How accurately can we predict the LOC of a dust cloud?

Evaluating the reliability of R Siwek's 1996 calculation method for estimating a material's Limited Oxygen Concentration (LOC).

Abstract

In situations where dust explosion risks are prevented by working at low oxygen atmospheres as the basis of safety, the limiting oxygen for combustion (LOC) value for the dust is the key parameter to establish a safe oxygen level. The LOC can be determined experimentally, but can also, allegedly, be estimated based on other dust explosion properties. The purpose of this study is to determine whether the calculation method of determining a material's Limiting Oxygen Concentration (LOC), as per the method proposed by R Siwek, 1996 is reliable enough to be used in explosion safety assessments, instead of determining the LOC by experimental method. The calculation method was set out in "Determination of technical safety indices and factors influencing hazard evaluation of dusts, R Siwek", 1996. The calculation uses the Minimum Ignition Energy (MIE) and Minimum Ignition Temperature (MIT) of the dust to calculate the LOC of the dust cloud. The findings of this study deem this calculation method inadequate.

Introduction

The LOC of a dust as defined in "Determination of explosion characteristics of dust clouds – Part 4; BS EN 14034-4:2004", is the maximum concentration of oxygen in a mixture of dust, air and an inerting gas, at which dust explosions cannot occur. The purpose of this data is for the proper application of inerting systems, which serve to avoid the formation of flammable atmospheres. The LOC is normally determined experimentally, by diluting air with a known inert gas (such as N2, CO2, or Ar) within an explosion vessel. The fuel, air and inert gases are dispersed within the reduced oxygen atmosphere at defined oxygen concentrations, and subjected to a high-energy pyrotechnic ignitor. Ignition is determined through the measurement of overpressure from a dust-cloud explosion. In this study, all data derived through testing used pure nitrogen used as the inert gas (the most common inerting gas used in industry).

However, there are also other means to derive the LOC of a dust, by way of calculation. The calculation of the LOC investigated, as detailed R Siwek's Determination of technical safety indicies and factors influencing hazard evaluation of dusts, uses the material's Minimum Ignition Energy (MIE), and Minimum Ignition Temperature (MIT) to determine the dusts' LOC, as outlined in the following equation:

$$LOC_{Calc} = 1.62 \log\left[MIE\left(1 + \frac{MIT}{273}\right)\right] + 12.9 \qquad [1]$$

Method

To determine the adequacy of the calculation, previously obtained results were found on materials that underwent all relevant testing in the DEKRA UK Explosions Hazards Laboratory. The MIE and (BAM corrected) MIT results were used to calculate the expected LOC from the equation [1] and compared to the experimentally measured LOC of the material.

The MIE as defined in "Potentially explosive atmospheres – Explosion prevention and protection – Determination of minimum ignition energy of dust/air mixtures; BS EN ISO/IEC 80079-20-2:2016" is the lowest electrical energy stored in a capacitor which upon discharge is sufficient to affect ignition of the most sensitive dust/air mixture under specific test conditions . By varying the level of inductance, the test can simulate either "electrostatic" or "mechanical" spark discharges. Electrostatic MIE tests utilise a discharge unit of ≤ 25 μH of inductance, whereas for a mechanical spark assessment, the discharge unit must have 1 – 2 mH. Because there is a greater level of inductance, mechanical MIEs are often referred to as inductive, whereas electrostatic MIEs are known as capacitive . The LOC calculation used requires an inductive discharge, so all references to MIE values in this study are referencing the mechanical MIE method (i.e. testing performed with an inductance of ≥1 mH). In addition, the MIE value of a material is typically given as a range, corresponding to the energy levels at which a dust-cloud ignition did and did not occur. We perform the calculation assuming the material can ignite at the lowest energy (for instance, if a material was found to have an MIE in the range 30mJ<MIEmat<40mJ, the value is taken to be 30mJ).

The MIT is defined in BS EN ISO/IEC 80079-20-2:2016, as the lowest temperature of a hot surface that ignites the most flammable mixture of dust and air under specified testing conditions. The MIT is determined by dispersing a material to form a dust cloud in a preheated oven or furnace, and then observing whether the temperature is sufficient to ignite the cloud generated. By varying the concentration of the material, and the dispersion pressure used, the mixture and conditions that are most sensitive to ignition are subjected to lower temperatures, until there is no longer any observed ignition.

For the sake of this study, all data has been acquired at DEKRA Organisational & Process Safety (DEKRA-OPS), using a Godbert-Greenwald (GG) furnace for the MIT, MIE III trickle-charge apparatus for the MIE, and 20-L sphere explosion vessel for the LOC.

