Technical Paper

Dusting off danger: Tackling resistive dusts using a non-combustible anti-caking agent for optimal ignition sensitivity

The study examines how **powder resistivity** affects dust cloud MIE, finding that adding Diatomaceous Earth can improve **dispersibility** and **ignition sensitivity**

Abstract

We investigated factors influencing dust-cloud Minimum Ignition Energies (MIEs) and their impact. MIE, defined by BS EN 13821:2002, is the lowest energy electrical spark-discharge capable of igniting the most combustible fuel-air mixture. Achieving a worst-case scenario in ignition testing can be challenging due to the variability of dust cloud formation, influenced by factors such as moisture content, particle size, and powder resistivity.

Powder resistivity can significantly impact measured MIEs, as high resistivity powders charge easily, leading to agglomeration and poor dispersion. This study introduced Diatomaceous Earth (DE), a semi-conductive, non-combustible dust, as an anti-caking agent to improve dispersibility. Tests on nine combustible powders showed that adding DE either maintained or increased ignition sensitivity. These findings show that DE can reduce the measured MIE via improved dispersibility, suggesting that forcibly breaking up agglomerations can lead to a richer fuel-to-air ratio.

Perform **risk assessments** to control and prevent **combustible dust explosions** effectively

Introduction

If you handle a combustible dust for commercial purposes, a risk assessment must be carried out for your specific process, under DSEAR (UK) or ATEX (EU) legislation. This risk assessment should identify whether the formation of explosive dust clouds is possible, and if so, the likelihood of ignition and subsequent harm to personnel. The overall risk should be reduced where possible, by firstly minimising activities that could give rise to an explosive dust cloud and furthermore by controlling ignition sources. To control ignition sources there needs to be a firm understanding of the combustible dusts' ignition sensitivity characteristics of which the minimum ignition energy is a key value.

Wherever there is manual, mechanical or pneumatic handling of dust, there is the potential for electrostatic 'tribocharging' to occur. This is the transfer of charge from one surface to another by way of contact. Electrostatic spark discharges are the result of a charged substrate breaking down against a conductive object, for example from equipment or personnel to a metal surface. For this reason, when defining a basis of safety for any given operation, you cannot discount electrostatic sparks as a potential ignition source and should assess the ignition sensitivity of your product. This is because the prevalence of sparks occurring in any given operation is likely to be high. Something else to consider in your risk assessment is the potential for "mechanical" sparks to occur. These are generated via the abrasive interaction of certain solid objects, such as metal or stone. For example, via grinding, impact or friction operations.

A typical dust-cloud MIE test is performed from 1 J down to 1 mJ. A low result indicates higher risk of ignition from electrostatic sparks, as less energy is required to trigger an event. For instance, based on the potential capacitance of a human being, protection for MIE results under 30 mJ would be additional earthing or bonding. However, less extensive intervention is required when MIE results exceed 100 mJ. This is because naturally occurring electrostatic spark discharges tend not to exceed 100 mJ (even for large objects such as tanks or silos). Therefore, more extensive prevention measures are required when a combustible powder has an MIE value under 100 mJ.

Dust cloud dispersions can vary wildly, due to differences in particle size, shape, and weight. Damp powders also disperse poorly, taking effect from as low as 5% moisture (by weight). As an employer or business owner, it is in your best interest to ensure any safety measures that are based on test data, encompasses the most severe risk scenario. At DEKRA Organisational & Process Safety (OPS), we control certain parameters to ensure greater reliability from measured dust-explosion data. How this is achieved practically is through particle size reduction (to a cut-off value of 63 μm) and drying (to < 5% moisture content). It is the fine and dry fraction that holds the greatest ignition-sensitivity and produces the highest potential explosion violence. Keeping these two parameters consistent reduces the number of variables that can greatly influence the result.

