrations (say 16 - 20 %v/v) instead of the fully inert concentrations (typically 8 - 12 % v/v).

One possible reason for accepting partially inert conditions would be if these could reduce the ignition sensitivity of the dust. This refers to the ongoing debate in safety engineering whether "avoidance of ignition sources" would suffice as sole explosion protection measure; the argument being that if the ignition sensitivity is low, all potential ignition sources can be eliminated.

It is well known that both the explosion severity and the ignition sensitivity are reduced with decreasing oxygen concentration. The fact that the value of the dust explosion constant K_{st} reduces approximately linearly between 21%v/v O_2 and the LOC, is often used in testing to estimate the likely value of the LOC and so reduce the effort involved. The maximum explosion overpressure (P_{max}) on the other hand is only reduced significantly when the LOC is approached.

The increase in minimum ignition energy (MIE) as the oxygen concentration is decreased is also well documented: the LOC can be interpreted as the oxygen concentration at which the MIE is equal to the igniter energy employed (typically 10 kJ).

3. EXPERIMENTAL PROGRAM

The research project discussed here considers various ignition mechanisms at two or three levels of oxygen concentration between 14 and 21 %v/v. A more detailed variation of the oxygen concentration was outside the scope of the project.

The ignition mechanisms considered were:

- thermal stability of dust deposits
- propagation of burning in dust layers
- hot plate ignition of a 5 mm thick dust layer
- hot surface ignition of a dust cloud
- ignition by electric arcs
- ignition by electrostatic sparks

For most of the tests, a range of 10 dusts of various sources was used. Since none of the original samples were able to propagate a fire in a dust deposit, four other samples were selected for those tests.

4. THERMAL STABILITY OF DUST DEPOSITS

Dust deposits or bulk material can be ignited by exothermic behaviour of the material under their storage or drying conditions. Once ignited, the burning material can act as an ignition source for a dust explosion.

The thermal stability was investigated using three tests as described in the "Institution of Chemical Engineers Drying Guide" Ref.1. The tests were:

- Air-Over-Layer, simulating conditions in dryers such as cross-flow, band and tray dryers and deposits in all types of dryers, in which hot air circulates above a layer of material. In the test the layer measures 75 mm x 40 mm x 15 mm deep.
- Aerated Cell, simulating dryers in which a hot air stream passes through the material.
- Bulk Powder (Diffusion) Cell, simulating conditions in silos or bags or at the bottom of dryers where material can collect in bulk.

All tests were performed as screening tests, i.e. the temperature in the oven was increased at a rate of 0.5 °C/min from ambient to 400 °C (or the melting point of the material if lower). Such screening tests allow a first estimate of the onset temperature of the exotherm, the temperatures at which the rate of temperature increase exceeds prescribed values, the temperature difference between sample and oven and the duration of the exotherm. However, screening tests are not normally sufficient to prescribe safe drying conditions.

Table 1 presents some of the results obtained in the Air-Over-Layer tests. The table shows that the onset temperatures were only slightly increased by the reduced oxygen concentration. Peak temperatures and the difference between peak and oven temperature generally decreased as well as the maximum rate of temperature increase. Some samples, however, showed a different behaviour. Most notably, sulphur showed a much more active exotherm at 14 % than at 21 % O2, but the duration was only 17 minutes compared to 80 minutes at 21 % O2.

The Aerated Cell and Bulk Powder (Diffusion) Cell tests showed in general similar trends. In detail, however, the various materials responded differently to the change in conditions. This is in agreement with the general observation at normal oxygen levels that no single test can predict the exothermic behaviour in all conditions. It is worth noting that in some cases the exotherm started earlier at low oxygen concentrations than at higher levels!

The results of the investigations indicate that there may be some (limited) room for increasing drying temperatures and hence reduce drying times in partially inerted conditions. Because of the variability in results, however, further testing is required to validate this approach for specific cases.

Table 1: Onset temperatures as determined from the Air-Over-Layer screening test.

