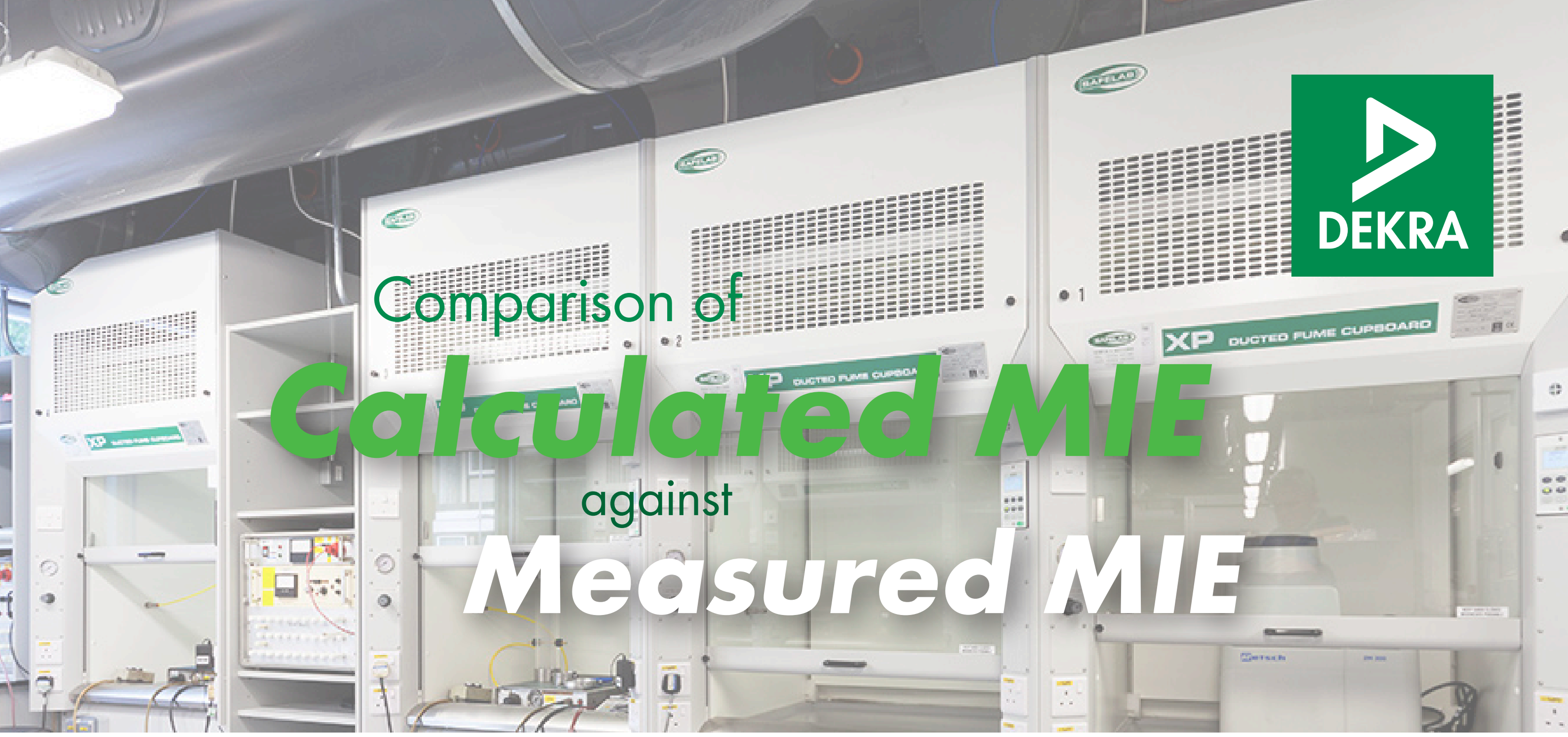

Comparison of calculated MIE against measured MIE following the conformity test procedure presented in BS EN 13821.