Challenges in **MIE assessment:** resistive and conductive powder agglomeration impacts results

From a practical standpoint, it is our experience at DEKRA OPS that highly resistive or conductive powders pose a greater challenge during MIE assessment. Resistive powders – such as plastics, have an affinity to agglomerate or clump at the site of the electrically charged electrodes, as seen in figure 1. Whilst conductive and dissipative powders do not behave in the same way, conductive powders – such as metals, can pose a different challenge. The electrostatic spark discharges from the MIE apparatus can intermittently choose a different path from that of the electrode-gap, as intended. It is common for discharges to travel through the conductive powder layer coating the inner wall of the Hartmann tube (fig. 1). Whilst both conductive and insulative challenges can prolong the overall duration of testing, agglomeration is deemed of higher importance to investigate. This is because dispersibility, and therefore the quality of the fuel in air atmosphere may influence the final MIE result. As agglomeration does not happen in a consistent manner under test conditions, there is the potential for erroneously high values to be measured.

It is well documented that agglomerated particles can lead to higher measured MIE values, indicating a material is less sensitive to ignition than in reality. This can be true of nano powders that have a significantly low particle size. As a rule of thumb, the MIE value typically decreases as particle size decreases. This is because finer particles have a greater surface area distributed within a dust-cloud and therefore can more readily propagate a reaction. However, it has been discovered that when extremely fine powders are assessed the MIE can in fact increase. When dropping below a very small particle size, agglomeration becomes dominant leading to larger particulates reducing the potential surface area in dispersion and hence a higher MIE value. The following study documented such findings: Journal of Physics: Conference Series 304 (2011) [2], whereby the MIE sensitivity of various metal powders was assessed using differing particle sizes.

Several solutions to the problem of dusts agglomerating were presented and trialled.

1. A glass Hartmann tube to replace the standard acrylic tube.
2. An insulative barrier to be applied to the electrodes.
3. An antistatic, anticaking agent to be introduced to the test-material.

1. It was initially predicted that glass would be less insulative than acrylic and thus would contribute less to the charging of a dispersed test-material. However, following in house resistance measurements, we found the following:

Table 1	Acrylic tube (E12 Ω)	Glass tube (E12 Ω)
Trial 1	66.3	55.4
Trial 2	87.3	72.3
Trial 3	103	77.4
Average	85.6	68.4

Table 1: resistance of acrylic vs glass.

The resistance values are very similar between both material-types. Therefore, the use of a glass Hartmann tube was discounted as a variable for the tribocharging of dispersed powders.

Addressing electrode dust buildup and spark sensitivity in **MIE testing**

2. The brass electrodes are the principal gathering point of unburnt, fugitive dust within the Minimum Ignition Energy equipment. During testing, dust is dispersed between the electrodes. During this process electrostatically resistive powders can gather at the site of the electrodes, due to the interactive forces. Although manual agitation can be used to remove this dust accumulation, it was proposed that insulating the electrode body may reduce the fugitive buildup during MIE assessment. This would have been via a resistive sheath to coat the brass electrodes. From a practical standpoint, the electrodes are manually adjusted within the Hartmann tube throughout each test, to alter the 'spark gap' size. During this time, it is unlikely that any insulative coating would remain 100% intact. It is also unknown the extent of which this coating would have impacted the intended spark energy e.g. via additional capacitance introduced to the circuit. For this reason, method 2 was also discounted as a viable option.

3. For the purposes of this study, the addition of an anti-caking agent was selected as the only practicable option.

Whilst there are various spark generating systems [1] that can be used to determine the MIE of a dust, the overall test-apparatus does not vary a considerable amount. Each design is based on an electrical circuit used to create a spark of known energy, across an electrode gap, housed within a Hartmann tube. The spark that discharges from a series of capacitors is intended to travel through a dispersed dust. Depending on the ignition sensitivity of the material-type, the spark may result in flame propagation through the dust-cloud, if the spark-energy is sufficient. If a high energy spark is required to ignite a dust (e.g. 500 mJ), then it is considered insensitive. However, if low energy sparks (< 5 mJ) cause ignition then the material is considered sensitive. Consider the prevalence of low energy sparks in the workplace. Based on the average capacitance of a human being, an operator has the potential to discharge a spark as high as 10 mJ. This capacitance may increase to 30mJ if large conductive tools are being handled. This information has been referenced from IEC 60079-32-1:2018 [3] which states:

"The worst-case voltage which may commonly be acquired by people is about 20 kV. With the typical capacitance of the human body being about 150 pF, the resulting maximum stored energy is about 30 mJ. However, due to the high ohmic resistance of the human skin about two thirds of the stored energy gets lost. For this reason, sparks from the human body have a maximum equivalent ignition energy of only 10 mJ except when handling large conductive tools."

