

Self-Ignition Behaviour of Growing Dust Layers

Bernd Broeckmann, Martin Gosewinkel, René Dworschak

Inburex Consulting GmbH, Hamm, Germany
 Martin.Gosewinkel@Inburex.com

Despite all precautions in the dust-processing industry there are often hazardous situations leading to damage to property and persons. In drying processes self-ignition respectively smoldering fires of dust accumulations are often considered as leading causes for such incidents.

Several well-established experimental test methods exist to analyse the self-ignition behaviour. These procedures have specific advantages and disadvantages, such as testing time, energy consumption and especially their different capability of scale-up from laboratory to industrial scale.

Within the scope of a safety assessment of drying procedures these test methods often lead to a temperature level at which heat production exceeds the natural heat losses of the bulk and lead to spontaneous combustion. For the assessment of deposits in dryers etc. the described methods can be directly applied in the majority of cases, as well as to process conditions, e.g. a dust deposit with a defined thickness at different operating temperatures concerning their thermal hazards (\Rightarrow ignition source: glowing particle).

But one point will be unidentified in this point of view: changes in the physical properties of dust accumulations over time and mainly for non-uniform layer thickness. These changes, however, remain unknown and are mostly ignored as possible source of spontaneous combustion.

These parameters often represent the decisive issue to trigger a run-away reaction, especially in case of spray dryers with variable dust accumulations on the inner wall. To take this effect into account the conventional test procedures were adapted to simulate experimentally the layer increase during the drying process depending on the operating temperature and time.

Initial situation

In addition to spray drying units, also conveyers or milling plants, are often affected by fires or explosions (BIA-Report, 1997). The following figure shows a possible setup of a spray drying unit.

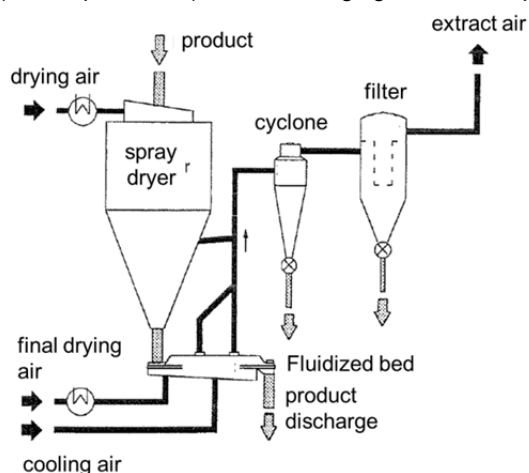


Figure 1: Charting of a spray-dryer-unit (VDI 2263/7, 2010)

There are several hazards that can develop into hazardous situations (Broeckmann et al., 1996).

Firstly, the hazard of developing an explosive atmosphere exists e.g. in the cone of the spray dryer, the subsequent fluidized bed or in the vessels for dust collection (VDI 2263/7, 2010).

Secondly, the possible presence of potential ignition sources has to be kept in mind as hazard.

Typical ignition sources are mechanical sparks, hot surfaces which are induced by friction on the atomizer disc in the spray dryer (Broeckmann et al., 1996) and self-ignition inside the spray dryer.

Results of the self-ignition process are smoldering dust deposits, open flames and fires as well as the development of additional smolder gas (main component carbon

monoxide) (Bartknecht, 1993). Smoldering nests are often located at the dispersion components of the spray dryer such as the atomizer disc (figure 2) and on the inside of the dryer as deposit (Gosewinkel & Meistes, 2010).



Figure 2: Example of a smoldering dust deposit at the atomizer system

Several safety measures are in place to prevent these hazardous situations and to reduce the risk to a minimal level. Among those safeguards are temperature alarms and interlocks, gas analysis devices (measuring the level of CO) (Zockoll, 1992), (Zockoll, 1996), (Gosewinkel & Meistes, 2010) and constructive measures (VDI 2263/7, 2010), (Abott, 1990), (Broeckmann et al. 1996).

1. Ignition source “Self-ignition”

Self-ignition of dust in bulk is caused by the rate of heat generation from oxidation and/ or decomposition reactions of the dust being greater than the rate of heat loss (Bowes, 1984) (DIN EN 15188, 2007).

The self-ignition behavior of combustible dusts depends on their chemical composition as well as on related substance properties (e.g. the size and geometry of the body of material, ambient temperature).

