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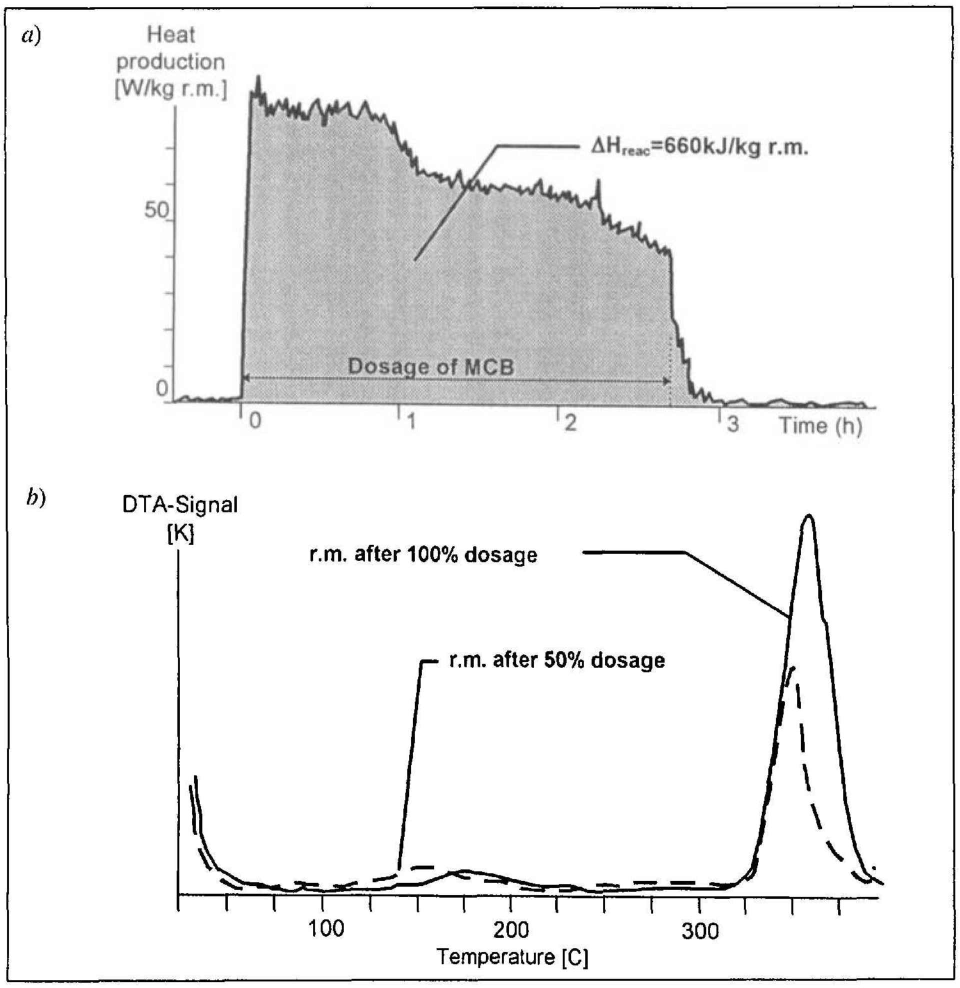

Fig. 4. a) *Reaction Calorimetry of the dinitration process.* The heat production almost immediately drops to zero after termination of MCB dosage, *i.e.*, there is only negligible accumulation of unreacted MCB. b) *Dynamic differential thermoanalysis* (DTA) *of the reaction mixture* (r.m.). After 50% of MCB addition, the small exothermic signal above 110° is attributed to the nitration of unreacted MCB/CMNB. The main exothermic signal above 300° results from the decomposition of the nitro compounds [2].

Concluding Remarks

The higher purity of treated acid stream produced by the new process is not classified as a hazardous waste stream by the authorities. This ruling allows the transportation and use of the treated acid without extra regulatory constraints other than those applying to acids in general.

An attractive rate of return is being realized on the capital investment: The savings of using the extraction column are such that the return on investment is as short as one year. 80% of this savings result from the cost avoidance associated with off-site regeneration. Instead of a paying for regeneration, the treated acid is being sold to cover transportation costs. Another 20% is contributed by improved CDNB yield, resulting from better organic recovery in the extraction column.

