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The Minimum Ignition Energy of Powder Mixtures

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The minimum ignition energy (MIE) is one of the most important parameters for the assessment of explosion risks and the design of protection concepts when handling combustible powders. With the introduction of the ATEX regulation in Europe, the issue of ignition source analysis has become relevant not only in large bulk production plants where mostly powders of pure solids are handled, but also in mixing and standardization plants, in particular in pharmaceutical production, where mostly mixtures of different solids are processed. Investigations have shown the effect of mixing dust explosive powders with inert or less sensitive auxiliary materials [Bartknecht, Janes et al].

These investigations have been extended in this paper. A phenomenological mathematical description of the MIE of mixtures depending on the MIE of its components and the composition is proposed.

The approach is the extended to the MIE of pure powders with complex granulometry.

1. Experimental

The MIE has been determined using the well established standard procedure programmed on a MIKE -3 apparatus. The procedure yields a range for the MIE in logarithmic steps from 1, 3, 10, etc. and a statistical most probable value Es. Where only the range was determined, the low boundary was used in the graphs. All measurements have been made without inductivity in the spark circuit.

The MIE of substances or mixtures which could not be ignited in the MIKE-3 equipment are indicated as “1000mJ” if ignition with the permanent spark in the modified Hartmann Tube occurred.

The granulometry has been determined by Laser diffraction on a Beckmann Coulter LS apparatus using the Fraunhofer Model and a Malvern Mastersizer 3000 with Aero S set-up.

Powder fractions were obtained by sieving on a vibrating sieve tower with brush aid. The preparation of powder fraction is not straight forward and requires special techniques. Careful adjustment of sieving time and fraction control particle size analysis is necessary.

The focus was on mixtures where one component had a rather low MIE, as these cases are of special interest related to the question, whether the admixture of a less sensitive substance would significantly increase the MIE and thereby have a kind of inerting effect.

2. Results

2.1 Mixtures with inert substances

As shown in Figure 1 MIE in binary mixtures is not a linear Function of the concentrations in terms of percentage by weight. Rather, MIE is largely determined by the lower MIE. Up 80%w/w of the less sensitive component, MIE increase (within the precision of the test method) not more than a half decade (a factor of approx. 3).

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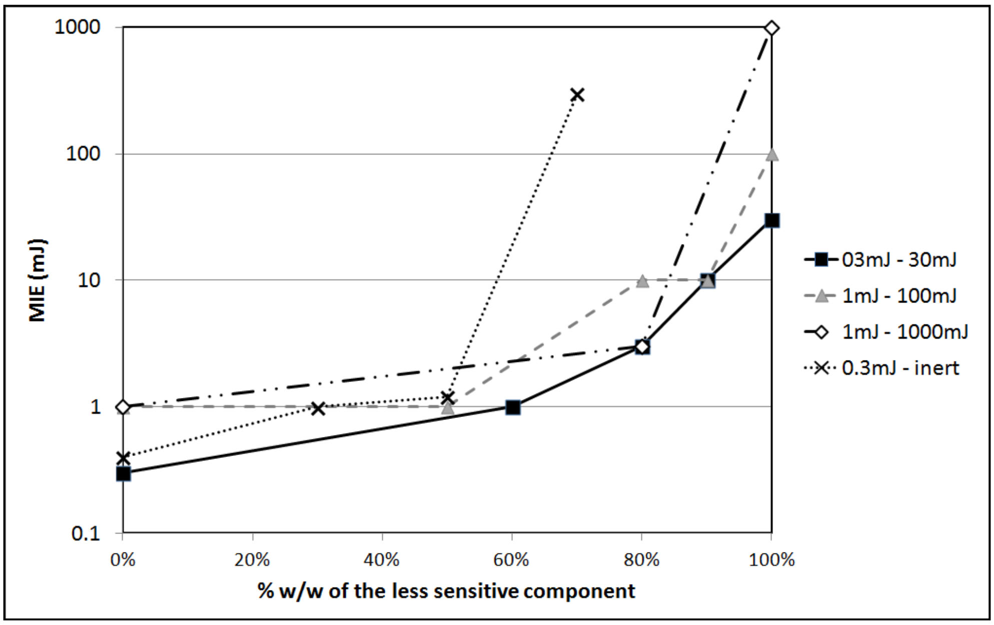