Test Powder Name	Onset Temperature (°C)		
	21 % O_2	17 % O_2	14 % O_2
Citrus Peel	167	183	
Lycopodium	167	169	
Milk Powder	150	161	128
Potato Starch	273		312
Polyolefin	210		249
Sulphur	195		205
Wood Dust	204	219	
Sugar	No Exotherm		
Bisphenol A	No Exotherm		
Activated Carbon	235		275

5. PROPAGATION OF BURNING IN DUST LAYERS

Propagation of a fire or smouldering combustion in a dust layer can introduce ignition sources in a flammable dust-air mixture. Alternatively, a burning dust layer can be dispersed into a dust cloud incorporating an ignition source.

The propagation of burning in dust layers was investigated using the "Fire Train" test as used in transport classification and in the assessment of physico-chemical properties of materials Ref. 2.

In the test, a 0.25 m long strip (10 mm high, 20 mm wide) of dust is made and ignited at one end. The time to propagate the reaction over 100 mm length is measured. The shortest time out of 6 repeat tests is reported. A dust is "highly flammable" when the time is less than 45 seconds. Tests at elevated temperatures were carried out by placing the dust on a hot plate and let the temperature stabilise.

Since none of the samples listed in table 1 "trained fire" at all, four new samples were selected: borneol, paraformaldehyde, metaldehyde and hexamine. It was found that at 17 % O_2 all four failed to train fire.

Next, the temperature was increased to 100 °C (50 °C for borneol since it degraded at 100 °C) and the tests were repeated. It was found that the burning rate decreased with decreasing oxygen concentration, but that the decrease was very small. All except hexamine would still be classified as "highly flammable" at the elevated temperatures, even at 14 %v/v O_2.

It must therefore be concluded that, for the samples investigated, the effect of reducing the O_2 concentration at ambient temperature is dramatic (halting the reaction), but very limited at elevated temperatures.

6. HOT PLATE IGNITION OF A 5 mm THICK DUST LAYER

The hazards associated with the ignition of a dust layer on a hot surface, such as electrical equipment or steam pipes, are similar to those of burning deposits ignited by other sources; see section 5.

The hot plate ignition was investigated by determining the minimum ignition temperature for a 5 mm dust layer (or "smouldering temperature") in agreement with a draft IEC document Ref. 3, which is essentially the same as a recent IEC standard Ref. 4.

In the test a 5 mm thick dust layer, diameter 100 mm, is made on a hot plate. Initially a screening test is performed by increasing the hot plate temperature slowly from ambient to 400 °C. Isothermal tests are used to find the limit temperature where no exotherm is detected. For the purposes of the tests, an ignition is assumed when a visible glowing, burning or an increase of the sample temperature to at least 20 °C above the plate temperature is detected.

In the screening tests, it was found that only four of the samples displayed exothermic activity at ambient conditions. Tests at reduced oxygen concentrations were only performed for those samples. The results are summarised in table 2.

Table 2: Layer ignition temperatures for 5 mm layer thickness.

Test Powder Name	Layer Ignition Temperature(°C)		
	21 % O_2	17 % O_2	14 % O_2
Citrus Peel	280	290	290
Lycopodium	240	240	240
Sulphur	270	270	270
Wood Dust	310	340	350

The table shows that for 2 samples there is no influence of the oxygen concentration on the layer ignition temperature. For the other two, a small increase was noted when the oxygen concentration was reduced.

The practical implication is that the maximum surface temperatures (where deposits can be present) that can be allowed in a plant are hardly influenced by partial inerting.

7. HOT SURFACE IGNITION OF A DUST CLOUD

The ignition of a flammable dust cloud by a hot surface will initiate an explosion immediately and must therefore be avoided. Whether a dust cloud ignites at a hot surface depends not only on the flammable material and the surface temperature, but also on parameters such as the residence time of the dust cloud near the surface, geometry of the system etc..

In accordance with common practice, the minimum ignition temperature of the dust cloud as used for specification of electrical equipment was used. The Godbert-Greenwald furnace Ref. 3,4 was modified to allow a variation of the hot surface area: inside the normal 36 mm diameter, 400 mm long tube another tube (I.D. 23 mm) was inserted consisting of two isolating parts with a 100 or a 200 mm long metal section in between. Apart from changing the surface area, also the residence time of the dust cloud is increased.