In general self-ignition or spontaneous combustion is limited to solid materials with high specific surfaces. Oxygen can react on the surfaces

throughout the bulk as long as the air exchange is sufficiently high, which has to be assumed for the beginning of a self-ignition process. The oxidation process may start at room temperature depending on the substance.

At the beginning of the self-ignition (better: self-warming) the reaction power is marginal and hardly to detect. If the heat production by oxidation exceeds the natural heat losses of the bulk the self-warming process turns into a self-heating or self-ignition process.

A heat balance, as can be seen in the following picture, involving the heat produced inside the bulk (quantity of reactive surface molecules, gross calorific value) and the heat loss to the surroundings (heat conductivity and dimension of the bulk, heat transfer coefficient on the outside surface of the bulk and the size of the latter) is decisive if

- A steady state temperature is reached at a slightly higher temperature level, that means that the heat loss terms are greater than the heat production terms (see Figure 3, case 1) or
- Temperatures in the bulk will continue to rise up to self-ignition of the dust (see Figure 3, case 2), if heat transport away from the system is insufficient, which results in greater heat production compared to the heat loss.

For a better understanding it is useful to use the so-called Semenov-diagram, which is often used in terms of thermal explosion. The following assumption forms the base for this type of application of the thermal theory

- The physical effects on large deposits and those on laboratory scale are equivalent.
- The chemical reaction is of zero order
- The initial temperature equals the ambient and surface temperature.

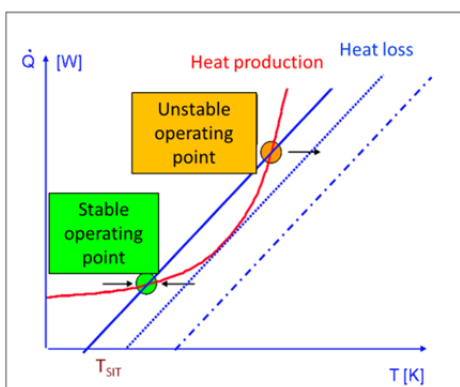
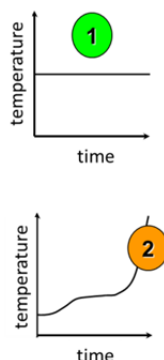


Figure 3: Semenov-diagram

production which is not matched by the rate of heat loss and an accelerating runaway will occur.



The exothermic reaction follows an Arrhenius type rate law, i.e. exponentially dependent, whereas the heat loss is assumed to be governed by Newtonian cooling, i.e. linearly dependent. The rate of heat loss intersects the exponential heat production curve at two points, where the chemical heat production is balanced by the heat removal capacity.

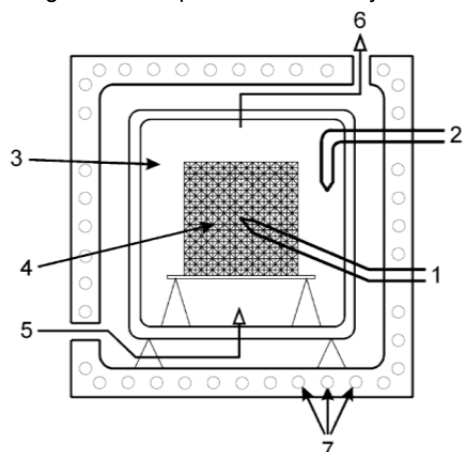
The low temperature point (Figure 3: point 1) represents a stable point. Here the rate of heat loss is greater than the rate of heat production and the temperature will return to this point. The other point (Figure 3: point 2) represents an unstable point because any slight temperature increase will cause an increase in the rate of heat

The tangential point of heat loss and heat production describes the critical situation where the heat production is just equal to the heat removal. The appropriate ambient temperature is equal to the self-ignition temperature T_{SIT} .

2. Experimental determination of the self-ignition

Several well-established experimental test methods exist to evaluate the spontaneous ignition behavior of dusts accumulations, e.g. Grewer-test, Aearated-cell test, Air-over-layer test, test A.16 Guideline 440/2008, hot-storage tests according to EN 15188).

These procedures have specific advantages and disadvantages, such as testing time, energy consumption and especially in their different capability of scale-up from laboratory to industrial scale. The main point of criticism regards to the prediction of safety criteria like safety temperature levels or storage time.