At DEKRA, we perform testing in compliance with the latest standards. However, in practice there are certain limitations impressed on the calculation due to the use of data derived through testing. Firstly, the MIE apparatus used cannot reliably measure < 2 mJ and testing typically begins at 1000 mJ, as stipulated in the current standards. The lower limit is due to the stray capacitance in the system, having a greater impact on the results with respect to the % tolerance at such low energies. Generally, MIE values this low are more typical of vapours than dusts, but there are some dusts (e.g. sulphur) that have this level of ignition sensitivity. This means that material types with associated MIE values lower than 2mJ, or greater than 1000mJ cannot have an LOC result calculated accurately. In addition, the MIE & MIT test procedures were updated in 2016, following the release of BS EN ISO/IEC 80079-20-2. Whilst the MIE method remained the same, there were subtle differences between the two MIT standards. Data used in this study, derived prior to 2016 will have been performed to the equivalent British/European standard: BS EN 50281-2-1. This is deemed to have low impact on the overall dataset involved in this study, because the only significant difference between the different standards is the maximum temperature tested; the 80079-20-2 method states a maximum test temperature of 600, whereas BS EN 50281-2-1 stipulated a maximum test temperature of 650. However, to ensure consistency between the results obtained between the two MIT standards, only the results obtained that have been found to be <600 have been included in this study.

The LOC calculation requires the MIT to be ≤ 600°C and via the BAM oven method. Given the MIT test performed at DEKRA-OPS was carried out using a Godbert-Greenwald (GG) Furnace, the following calculation was used to convert the result, rounding down to the nearest 10:

$$MIT_{BAM} = \frac{MIT_{GG} + 10°C}{1.1}$$

This equation is valid only for 100 ≤MITGgg≤600°C [2]. These values correspond to limits for the BAM furnace results of 100 ≤MITbam≤550°C

These limits i.e. 2mJ<MIE<1000mJ, and 100 ≤MITbam≤550 , effectively limit the range of the calculated LOC values. Taking the largest, and smallest possible values, the calculation is limited to; 8.75%≤LOCcalc≤13.68%.

The calculation method proposed by R Siwek, 1996 is deemed to be "of concern" if the calculated LOC result (% O2 concentration) is greater than that of the experimentally determined result, under the assumption that any relevant safety factors would be applied to both values equally.

In total, 69 different materials were assessed.

Results

Out of the 69 different materials assessed, it was found that 25 had a calculated LOC that was greater than the experimentally determined value. This indicates that approximately c. 36% of the results are concerning, as they represent an optimistic view of the LOC contradicting the experimental data. 7 of these results were within 1% O2, and thus fall within an expected level of experimental error . However, the remaining calculated values differed from the experimental values by as much as 4.8%, which is considered to be a significant margin of deviation. Figure 1, displayed below, shows the relative calculated and measured results of each sample:

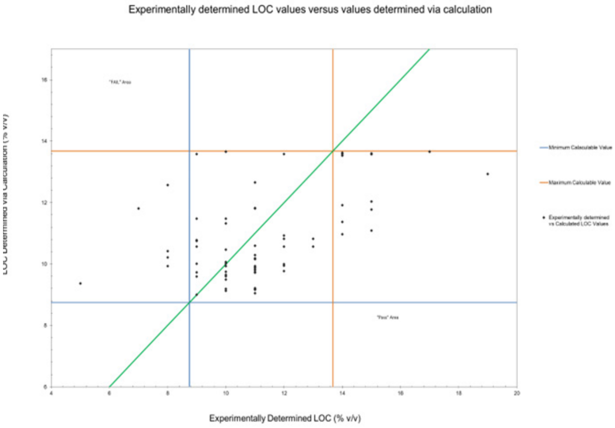

Figure 1: Experimentally determined LOC values, against calculated LOC values.

The figure shows boundaries that represent the minimum and maximum values the calculation can obtain (in blue and orange, respectively), and a trend line indicating where the calculated and measured results would be equal (in green). Therefore, the points above the line represent where the calculation produced a LOC result higher than the measured result (i.e. a "result of concern"), whereas points below the line represent where the calculation produced a LOC result lower than the measured value (i.e. a "pass"). Notably, points outside the "box" created in the middle by the maximum and minimum value lines, could not possibly agree with the equation. It was found that out of the 69 points of data, 6 had measured LOCs below the minimum possible calculable result, and 14 had measured LOCs above the largest calculable result.