The results for the 200 mm long tube are presented in table 3. Results for the shorter tube were only slightly different and the observed trend was similar.

Table 3: Minimum ignition temperature of a dust cloud as determined in the modified Godbert-Greenwald furnace (tube length 200 mm).

Test Powder Name	Minimum Ignition Temperature (°C)	
	21 % O_2	17 % O_2
Citrus Peel	340	380
Lycopodium	340	380
Milk Powder	360	420
Potato Starch	360	380
Polyolefin	400	420
Sulphur	180	180
Wood Dust	380	400
Sugar	300	320
Bisphenol A	480	590
Activated Carbon	520	540

Table 3 shows that there is an effect of the oxygen concentration on the minimum ignition temperature, but it is not large. On average, a decrease of the oxygen concentration of 4 %v/v gave rise to an increase in minimum ignition temperature of about 10 %.

In view of the limited effect observed, the variation of the minimum ignition temperature as a function of oxygen concentration was investigated over a wider range for two dusts. The results are presented in table 4. It is clear that ignitions occurred at oxygen concentrations far below the LOC, making the concept of LOC invalid for ignitions of dust clouds by hot surfaces. These ignitions were observed to be very slow burning of the dust cloud, resulting in almost invisible red light. As in these conditions the whole dust cloud is raised in temperature, causing a more or less uniform auto-ignition, it cannot be certain that the flame would be able to propagate. In fact, as long as the initial fireball created by the hot surface ignition is no greater than that created by the chemical igniters used in the LOC determination, it is likely that propagation in the "cold" part of the dust cloud would not be possible. Therefore, oxygen concentrations below the LOC may be sufficient to prevent a dust cloud explosion, but not necessarily a dust cloud ignition.

The conclusion from this work is that there is a limited possibility to allow higher surface temperatures (where only cloud ignition is expected) by using partial inerting, as long as the surface temperatures are known and stable. The increase in the minimum ignition temperature, however, is too limited to expect any protection against ignition by hot surfaces that are caused by malfunction, since the temperatures tend to increase rapidly to a high value in such cases.

Table 4: Minimum ignition temperature of a dust cloud for two samples as determined in the modified Godbert-Greenwald furnace (tube length 200 mm).

Oxygen Concentration (%v/v)	Minimum Ignition Temperature (°C)	
	Sugar	Lycopodium
21	300	340
17	320	380
15	400	440
13	400	480
11	-	510
9	450	530
6	-	570
4.5	480	-

8. IGNITION BY ELECTRIC ARCS

Electric arcs arise for example during failure of electric circuits. It is known that a continuous arc has a very high ignition energy and it is often used to screen dusts for flammability. Although its power is recognised, it seemed worthwhile to investigate if this ignition source could be tempered by partial inerting.

The tests were performed by generating a continuous arc from a high-voltage transformer between the electrodes in an open version of the Hartmann tube (with the top closed by a bursting disc). Normally the transformer would be operated with an input voltage of 220 V, providing a 10 kV, 0.02 mA output. As a variation also a "weak arc" was generated by reducing the input voltage to about 110 V, which was possible without creating problems with regard to arc generation.

The dust concentration was varied (500, 750 and 1000 g/m3) and each condition was tested 3 times if no ignition (defined as a self propagating flame over at least 10 cm) occurred.

Almost all dusts ignited with both the strong and the weak arc in 21 and in 17 %v/v O_2. The two exceptions were Activated Carbon that could not be ignited in any of these conditions, and potato starch. Potato starch did ignite with the weak arc at 21 % but not at 17 %v/v O_2. After increasing the input voltage to 160 V, potato starch did ignite at 17 %v/v oxygen.

The potato starch results indicate that there is an influence of the oxygen concentration on the ignition by continuous electric arcs, but the results are too limited to draw any general conclusions.

9. SPARK IGNITION OF A DUST CLOUD

The direct ignition of a dust cloud by an electric or electrostatic spark is a well-known phenomenon. It is common to use the minimum ignition energy, i.e. the smallest spark energy that is just capable of igniting the optimum dust cloud, as one of the main parameters to indicate the ignition sensitivity of the dust.