MIE assessments continue at lowering spark energies until there is no flame propagation witnessed across a series of powder-concentrations. Multiple repeat trials are performed to increase the reliability of your 'no-ignition' energy level. This is a common theme for dust-explosion tests (e.g. MIT, MEC, Pmax/Kst), more so than gas or vapours as stoichiometric concentrations of dust dispersions cannot be reliably reproduced.

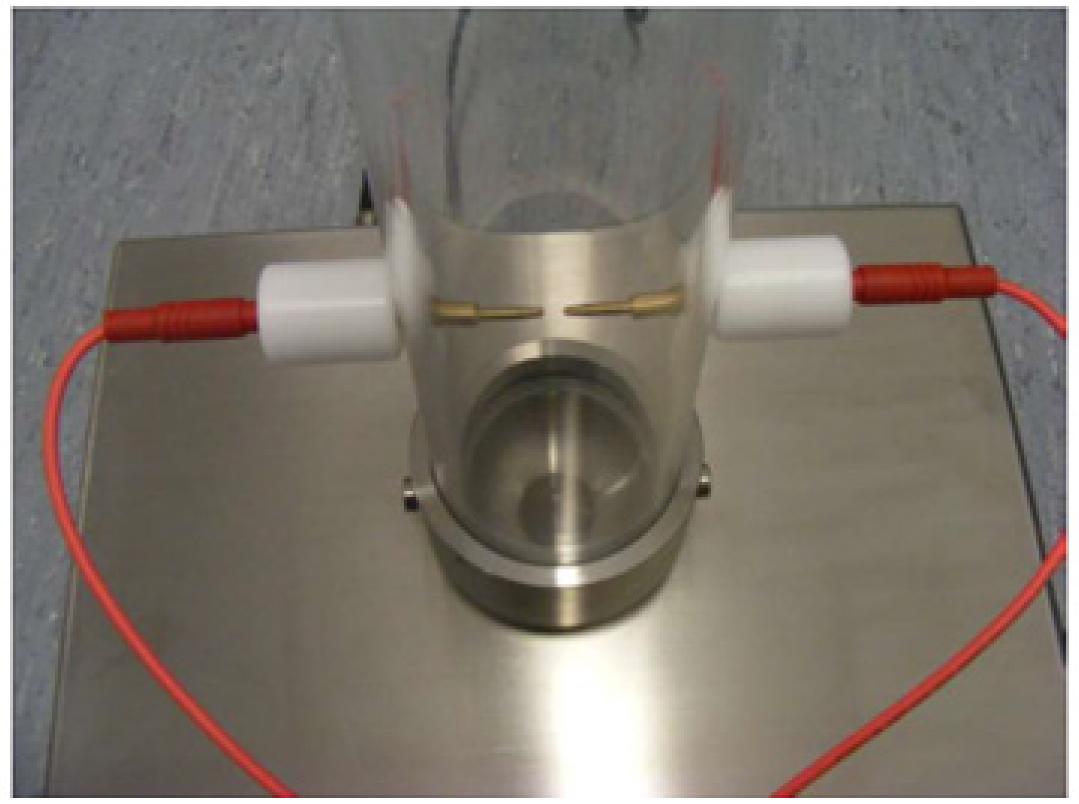

Figure 1: Hartmann Tube, MIE apparatus (DEKRA Instrumentation dept.)

Testing **Diatomaceous Earth's** effectiveness as a non-combustible anticaking agent to improve **ignition sensitivity**

Method

The overall aim was to identify whether the addition of an anticaking agent could increase ignition sensitivity of resistive powders, that readily agglomerate. It is vital that the anticaking agent is non-combustible, to have confidence that any influence on the ignition sensitivity can be linked to dispersion characteristics only. In this study, a known non-combustible powder, Diatomaceous Earth was selected as it falls into the boundary of a semi-conductor with a powder volume resistivity (PVR) of 1.0E+7 Ωm.

Stage 1: Control

The first stage of this study will be a screening of four separate Diatomaceous Earth percentages, to find the optimum ratio to add. The following would be assessed: 2.5%, 5%, 10% and 20% by weight.