Abstract

The Minimum Ignition Energy (MIE) is defined as the lowest spark discharge energy capable of igniting a flammable dust, gas, or vapour, dispersed in air. There are various experimental methods for determining the MIE, as well as empirical formulae for estimating the MIE. The focus of this study compares estimated MIE values derived using empirical methods, against experimental results. Empirical formulae contained in BS EN 13821:2002 has been applied whilst experimental MIE data was derived following methodology also contained in BS EN 13821. For the materials considered it was noted, whilst some yield similar results, this is not true for all. Therefore, whilst the conformity test would serve as a means of test equipment validation, it is not deemed sufficiently reliable to provide data intended for practical application of any process safety measures i.e., formulating a robust Basis of Safety.

Introduction

With regards to combustible dusts, the Minimum Ignition Energy (MIE) is the lowest spark energy required to ignite a flammable dust-cloud. The data derived from this test can be used to assess a dusts' incendivity from a number of electrostatic and spark discharges. For example, spark discharges that can occur from isolated plant, equipment and personnel.

A powder MIE typically ranges from 1mJ to 1J but can also be much higher. The lower the MIE, the greater the risk of ignition. The flammability hazards associated with combustible dusts that might normally been seen as innocuous are often overlooked. For example, icing sugar has an MIE between 5 and 10mJ, provided it is sufficiently fine and dry [1]. To put this into perspective, an operator is theoretically capable of producing an electrostatic spark energy up to 10mJ [2].

There are several different experimental methods and apparatus used to determine the MIE of a dust. However, each design is essentially an electrical circuit capable of generating a discrete spark of known energy, across two electrodes. The dust under investigation is dispersed in the form of a cloud, through the spark, and observation made whether ignition occurs. If the energy of the spark is sufficiently high, it will ignite the dust cloud, resulting in a flame propagating through the cloud, with some heat and pressure effects. Testing is repeated at lower spark energies until no flame propagation is observed. A range of concentrations and a set number of repeat tests are then conducted. Due to the non-homogeneity of dust clouds, a significant number of negative, successive trials are required before a 'no ignition' is recorded. Stoichiometric concentrations of dust dispersions cannot be reliably reproduced as per flammable gases and vapours.

For the purposes of this study, a 'conformity' calculation is used to determine whether an alternative, empirical means will provide comparable results to experimental MIE. To calculate the conformity value, equation 1 taken from EN 13821:2002, Potentially Explosive Atmospheres – Explosion prevention and protection – Determination of minimum ignition energy of dust/air mixtures, pg 7, [3] has been considered.

Equation 1

$$E_S = 10^{\log E_2 - \frac{I\left(\log \frac{E_2}{E_1}\right)}{(NI+I)+1}}$$

Where:

- Es is the calculated Minimum Ignition Energy
- E2 is the energy at which the sample ignited
- E1 is the energy at which the sample failed to ignite
- NI is the number of concentrations at E2 where the sample did not ignite
- I is the number of concentrations at E2 where the sample did ignite

Equation 1 is valid for evenly distributed concentrations of dust, for at least a minimum of 5 different concentrations and for any given range of spark energy tested. The intention of the conformity test calculation is to verify different MIE apparatus; if two different apparatus yield results within a factor of 3, they are said to be "within conformity" [3].

Method

To determine the accuracy of Equation 1, 11 different combustible dusts were considered, where the MIE using both approaches were determined. DEKRA Organisational and Process Safety manufacture the MIE III apparatus which operates a voltage increase, trickle-charging circuit in accordance with the design principles contained in BS EN ISO IEC 80079-20-2 2016, page 32 [4]. MIE test results are specified as an ignition – no ignition range. This is the energy at which ignition is observed, and the energy where no ignition is observed, e.g., 40mJ – 50mJ over a range of powder concentrations. The materials were also tested in accordance with the conformity test procedure [3]. Using this method, the MIE is determined as a definitive, value. The ratio of the calculated result from the conformity test and the average of the upper and lower bounds of the range from the experimental method is determined using the equation below, referred to as R.