Key

- 1 Thermocouples for measuring dust temperatures
- 2 Thermocouples for measuring ambient temperature (SIT)
- 3 Thermocouples for measuring ambient temperature (SIT)
- 4 Wiremesh cylinder with dust sample
- 5 Air inlet (for natural convection)
- 6 Air outlet (for natural convection)
- 7 Heating elements

Figure 4: Positioning of a sample and thermocouples in the drying chamber for hot storage tests (DIN EN 15188, 2007)

Mostly the investigation of the self-ignition behavior of dusts will be provided by so-called hot storage tests according to (DIN EN 15188, 2007). The sample is kept in a wire basket of cylindrical or cubic form of different volumes in an oven of sufficient volume.

The oven can work with natural convection or air circulation. They shall have an air inlet opening in the lower section and an air outlet opening in the upper section, as shown in the following schematic drawing.

They should have a useful volume of about 120 litres and be controllable in a temperature range from 25 °C to 300 °C. The samples have to be loosely filled into mesh wire baskets of different volumes. The baskets have to be open at the top and closed at the bottom.

They consist of a narrow-meshed wire net, made of stainless steel. Recommended shapes of the mesh wire baskets are that of a cylinder with a height to diameter ration of 1 or that of a cube.

3. Assessment of the self-ignition behaviour and their problems

According to (DIN EN 15188, 2007) the experimental basis for describing the self-ignition behavior of a given dust is the determination of the self-ignition temperature (SIT) of differently-sized bulk volumes of the dust by isothermal hot storage experiments in commercially available drying ovens. The results thus measured reflect

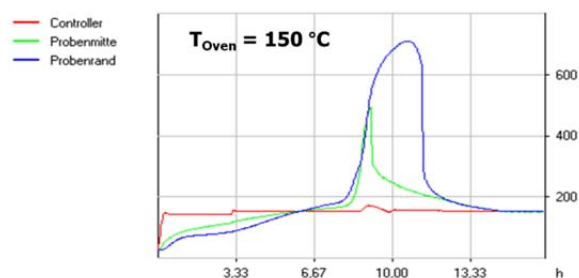


Figure 5: Typical temperature-time-plot of a hot-storage-test according to (DIN EN 15188, 2007);sample: cinnamon, basket volume: 400 ml

the dependence of self-ignition temperatures upon dust volume. The following picture shows a typical temperature-time-plot of such a hot-storage test.

This test result is classified as self-ignition because the sample temperature exceeds the oven temperature of more than 60 K (DIN EN 15188, 2007).

Plotting the logarithms of volume/surface ratios ($\log V/A$) of differently sized dust deposits vs. the reciprocal values of the respective self-ignition temperatures ($1/T$ in K^{-1}) (\Rightarrow Pseudo Arrhenius Plot) produce straight lines, allowing interpolation, to characterize the self-ignition behavior of dust deposits of different scale.

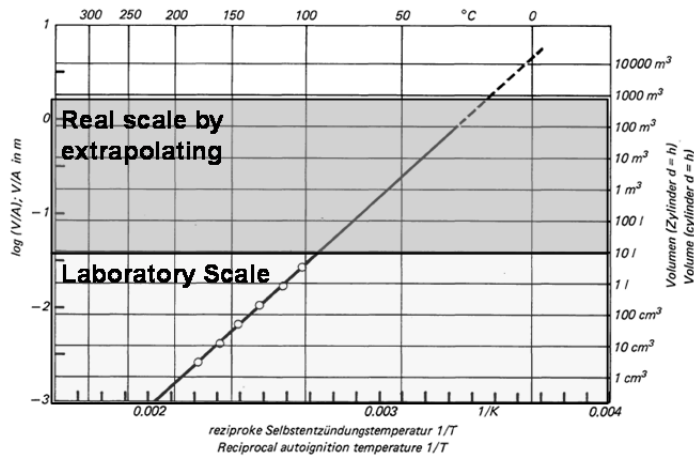


Figure 6: Evaluation of the tests to determine self-ignition behavior of the tested products: Pseudo-Arrhenius plot of self-ignition temperatures (DIN EN 15188:2007)