To verify that the optimum Diatomaceous Earth ratio does not negatively impact ignition sensitivity of a test-powder, it would be added to a material that is known to be highly sensitive to electrostatic sparks. Niacinamide is a globally accepted 'reference powder', tests will be performed using this material as a 'control' due to its consistent dust-explosion characteristics. Niacinamide is also resistive, as detailed in stage 2.

Reference materials are used to verify that a test-system is functioning correctly. A worldwide round robin is performed annually using a specific batch of Niacinamide. In 2022, 27 different global laboratories submitted their findings (including DEKRA OPS), with a result of 0.5 – 4.6 mJ, defined as the acceptable tolerance for correctly functioning MIE apparatus [4].

Stage 2: Resistivity assessment

The next stage will be to assess the impact of diatomaceous earth on the overall resistivity of the powder, to assess the impact of the semi-conductive anticaking agent. Powder Volume Resistivity tests will be performed on scaling concentrations of the anticaking agent (Diatomaceous Earth) against the Control Sample (Niacinamide). Tests to be performed at 25% Relative Humidity in accordance with BS EN 61340-2-3:2000 [5].

Stage 3: Optimal Testing

Once the optimum Diatomaceous Earth ratio is determined, the final stage will be to assess how effective the anticaking agent can disrupt resistive powder agglomerations across various test-powders of differing material types. In turn, efficacy would be a measure of a decrease in MIE result, with the lower the value, the greater the influence. A series of trials would be performed on various powders that were observed to agglomerate when dispersed, using the optimum Diatomaceous Earth ratio determined in the stage 1 control.

Identifying optimal **Diatomaceous Earth** ratios to enhance ignition sensitivity in resistive powders

Results

Stage 1: Control

Upon assessment it was determined that at 5, 10 and 20% Diatomaceous Earth concentrations, the MIE value increased. A higher MIE value means that the material becomes less sensitive to ignition, meaning the anticaking agent was having a negative desired influence at ratios > 5% (w/w). At 2.5% Diatomaceous Earth the MIE result was seen to reduce (figure 2), which is a sign of increased ignition sensitivity.