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Electrostatic Hazards in Powder Handling Operations

Martin Glor*

Abstract. Charge build-up is observed in most powder handling operations as soon as the powder is highly insulating or the equipment is made from highly insulating material. The ignition probability by discharges due to static electricity can drastically be reduce in practice, if only conductive material is used for all parts of the equipment and installations and if all these parts are safely earthed. The question remains, however, whether the charge retained on a highly insulating product in powder form will be able to ignite this powder, even if the powder is handled and processed in conductive and earthed equipment. Based on extensive research performed during the last decade in this field, ignition of highly insulating powder must be expected under certain circumstances (formation of so-called cone discharges), even if the powder is handled or processed in metallic and earthed equipment.

1. Charge Build-up on Powders

Charge build-up is intrinsically related to the handling and processing of electrically insulating powders. In most physical operations with powders such as filling, emptying, sieving, grinding, mixing, dust separation, pneumatic transfer, *etc.*, separation processes between the powder particles themselves and the walls of installations occur. As a result, the powder as well as parts of the installations may become charged. Different types of discharges may be generated from charged installation parts and from the charged product. The charge build-up on insulating powders is

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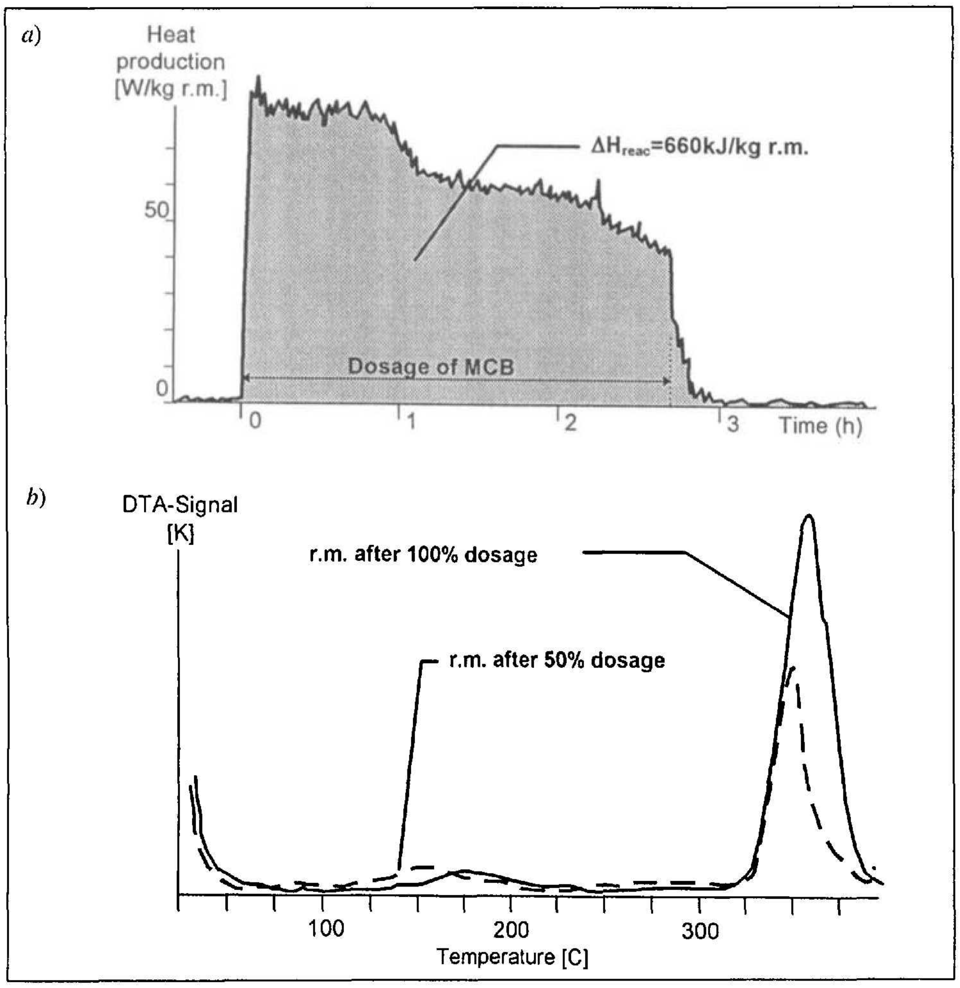

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characterized by the charge-to-mass ratio. The charge-to-mass ratio of insulating powders mainly depends on the operation and the fineness (specific surface area) of the powder and only to a lower degree on the substance of the powder itself and the material of equipment [1]. The latter is, however, essential for the dissipation of charges.