Equation 2

$$R = \frac{Calculated\ Result}{\left(\frac{Ignition\ Energy + No\ Ignition\ Energy}{2}\right)}$$

Where:

- R is the ratio
- Calculated Result is the MIE value calculated from the conformity test.
- Ignition Energy + No Ignition Energy are those of the range determined by the full MIE Test.

Values of R < 1 imply the calculated result was lower than the measured range, whereas for values of R > 1, the calculated result was higher than the measured range. A result close to 1 indicates the calculated result was similar to the measured result. As the purpose of the study was to assess the accuracy of the calculated method using as little data as possible, the minimum, valid number of concentrations was used in the conformity tests (i.e., 5).

Conformity tests between two apparatus would be deemed acceptable if the calculated result is within a factor of 3. Therefore, it was deemed acceptable for the purpose of this study, if the calculated result falls within a factor of 3 from the full measured test result (i.e., 0.33 < R < 3).

A range of different combustible dusts including organic, non-organic, metals etc. were tested. The measured MIE tests were undertaken by different, trained test operators, each following the same procedure. The subsequent conformity tests were performed by two different technicians. All experimental testing was carried out across two MIE III apparatuses. The dusts were prepared prior to testing, to be the driest and finest as was feasibly attainable, to ensure the most readily ignitable conditions. Each material was tested in the same condition for both test methods, as detailed in the table below. The energies used in the conformity tests were the same, to reduce potential bias. The initial energy used in the Conformity tests was 1J, reducing by a factor of 3 (and rounding to one significant figure) at subsequent energies (i.e. 1J, 300mJ, 100mJ, 30mJ, 10mJ, 3mJ).

The results of the MIE tests are summarised in the following table, from smallest to largest values of R.

Analysis

Type of material	Preparation (µm)	Meaured Result (mJ)	Calculated Result (mJ)	Calculated/Measured ratio (R)
Caramel Powder	<63	25-30	14	0.51
Synthetic organic powder	<63	6-8	4	0.57
Natural organic powder	<63	15-20	12	0.69
Locust bean husk, germ and endosperm grinded	As rec'd	300-400	250	0.71
Cocoa power	As rec'd	400-500	366	0.81
Ethylene/propylene copolymer	<63	40-50	37	0.82
Zinc Stearate	As rec'd	8-10	8	0.89
Unknown	<63	50-60	81	1.47
Ethylene/propylene copolymer	<500	300-400	670	1.91
Hay like material	<500	80-100	208	2.31
Aluminium dust	<63	50-60	818	14.87

The R values, with 1 notable exception, vary in the range of ~0.5 to ~2.5. According to the standard, these values are "within conformity" (i.e. 0.33<R<3). Out of the eleven test dusts, there was only one outlier. This was aluminium powder, which ignited as low as 60mJ in the experimental test method. However, in the conformity test, there were infrequent ignitions at the upper energy level (1000mJ), and no ignitions observed at the lower level (300mJ). This results in a very high calculated result. The reason for this discrepancy is apparent from the Conformity test method; as the procedure states; where there is no ignition at a set energy level, it can be assumed that the same powder concentration would not ignite at a lower energy level [3]. From experience of Minimum Ignition Energy testing at DEKRA, this is too assuming. Dust clouds are non-homogenous in nature; there will invariably be pockets of dust where the concentration will be either too lean or too rich. For this reason, the calculated, 'conformity' method is not suitable for consideration when determining a Basis of Safety.

A summary of the conformity test for the aluminium dust is shown below.

Energy (mJ)	Weight Distribution (g)				
-	0.8	1.6	2.4	3.2	4
1000	No ignition	No ignition	No ignition	No ignition	Ignition
300	-	-	-	-	No ignition

As can be seen, due to the sample not igniting at low powder concentrations at 1000mJ, it was assumed that the same concentration would not ignite at a lower energy. Therefore, only one weight was considered at the lower energy. In the full test, a powder concentration of 4g failed to produce an ignition at 80mJ. However, at 3.5g, an ignition occurred at this same spark energy. Similarly, four varying concentrations between 1.5 and 3.5g were assessed against a 60mJ spark, without ignition occurring. At this same energy, the aluminium powder ignited at a powder weight of 1g. This is shown in the table below.