Figure 1: MIE of binary mixtures of combustible substances

The MIE of the mixture with the inert substance increases already above 50% of the inert substance. This is agreement with the observation by Bartkecht [Bartknecht, 1993, page 337-341]: Mixtures of organic materials with various inorganic salts (i.e. potential extinguishing media) were no ignitable even in the 20L sphere with 10kJ igniters above concentrations of 40 to 65%. On the other hand, the data by Janes [Janes 2013] show, that some mixtures of organic powder with inert substance were ignitable with energies below 1000mJ even at 80% inert content. This difference indicates that not all inerting substances have the same phlegmatizing power – may be due to different phlegmatizing mechanisms. Also, the density, which is comparable within the family of organic powders may be quite different for the inert substances. a comparison based on % by weight is therefore inherently incorrect.

2.2 A phenomenological Model for organic mixtures

If we focus on mixtures of combustible substances with different MIE, it becomes obvious, that the MIE of the mixture is determined mainly be the more critical (lower) MIE.

A useful mathematical description of the data observed in Fig. 1 would be

$$\mathrm{MIE(mixture)} = \left[\frac{C_1}{\mathrm{MIE}_1} + \frac{C_2}{\mathrm{MIE}_2}\right]^{-1} \tag{1}$$

i.e. the MIE of the mixture can be estimated with the geometrical mean of the MIE of the components.

Taking into consideration that the test method yields only MIE-ranges of a half-decade (i.e 1-3mJ; 3-10mJ) the calculated lower boundary of these ranges is shown in figure 2. No experimental value is below the estimated curve. Therefore equation (1) can be used to obtain conservative estimates for MIE for binary organic mixtures, e.g. tablet mixtures in the pharmaceutical industry.

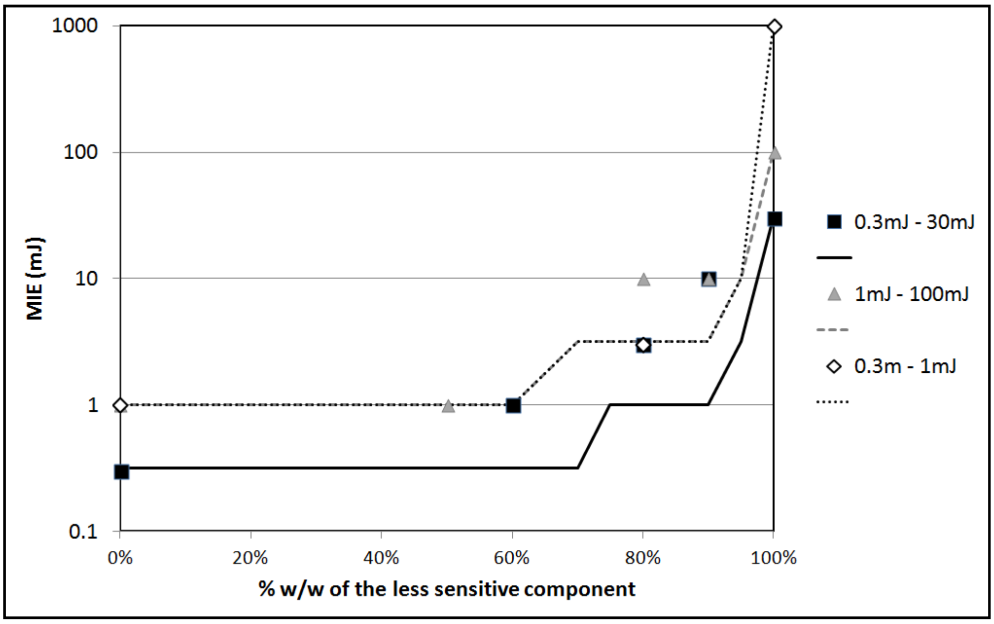

Figure 2: Calculated MIE values as a function of the composition according to equation (1).

2.3 Application to the Granulometry

It is well known that the MIE decreases with decreasing particle size [Bartknecht]. Therefore testing standards require that MIE is determined with a defined sieve fraction of the powder, i.e. <63 µ according to [EN 13821] and <71µ according to [ASTM E2019-03].