There are two ways to approach the minimum ignition energy:

- By using a purely capacitive spark the best simulation of electrostatic discharges is obtained and the test results are directly comparable to discharge energies occurring in practice. This approach is favoured in e.g. the BS method Ref. 5.
- By inserting an inductance in the discharge circuit, lower values are obtained in most cases which gives a "conservative" measure of the ignition sensitivity. This approach is used in the VDI method Ref. 6.

Both methods were used in the tests. The tests were performed in a Hartmann tube as in section 8. The dust concentration was varied (500, 750 and 1000 g/m3, based on earlier research at TNO that most dust ignite most easily around 750 g/m3). The ignition delay time (between dispersion of the dust and spark) was chosen as 200 and 300 ms, again on the basis of the experience at TNO. The highest spark energy that failed to ignite the dust was tested 3 times at each condition, yielding a total of 18 no-ignitions. The procedure results in an energy band between the highest no-ignition and the lowest ignition. The range of energies available in the tests was between 3.2 and 8670 mJ.

Table 5 presents the results for the capacitive sparks (BS method). The inductive sparks (VDI method) resulted in similar observations. When the 3.2 mJ energy level produced an ignition, the results is given as "< 3.2". Similarly if 8670 mJ failed to ignite, the table reads "> 8670".

Table 5: Minimum ignition energy for capacitive sparks, determined in a Hartmann tube.

Test Powder Name	Minimum Ignition Energy (mJ)	
	21 % O_2	17 % O_2
Citrus Peel	> 8670	> 8670
Lycopodium	49-89	385-891
Milk Powder	385-891	2145-3810
Potato Starch	3810-8670	> 8670
Polyolefin	891-2145	3810-8670
Sulphur	< 3.2	26-49
Wood Dust	385-891	> 8670

Sugar	385-891	891-2145
Bisphenol A	< 3.2	49-89
Activated Carbon	> 8670	> 8670

The table shows that the minimum ignition energy rises sharply with decreasing oxygen concentration. This means that it is possible to reduce the sensitivity of a dust by partial inerting to a level where specified electrostatic discharges are no longer able to ignite the dust cloud. For example, it is commonly assumed that brush discharges must be considered when a dust is highly sensitive, e.g. minimum ignition energy less than 3 or 4 mJ. Table 5 shows for Sulphur and Bisphenol A that partial inerting to 17 %v/v O_2 will suffice to raise the minimum ignition energy well above that level. If no other ignition sources can be identified, it could be argued that no further inerting is necessary for these dusts.

10. GENERAL CONCLUSIONS

Overall the following conclusions can be drawn from this research project:

- The minimum ignition energy for spark ignition is increased dramatically by partial inerting, which makes partial inerting very effective in reducing the likelihood of a spark ignition.
- The onset temperatures for thermal stability were only slightly increased in most of the tests, but the peak temperatures and the duration of the exotherm ware generally reduced. This shows that the main benefit of partial inerting might be a reduction of the consequences of thermal instability, rather than prevent it from starting.
- For all parameters except the minimum ignition energy, any effect of partial inerting was very dependent on the material under investigation and some materials did not follow the general trend. This means that specific data need to be obtained for a specific material if partial inerting is to be used.
- It appears as if slow ignition processes such as ignition of dust layers and bulk material are less influenced than the faster processes such as dust cloud ignition. This may be due to the fact that oxygen can diffuse to the reaction zone in slow processes, but not in fast processes.
- The fact that the minimum spark ignition energy is so much more influenced than the other dust cloud ignition parameters (minimum ignition temperature, ignition by continuous arc), could be attributed to the transient nature of the spark: the reduced reaction rate caused by partial inerting will be less effective in counteracting the heat losses in the initial ignition kernel; in the other cases the persisting arc will assist the ignition process till the kernel reaches the critical size or the more or less homogeneous temperature field will reduce the heat losses.
- The practical implications of this research are that partial inerting can be a promising technique, making inerting simpler and more cost-effective, but that detailed designs can only be based on specific testing following a thorough assessment of the likely incident scenarios in a particular plant.

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