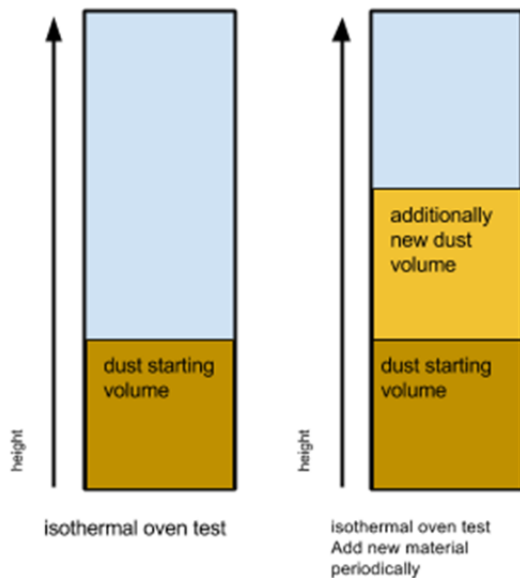
Within the scope of safety assessment of drying procedures the mentioned test methods often lead to a temperature level at defined geometry with defined dimensions (⇒ cube, cylinder, layer thickness....) at which heat production exceeds the natural heat losses of the bulk and lead to spontaneous combustion.

For the assessment of deposits in dryers etc. the results of these methods on self-ignition behavior can be directly applied in the majority of cases, as well as to process conditions, e.g. a dust deposit with a defined thickness at different operating temperatures concerning their thermal hazards (⇒ ignition source: glowing particle).

But one point is not kept into consideration: changes in the physical

properties of dust accumulations over time (glass transition temperature, density, viscosity, thermal conductivity and heat capacity) and mainly for non-uniform layer thickness. These changes, however, remain unknown and are mostly ignored as possible source of spontaneous combustion. But these parameters often represent the decisive issue to trigger a run-away reaction, especially in case of spray dryers with variable dust accumulations on the inner wall. To take this effect into account conventional test procedures were adapted to simulate experimentally the layer increase during the drying process depending on the operating temperature and time. The basic approach of this new method is displayed in Figure 7.

This test is based on the “Air-over-layer”-test according to Abbott (1996). The “Air-over-layer”-test procedure simulates the full scale situation if the powder layer thickness reflects that likely to arise in the dryer, and therefore the temperature from which an exothermic reaction can progress to a self-ignition can be used for defining safe drying procedures. The adapted test procedure should give general information about the layer thickness depending on the drying temperature.



Therefore, a layer with a defined layer thickness of the sample is inserted in the wire basket with a defined edge length and placed into the oven at ambient temperature. The sample is heated up to a specific temperature, normally equal to a typical drying temperature, and held isothermally at this temperature for e.g. 8 hours.

Then a new layer with the same height as the first layer is filled into the wire basket, i.e. the old layer is 8 h old, and now a new layer is brought up to this old layer and is stored at specific temperature for 8 hours. Subsequently, a new layer with the same height as the other ones is brought up to the old layers and should be stored for several hours.

The oven will be continuously purged with a defined air flow rate. The CO-concentration is continuously measured in the outlet air. Before the CO-concentration is measured, the air will be conditioned (cleaned and diluted). Furthermore, the temperature profiles of the sample (centre of the sample layers, sample surface, half width of the sample) and the oven temperature will be continuously recorded.

An additional thermocouple can be placed above the sample to determine potential inflammations.

Figure 7: Basic approach of the adapted test procedures

An additional thermocouple can be placed above the sample to determine potential inflammations.

4. Example of the application of the new test method

In the experiment, the process conditions of a spray dryer for the manufacturing of infant food should be evaluated. The spray dryer is displayed in figure 8.

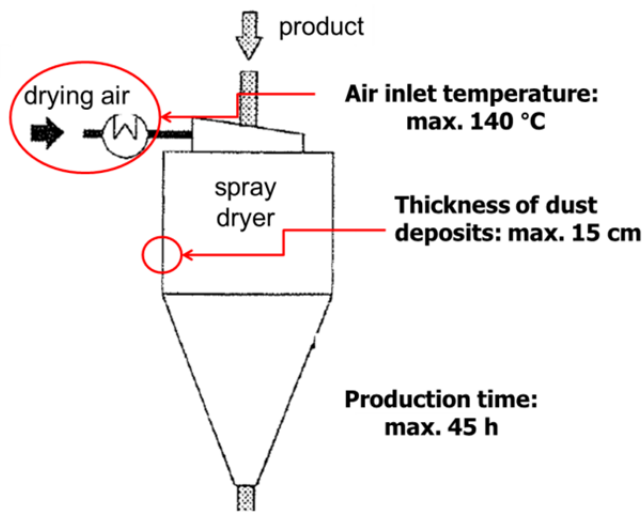


Figure 8: Manufacturing process conditions of children's food

The introduction of new layers can be seen by a sharp decrease of the temperature profiles, which are represented by the upper lines. The CO-detection is described by the line at the bottom of the diagram.