The dependence of MIE on the particle size has been investigated [Bartknecht, page 216]. The Ex-Tools Program by Kühner [Cesana] (the manufacturer of MIKE-3) uses the following equation to estimate the MIE as a function of the particle size (M=median values):

$$\mathrm{MIE(M)} = \mathrm{MIE_{ref}}\left[\frac{\mathrm{M}}{\mathrm{M_{ref}}}\right]^{2.5} \tag{2}$$

Combining equations (1) and (2) results in the following equation for MIE of known granulometry C(D), where D is the particle diameter:

$$\mathrm{MIE(observed)} = \left[\int_0^{500}\frac{\mathrm{C(D)}}{\mathrm{MIE(D)}}\mathrm{dD}\right]^{-\mathbf{1}} = \mathrm{MIE_{ref}} \times \left[\int_0^{500}\mathrm{C(D)} \times \left[\frac{\mathrm{D_{ref}}}{\mathrm{d}}\right]^{2.5}\mathrm{dD}\right]^{-\mathbf{1}} \tag{3a}$$

or, if the distribution function is given in defined intervals i

$$\mathrm{MIE(observed)} = \left[\sum\nolimits_{\boldsymbol{i}}\frac{\mathrm{C_i}}{\mathrm{MIE(D_i)}}\right]^{-\mathbf{1}} = \mathrm{MIE_{ref}} \times \left[\sum\nolimits_{\boldsymbol{i}}\mathrm{C_i} \times \left[\frac{\mathrm{D_{ref}}}{\mathrm{D_i}}\right]^{2.5}\right]^{-\mathbf{1}} \tag{3b}$$

Based on equations (1) and (2), it can be conclude that the MIE of a powder is largely determined by the fines. This is illustrated by the following model calculation, combining the two equations for a model case with $\mathrm{M_{ref}}$ = 63µ and $\mathrm{MIE_{ref}}$ = 10mJ

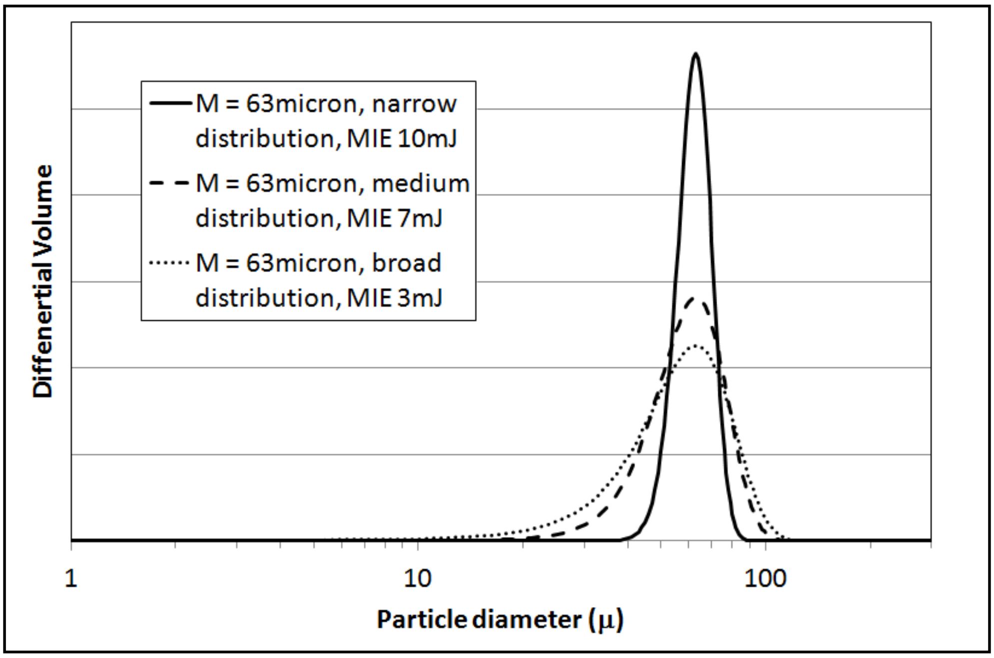

Figure 3: Effect of the particle size distribution on MIE based on equation (3a)

2.4 Experimental Examples

The MIE of two samples (A and B) with almost identical median values were determined. Sample B had a broader distribution. i.e. more fines and more coarse particles. The observed Es values were compared to the values obtained with equation (3b), using MIE_{ref} = 970mJ and D_{ref} = 63μ as best fit:

Es(A) =67mJ,	MIE(calculated) = 55mJ
Es(B) = 120mJ,	MIE(calculated) = 126mJ

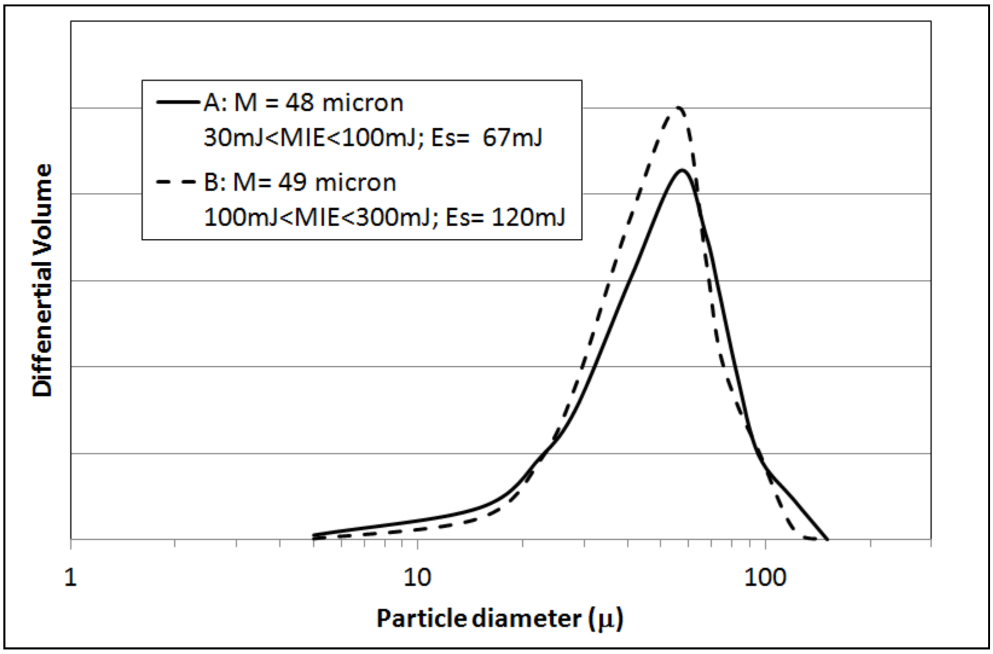

Figure 4: Experiential example of the effect of the particle size distribution on MIE.

The formula was also applied to powders and powder mixtures of complex granulometry (Figure 5). The results are shown in Figure 6.

Notice that the sample (c) has a lower MIE than sample (b) despite the low median value, due to its high content of fines.

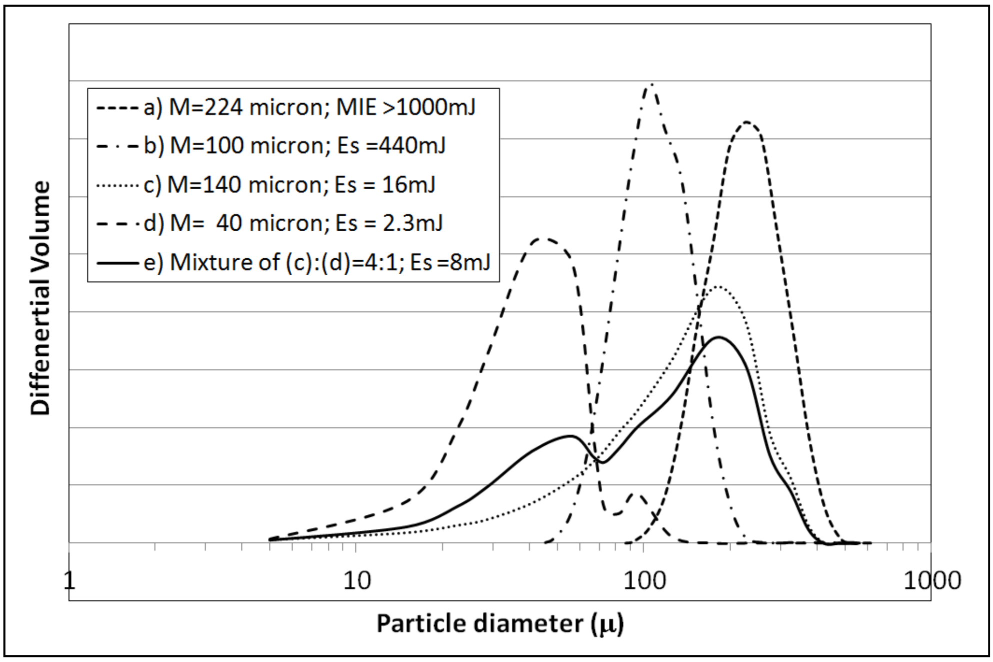

Figure 5: Different granulometries of the powder to investigate the effect of the granulometry on the MIE.