However, this does not necessarily mean that the equation has no value, as the testing was performed on a myriad of different material types. There is the possibility that the equation only does, or does not work effectively for dust clouds made up of certain material types. To investigate this, the materials that had "results of concern" were categorised in order to assess whether any trends between material types could be determined. The results are outlined below, in Table 1:

Material	Count
Unknown	9
Pharmaceutical	2
Antibiotic	2
Coal	1
Sewage	1
Paracetamol	1
Olive Pellets	1
Chemical Emulsifier	1
Ruthenium(III) acetylacetonate	1
Pigment	1
Peanut Husk	1
Titanium	1
Insecticide	1
Pesticide	1
Fungicide	1

Table 1: Material types of materials with a concerning calculation result.

As can be seen, the materials that have "concerning" results constitute a wide variety of materials. Therefore, we cannot suppose that the equation is valid (or otherwise) for any given material type.

There also exists the possibility that the calculation could be used with an additional safety factor , effectively modifying the constant at the end of the equation. To investigate this, we calculated how much of an extra safety factor (to 1 decimal place) would be required to lower the number of concerning results, the results of which are outlined in Table 2 below:

Rate of Concerning Results (%)	Required additional LOC safety factor (%)
36.2	0
15	1.8
10	2.5
5	4.4
1.5	4.6
0	4.9

Table 2: The „Concerning results" rates of the calculation, with an additional safety factor

The 1.5% concerning result rate cut-off in the table represents the safety factor at which only one of the sixty-nine samples investigated had a concerning result, with an added safety factor of 4.6%.

It should be noted that even when the calculated LOC was considered a "pass", it could generate a result that is significantly below the measured LOC. Therefore, if the conservative calculated LOC result is used within a basis of safety, this would result in requiring a level of inertion that would far exceed what would be required in practice. To determine how often the calculation produced a result significantly below the measured result, we used the results of the 44 "passes", taking the "calculated" LOC away from the "measured" LOC, and counting how many values were above certain thresholds, as detailed in Table 3, shown below:

Threshold (%)	Number of Materials above threshold (number)	Number of Materials above threshold (%, 1 d.p.)
1.0	18	40.9
2.0	9	20.5
3.0	6	13.6
4.0	4	9.1
4.8	1	2.3
4.9	0	0

Table 3: Number of materials with calculated results above certain thresholds above the measured result.

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As it can be seen, over 40% of the passes had a calculated result of more than 1% above the measured LOC, over 20% had a result more than 2% above the measured LOC, and the calculations go as high as 4.8%, higher than the measured result.

Conclusion

In this study, we have examined and compared the results between the calculation method and experimentally determined result of the LOC from 69 different materials and found that c. 36% of the values derived from the calculation set out by R. Siwek, 1996, gave a result higher than those determined experimentally. This raises concern as it shows that the model is not sufficiently conservative when compared to empirical data.

It should be noted that because the MIE is normally taken as a range (e.g. 10mJ-15mJ), we have assumed that the MIE is the lowest value within that range. This would reduce the determined value from the calculation, thus lowering the expected number of "concerning" results. Despite this, a significant number of "concerning" results remained. Even if the equation were to be used with an additional safety factor, it would need to be substantially large (as much as 5%) which raises doubts about the practicality of a modified equation for the majority of applications. Additionally, when the calculation does provide a "safe" result for a material, a significant number of cases would have a result that would require a significant amount of protection above what is actually necessary.

It is notable that the Siwek equation is based on a trend generated from empirical data, and there is no theoretical justification given as to why the LOC of a material should be related to its MIT and inductive MIE results. This does not discredit the possibility that a link between them exists. From DEKRA experience, materials with low MIE and/or MIT values generally have lower LOC values, but the data we have generated suggests that the link is too tenuous to be used in real life situations. We posit this is likely due to the non-uniformity of dust clouds, and/or physical properties of the materials that may, or may not, have been accounted for (e.g. particle shapes, the resistivity of the sample, etc), that affect the real results of the testing performed.

Based on these findings, we conclude that the equation set out in "Determination of technical safety indicies indices and factors influencing hazard evaluation of dusts" by R Siwek, is insufficient at providing an accurate, reliable way of determining a material's LOC. Consequently, it should not be relied upon in replacement of an experimentally determined LOC when it is required for the purposes of managing explosion hazards.

References:

[1] "Determination of technical safety indices and factors influencing hazard evaluation of dusts", R Siwek.

[2] Guidelines for Safe Handling of Powders and Bulk Solids, CCPS (Center for Chemical Process Safety)

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