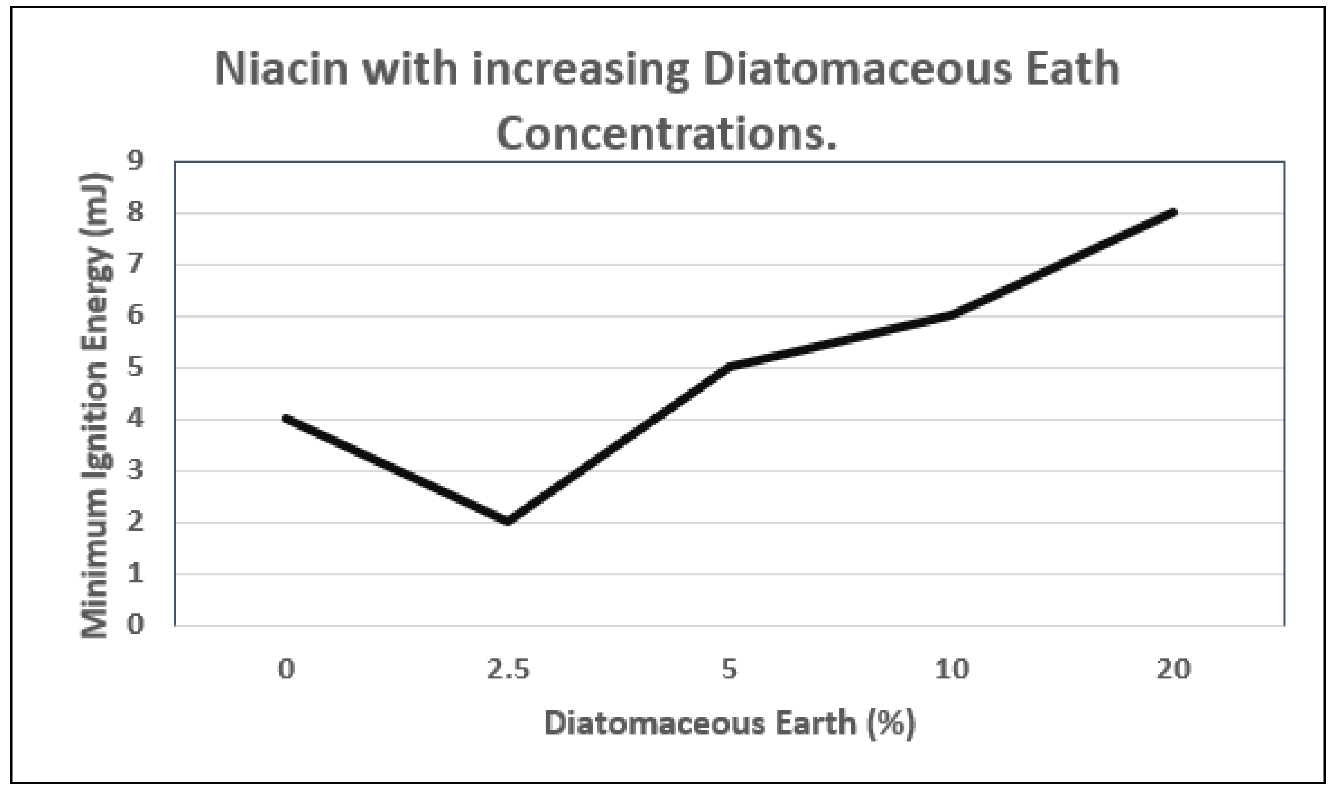

Figure 2: Diatomaceous Earth (%) against Minimum Ignition Energy (mJ) sensitivity test on Control Sample (Niacin)

	No Ignition (mJ)	Ignition (mJ)
Niacin	3	4
Niacin 97.5%:DE 2.5%	N/A*	2
Niacin 95%:DE 5%	4	5
Niacin 90%:DE 10%	5	6
Niacin 80%:DE 20%	6	8

Table 2: Minimum Ignition Energy of Niacin : Diatomaceous Earth Ratio's

*The lowest achievable energy value is 2 mJ using the MIE III apparatus at DEKRA OPS. This means that the material was so sensitive to ignition that a very low energy spark-discharge is capable of initiating a dust-explosion. Whilst figure 2 displays a result of 2 mJ, this in fact conservative as the end result could be lower, however further assessment was beyond the measurement limits of the test equipment.

Stage 2: Resistivity assessment

As the overall electrostatic properties may change with the addition of Diatomaceous Earth, Powder Volume Resistivity (PVR) testing was conducted on scaling concentrations with Niacinamide.

Table 3	Volume Resistivity (Ωm)
Niacinamide - 100%	1.3E+14
Niacinamide : Diatomaceous Earth - 95%:5%	8.3E+13
Niacinamide : Diatomaceous Earth - 90%:10%	4.7E+13
Niacinamide : Diatomaceous Earth - 80%:20%	8.3E+12
Diatomaceous Earth - 100%	8.4E+07