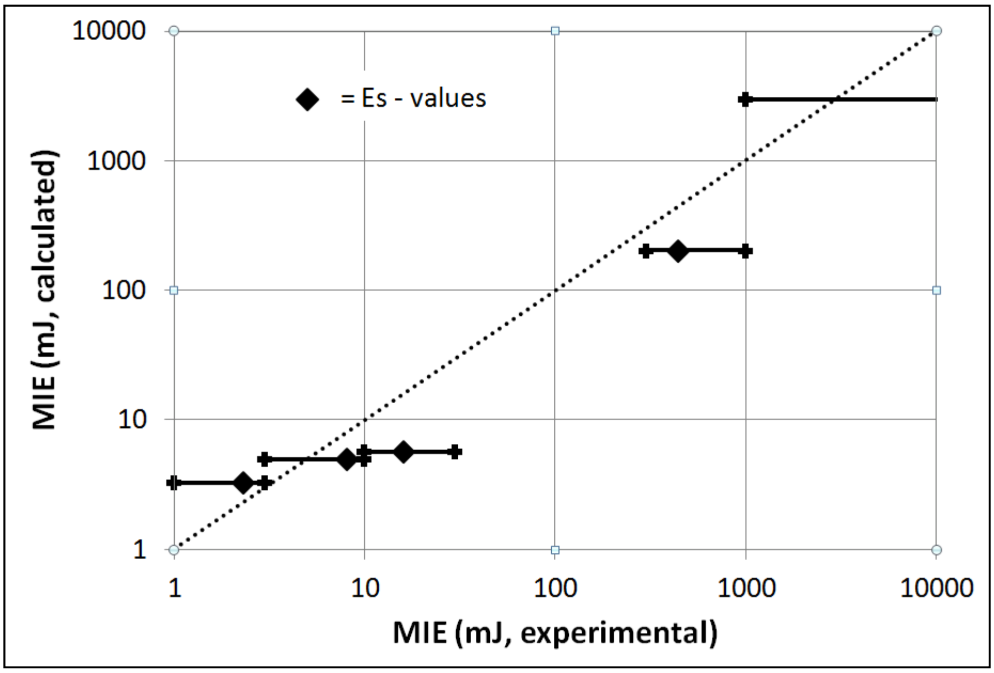

Figure 6: Experimental and calculated MIE values for different granulometries of a powder. The calculated values were obtained, using the "best fit reference value MIE_{ref} = 100mJ for M_{ref} = 63μ

The correlation in figure 6 shows at least the correct sequence of MIE values, but the quality of the calculated values is still unsatisfactory. Reason for this are to be investigated. Some of the quite evident points to be questioned are:

- The power 2.5 in equation (2) is obviously not generally applicable [].
- The determination of particle size distributions depends on the method. It is not straightforward to prepare a sample for particle size determination which is representative for the distribution in the dust cloud in MIKE-3. Even during the particle measurement itself the distribution be influenced by the pneumatic insertion of the powder.

3. Conclusions

The inerting effect of non-combustible powders depends more on the nature of the inert material than on the MIE of the combustible powder.

Mixtures with inert additives may be ignitable even when containing more than 80% of the inert material.

The MIE of a powder mixture is defined by the finest fraction. The content of fines after granulation and compacting processes used in pharmaceutical is normally too high to justify the use of non-standard MIE values obtained from coarse fractions. Protection concepts may only be based on MIE obtained form samples with high median values, if the granules are stable and no disintegration by the process (i.e. friction during sieving or transfer operations) occur.

Due to granulation or compacting processes, granulometry of powders in galenical production can be quite complex, i.e. the particle size distribution may shown several maxima, such that the median value is insufficient to characterize the powder.

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