After reaching the oven temperature the first sample layer temperature (layer 0) showed stable behaviour and no significant CO-emission. After bringing up the second layer (layer 1) the temperature of this layer also showed a normal a stable behaviour.

Shortly after bringing up the second layer, that means after a complete testing time of about 30 hours, the temperature profile of the middle layer (layer 1) does sharply decrease because of the cooling effect of the new layer.

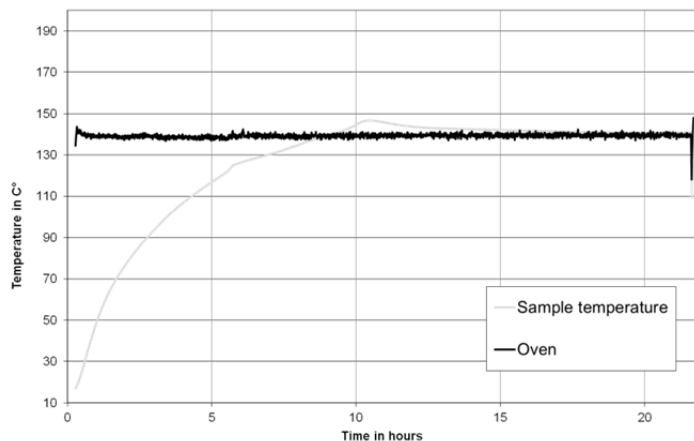


Figure 9: conventional hot storage test according to EN 15188, 15 cm wire basket

But now the middle layer and the new layer are heated up quickly and after a testing time of about 33 hours the sample runs directly in the exothermic reaction.

In contrast to figure 9, figure 10 shows a direct self-ignition process of the sample.

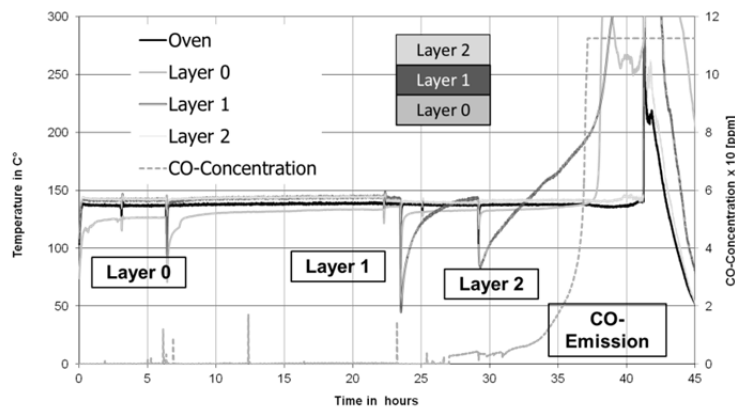


Figure 10: adapted test procedure, 15 cm wire basket

determined as follows after finishing the testing procedures:

- Maximum air inlet temperature: 140 °C
- Maximum thickness of dust deposit in the spray dryer: 10 cm
- Production time: 45 h

5. Conclusions

For the assessment of deposits in dryers etc. the results of the conventional experimental test procedures e.g. regarding CEN 2007, will be directly applied concerning their thermal hazards (\Rightarrow ignition source: glowing particle). But these procedures don't consider changes in the physical properties of dust accumulations over time and for time-dependent layer growth. Therefore these points often remain unknown and disregarded as possible source of spontaneous combustion, but often represent the decisive issue to trigger a run-away reaction, especially in case of spray dryers with variable dust accumulations on the inner wall.

To take this effect into account a conventional test method was adapted to simulate experimentally the layer increase during the drying process depending on the operating temperature and time.

Due to this modified method spontaneous combustion processes could be identified precisely as ignition source in several incidents compared to the results of conventional test procedures (Broeckmann et al., 2014).

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The introduction of a new dust layer (sharp decrease of the temperature profiles) leads to heat accumulation at the interface between "old" (thermal aged) and "new" layer due to a deteriorated heat removal and changes of material properties and so promotes the entire sample in a supercritical state (\Rightarrow self-ignition process).

This behavior can be explained by a change in the thermal properties of the sample during deposition (thermal conductivity, heat capacity) and their direct impact on the heat balance as displayed in figure 3. The process conditions of the future production were finally