Energy (mJ)	Weight Distribution (g)						
-	1.0	1.5	2.0	2.5	3.0	3.5	4.0
80	-	-	-	-	No Ignition	Ignition	No Ignition
60	Ignition	No Ignition	No Ignition	No Ignition	No Ignition	No Ignition	-

Limitations

The results from the conformity tests are considered more accurate for dust samples with 'full' measured MIE values, just above the no-ignition energy result. For example, dried straw has an MIE value in the range of 80mJ to 100mJ. However, the sample did not ignite at 100mJ in the conformity test. If it had ignited at 100mJ, as it did in the 'full' test, the calculated result would have dropped from 208mJ to 81mJ (assuming the material would not have ignited at 10mJ, or 30mJ). In addition, if the sample had not ignited at the highest energy tested (1J), there are insufficient powder concentrations considered to conform to the BS EN ISO IEC 80079-20-2 2016 standard, to definitively claim the Minimum Ignition Energy is above the highest value tested [4].

The conformity test appears unsuitable for certain combustible dusts because the procedure requires the material to ignite consistently. Other than the particle size and moisture content, dust-cloud concentration is one of the most influencing factors on a materials ignition sensitivity. In theory, if you could consistently produce a homogenous dust dispersion, reproducing the stoichiometric concentration would be relatively straightforward. If you could reliably reproduce the most ignitable conditions, finding the MIE value would be a simple and accurate process. In reality, however, this is not possible; therefore, the MIE will always be an indicative test, due to this level of uncertainty.

There are a large number of factors that can influence the ignitability of a dust-cloud. For example, experience has shown highly resistive powders have a tendency to agglomerate, especially when particles are repeatedly dispersed. Particle shape can vary considerably depending on how the material is manufactured (e.g., acicular versus spherical particles). The ignitability of poly-constituent materials can depend on the composition of the mixture. All of these variables can be somewhat mitigated by performing a large number of repeat trials over a wide range of powder concentrations. The conformity test generates results based on a smaller number of trials. This increases the likelihood of a material not igniting at a given energy. Should the same material be subjected to a larger number of repeat trials, such as in the 'full' test method, ignition has a higher probability of occurring.

Conclusion

Statistically, using the voltage increase, trickle-charging circuit test method, the conformity test has the potential to provide an estimate of a dusts Minimum Ignition Energy. Based on the trials performed in this study, the calculated method can be applied to some dust-types. However, it is not feasible to gauge which dusts this method would be applicable to, or not, least not without performing an initial 'full' MIE test. For this reason, the conformity test appears to be a suitable method in determining indicative MIE values but, only when the result has already been determined. This could serve useful for spot-checks, briefly verifying different MIE apparatus – as is the purpose of the test [1]. However, the method is inconsistent, in that it assumes a material will always behave ideally. There are dusts that are established as having greater consistency, which are utilised as 'reference materials' [5]. If this procedure is to be adopted for quality assurance purposes, equipment verification testing should be conducted on an appropriate material. This study has identified that even for comparative purposes, the calculated method may not accurately demonstrate a powders true MIE, due to the variables. The full, comprehensive test method covers a wider range of powder concentrations, across all energy levels, to account for the number of influencing factors of dust-cloud ignitability. Therefore, the conformity method should not be considered a substitute for the experimental method or when applying the results as part of a Basis of Safety.

References:

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[2] Explosive atmospheres - Electrostatic hazards, guidance, PD CLC/TR 60079-32-1:2018

[3] BS: EN 13821:2002, Potentially Explosive Atmospheres – Explosion prevention and protection – Determination of minimum ignition energy of dust/air mixtures

[4] BS EN ISO IEC 80079-20-2 2016

[5] ASTM 2019 2003 Standard Test Method for Minimum Ignition Energy of a Dust Cloud in Air

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