Table 3: Powder volume Results for Niacin/DE powder mixtures

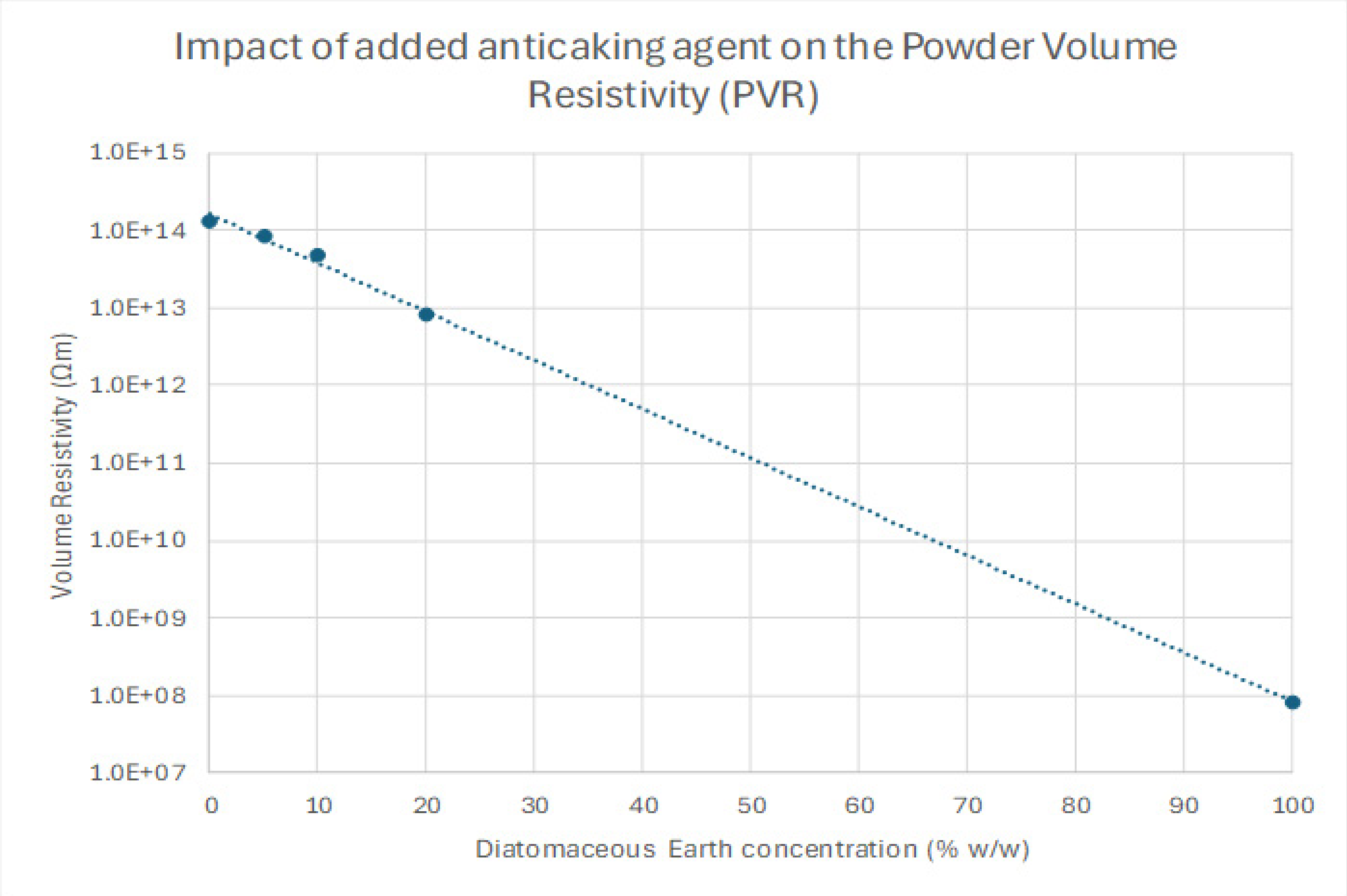

Figure 3: Resistivity extrapolation of Niacin + DE

Table 4	Volume Resistivity (Ωm) 25% Relative Humidity
Niacinamide	1.3E+13
Diatomaceous Earth	8.4E+07

Table 4: Volume Resistivity measurements

The trend in resistivity displayed in figure 3 shows that as the ratio of Diatomaceous Earth increases, the resistivity of the material decreases, consistently. Diatomaceous Earth is therefore influencing the overall conductivity of the powder bulk, when introduced.

Adding **Diatomaceous Earth** reduces MIE of resistive dusts by disrupting **agglomerations** and **enhancing** dispersion

Stage 3: Optimal Testing:

A range of six combustible dusts were selected as test-powders, based upon their affinity to readily agglomerate. The assessment was carried out in two parts, measuring the MIE of each as a control, then again with added diatomaceous earth (2.5% w/w). Each MIE assessment was carried out to the same standard operating procedure (SOP) following the methods outlined in the most up to date testing standard [7]

During the course of this study, two experienced operators were responsible for conducting each MIE assessment. All experimental testing was carried out using MIE III apparatus, designed and manufactured in house at DEKRA Organisational & Process Safety. To decrease variables, test-powders were initially prepared prior to testing to a moisture content of < 5% (w/w) and a particle size of ≥ 75% < 63µm.

The results of the testing are summarised in the following table:

Table 5	No Diatomaceous Earth (control)		2.5% Diatomaceous Earth added (w/w)	
	No ignition (mJ)	Ignition (mJ)	No ignition (mJ)	Ignition (mJ)
Cosmetic material	6	8	5	6
Plant-based material 1	10	15	4	5
Plant-based Material 2	>1000	>1000	>1000	>1000
API 1 (Antibiotic)	70	80	50	60
API 2 (Antibiotic)	60	70	50	60
API 3 (Antibiotic)	60	80	50	60

Table 5: Minimum Ignition Energies of Distinct Samples.