In industrial operations involving high-speed separation processes between particles and wall of equipment such as, *e.g.* in pneumatic conveying, typically a charge-to-mass ratio between 10^{-4} to 10^{-6} C/kg is observed. Operations with a lower speed of separation such as, *e.g.* pouring, may lead to a charge-to-mass ratio in the range of 10^{-7} to 10^{-9} C/kg. It must, however, be emphasized that already a moderate charge build-up on the powder of 10^{-7} C/kg may lead to a possibly hazardous situation in practice.

The electrical field at the surface of a spherical or cylindrical heap of charged powder in a silo or container is proportional to the volume charge density within the heap and to the radius of the sphere or the cylinder. This means that in the case of highly insulating products, discharges must occur from the powder heap to the silo wall even for small silos if the charge-to-mass ratio is high enough, or that discharges must occur for moderately charged product if the silo diameter is large enough. Simple model calculations show that this will happen under conditions prevailing in industrial practice. Thus, the question to be answered is not, whether such discharges will occur at all, but whether these discharges have sufficient energy to ignite the powder.

2. Electrostatic Hazard Assessment – a General Approach

The charge-to-mass ratio is, however, not the only essential quantity. Many other factors exist which are as important in an electrostatic hazard assessment. These factors are in case of the *equipment*: Material of construction (conductivity), connection to earth of all different parts of the equipment, volume of equipment, geometry of equipment, *etc.* In case of the *powder* these factors are: Resistivity of bulked powder, mass flow rate, charging current, particle-size distribution, solvent content, minimum ignition energy, *etc.*

In every assessment of electrostatic hazards, it must be kept in mind that charge build-up is always based on separation processes. In the process industry, these separation processes usually take place between the products and the equipment. Thus, as a result, the equipment as well as the product may become charged to a hazardous level. A systematic approach to an assessment of electrostatic hazards based on these considerations is outlined by the author in [1].

3. Filling of Silos and Containers with Highly Charged Insulating Product

If charge is accumulated on parts of the equipment or on the product itself, a substantial amount of energy is stored. However, the total energy stored in such charge accumulations is not the only criterion important in evaluating the electrostatic hazard. For an electrostatic hazard assessment, the amount of charge released in an individual discharge and its incendivity are of major interest. For this purpose, classifying electrostatic discharges into different discharge types, as described in the English and German literature, codes of practice and guidelines [1–4] provide a practical way of attributing different incendivities to the various discharge types.

Discharges associated with charge accumulated on parts of the equipment or installations have been extensively investigated in the past. Depending on the conductivity and the geometry of charged objects and their surroundings, spark, corona, brush, and propagating brush discharges may occur. Details can be found in [1].

Much less was known so far about discharges associated with charge accumulated on the product (powders and granules) itself. The question whether insulating powder can be ignited by the charge accumulated on the powder itself, even if it is handled and processed in earthed conductive equipment, has been the cause of numerous discussions and speculations in the past.

Before anything was known about discharges along the surface of powder in bulk, the charged dust cloud and the possibility of the occurrence of lightning like discharges within such dust clouds was considered to represent the major electrostatic hazard in silos. This speculation was based on observations during the eruption of volcanoes, where lightning activity has been seen in the ash and dust clouds. However, so far no lightning activity has been reported from industrial scale equipment. In addition, systematic experimental investigations with highly charged dust clouds in a 60-m^3 bunker also showed negative results [5]. Based on this practical and experimental evidence as well as on theoretical considerations, it is nowadays generally agreed that the charge accumulated within highly insulating settled product represents a much more likely ignition source than the charge within the dust cloud.

The phenomenon of discharges along the surface of highly charged bulked polymeric granules, which nowadays is called 'cone discharges', is known since about 15 years. First reports by *Maurer*, and *Blythe* and *Reddish* [6][7] date back to 1979. These discharges are caused by the extremely high electrical fields resulting from the high space charge density which is generated when charged insulating particles are accumulated in silos or containers. Model calculations have shown that conditions necessary for the appearance of cone discharges do exist when filling silos with highly insulating powders or granules. The discharges may already be initiated at a low level of the charge-to-mass ratio of the incoming product (10^{-7} C/kg in the case of a powder heap with a radius of 1 m) [8].

A comprehensive research project on the occurrence and incendivity of cone discharges sponsored by German and Swiss chemical industries and other institutions has been recently completed. The results are published in numerous publications [9–13] and in two final reports [14] [15]. For the investigation of the cone discharges, a test rig with a silo of 50 m^3 volume was set up. Cone discharges could reproducibly be generated in this silo. Special measuring techniques have been developed to characterize the occurrence and the strength of the cone discharges. Successful ignition tests with gases and powders have been performed. Based on these results, an equation for an estimation of the upper limit of the equivalent discharge energy of cone discharges as a function of the silo diameter and of the median of the particle-size distribution of the powder forming the powder heap within the silo can be given:

$$W_{Ae} = 5.22 \cdot D^{3.36} \cdot d^{1.462} \qquad (1)$$

with

W_{Ae}: upper limit of equivalent energy of the cone discharge in mJ,

D: diameter of the silo (metal and earthed) in m,

d: median of the particle-size distribution of the powder forming the powder heap in mm.