Results obtained indicate that with a small addition of inert, anticaking agent the Minimum Ignition Energy of electrostatically insulative powders can reduce. Plant Material 2 is an outlier, as there is no measured reduction in the MIE result. In this case there was no ignition observed up to the maximum permitted energy level in the test procedure (1000 mJ). It may be that the MIE result does drop with the addition of diatomaceous earth (e.g. from 1300 to 1100 mJ). However, this information would provide no additional, worthwhile information from a process safety perspective. This is because an MIE result exceeding 1000 mJ indicates a high insensitivity to electrostatic spark discharges. There would be no requirement for any additional safety measures to mitigate electrostatic sparks as a potential ignition source with an MIE value this high.

Conclusion

Results indicate the addition of a small quantity (2.5% w/w) of Diatomaceous Earth can lower the measured MIE of a resistive dust. There are two theories as to why this is. Firstly, the anticaking agent forcibly disrupts agglomerations from forming by creating a physical barrier between charged particles. Second, the overall resistivity reduces as diatomaceous earth is more conductive. This reduces the ability to retain attractive intermolecular forces, and therefore reduces the overall affinity for agglomerations forming.

It was noted by operators that with the addition of a Diatomaceous Earth there was a visible reduction in agglomerations seen under assessment. It was also noted that there was a more consistent spark discharge through the test-sample, when dispersed within the trickle-charge sparking system. From our experience, this ease of sparking can be typical of a well dispersed dust-cloud passing through the

Adding Diatomaceous Earth improves **dust dispersibility**, impacting MIE values and ignition sensitivity

With improved dispersibility, there is a greater prevalence of airborne fuel. With a higher proportion of suspended particles, there is an increase in potential surface area for a reaction to occur and propagate. This added surface area increases the exposure to atmospheric oxygen and the spark discharge from the MIE apparatus. These factors lead to a greater chance of forming a flammable atmosphere as there is a higher probability of achieving stoichiometric conditions.

As the percentage of anticaking agent increased above 5% by weight, the ignition sensitivity was found to decrease. This is because, whilst limited agglomeration occurred, there was a greater proportion of non-combustible dust in the mixture. This increase in non-combustible component negated the positive influence from the improved dispersibility. Whereas, with an added 2.5% anticaking agent, there was less impact from the additional non-combustible component, with improved dispersibility the greater influential factor on dust-cloud ignition sensitivity.

Discussion

Based on the data obtained in this study, the introduction of an antistatic, anticaking agent can either positively or negatively impact the measured MIE value, dependant on the ratio introduced. The MIE test is designed to demonstrate the ignition risk of a dust, assessing the sensitivity against spark discharges. It is always recommended that materials are assessed in a representative condition for dust-explosion characteristics, to encompass the highest level of risk. Consider all potential ignition sources that could be present in the vicinity of a combustible dust, and what aspect of the process presents the most explosible conditions. In practice, testing in the finest and driest condition is not always achievable, e.g. for powders that do not disperse well. However, in this study, it has been found possible that with the addition of a small proportion an antistatic, anticaking agent, dispersibility can improve and in turn generate more conservative test data. Depending on the level of influence, this could result in a shift of the hazard classification of a given material.

It may be deemed overly conservative to artificially prepare a test-sample to a more ignition sensitive condition, ahead of MIE assessment. However, when test data is used in the design or implementation of safety measures in the workplace, how thorough should this be? Consider both normal and abnormal operations and the potential risks that could arise. With limited information available around the influence of anti-caking agent on dust explosion characteristics, it is not recommended that any aspect of this study be put into any real-world practice. However, it is thought provoking that with the addition on a non-combustible ingredient, a powder has the potential to be more ignition sensitive. In fact, the exact opposite would seem logical, as dilutions are often utilised as methods of risk control. With the vast quantity of influential variables that can impact the sensitivity and severity of dust explosions, there should be confidence that test data is reputable and truly representative.

At DEKRA Organisational and Process Safety, we have been performing such testing for 30+ years and pride ourselves on our robust quality processes, holding both GLP and various ISO accreditations.

References

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