Based on the experimental data obtained in the research project mentioned above including data from [16], the limits of application of *Eqn. 1* are the following:

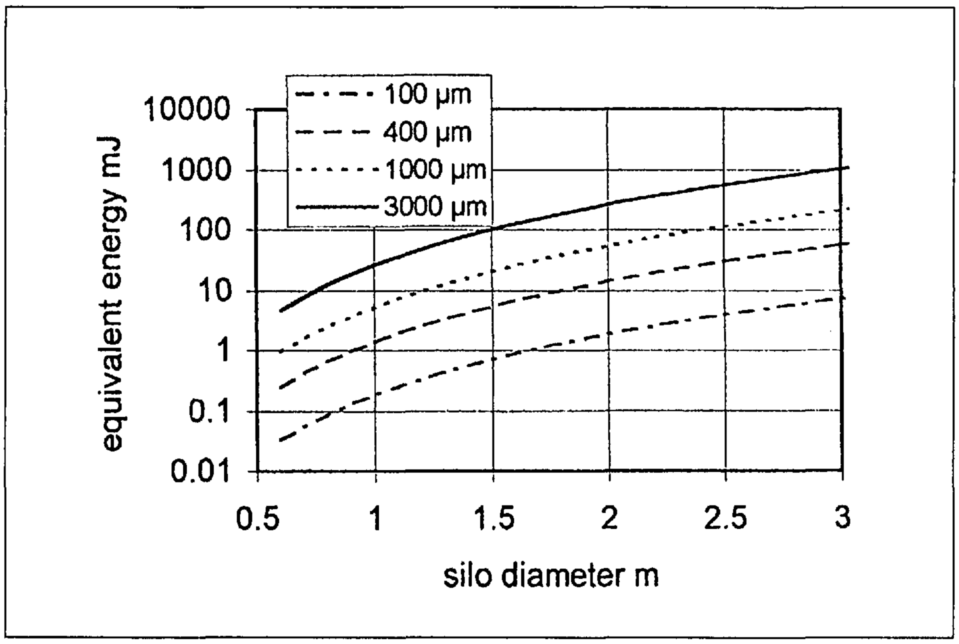

Figure. *Upper limit of the equivalent energy of cone discharges as a function of the silo diameter and the particle size*

$0.5\ \text{m} < D < 3.0\ \text{m}$ and $0.8\ \text{mm} < d < 3.0\ \text{mm}$.

Very recently, measurements could be performed in a production site during normal production. Highly insulating, non-polymeric, fine ($d = 0.1$ mm) product was filled into stainless steel silos by pneumatic transport [17]. The results are in very good agreement with *Eqn. 1*. Therefore, the particle-size limit for the application of *Eqn. 1* can be extended to:

$$0.1\ \text{mm} < d < 3.0\ \text{mm} \qquad (2)$$

This recent results clearly show that cone discharges may also be generated with fine powder and not only with granules, as was previously assumed. Cone discharges from fine powder have, however, much less energy compared to those associated with granules. The *Figure* is derived from *Eqn. 1* and shows the equivalent energy of cone discharges as a function of the silo diameter and the median of the particle size of the product generating the cone discharges. It is now possible to estimate the equivalent energy of cone discharges if the silo diameter and the particle size of the product generating the cone discharges are known, and thus to specify safe operating conditions. However, it is important to know that all values of minimum ignition energy (MIE) referred to in this paper have been determined with the commercially available apparatus Mike III [18] using no additional inductance in the discharge circuit.

Whether cone discharges will occur at all depends on other parameters such as, most importantly, on the resistivity of the bulk powder. Additional parameters are the charge-to-mass ratio, the mass flow rate, and the geometry of the heap and of the silo. In earthed metallic silos cone discharges will hardly occur if the resistivity of the bulk powder is lower than $10^{10}\ \Omega \cdot \text{m}$. In the region between 10^{10} to $10^{12}\ \Omega \cdot \text{m}$, an assessment should be based on model calculations. Above $10^{12}\ \Omega \cdot \text{m}$, cone discharges can hardly be excluded.

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