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“Validation of formal kinetic parameters from adiabatic heat storage”

Porous bulk materials made of flammable solids often pose the risk of spontaneous ignition. Iso-peribolic hot storage tests are usually performed in line with DIN EN 15188 [1] to define the spontaneous ignition behaviour. However, these tests are very material, labour and time-intensive, because ascertaining the dependency of spontaneous ignition temperatures on the volume/surface ratio and the process of gradually working towards the critical temperature requires numerous individual tests. The results reveal the spontaneous ignition temperatures (SIT) of the tested solid as functions of the volume/surface ratio. Additionally, appropriate evaluation methods can be used to determine the formal kinetic reaction parameters “apparent activation energy” (E_A) and “frequency factor” (k_0).

The spontaneous ignition behaviour of actual bulk materials can either be predicted using a method of empirical extrapolation [1] or through numerical simulation, with the formal kinetic parameters measured in the test serving as the input parameters.

The requisite reaction parameters can also be determined by a single adiabatic hot storage test [2]. In this case, the ambient temperature is adapted to the temperature inside the sample, so that ideally no heat is transferred to or from the sample. However, at present there is insufficient knowledge on the accuracy and reliability of these adiabatic hot storage tests. This applies to both the test procedure and the evaluation methods.

This study shows that even slight deviations in the adiabatic reaction procedure can lead to considerable differences in the formal kinetic reaction parameters.

It presents and compares various evaluation methods and also demonstrates the statistical reliability with which formal kinetic parameters can be determined from a single adiabatic hot storage test.

The adiabatic hot storage tests were carried out for cork powder, brown coal and a PVP polymer in equidistant cylinders with various sample volumes and

start temperatures. The tests showed that the results were not dependent on the start temperature of the oven and the sample volume.

There was good consistency between the reaction parameters determined from the adiabatic hot storage tests and those from the isoperibolic hot storage tests.

The effect that a variation in the adiabatic conditions has on the formal kinetic parameters was examined by adjusting the oven temperature to the sample temperature throughout the test and maintaining a constant difference. As a result, the formal kinetic parameters were either too low or too high, which in turn meant that the SITs determined were also either too low or too high. The latter can lead to risks when determining safe storage conditions (storage geometry, length of storage) for large volumes of bulk materials.

A statistical evaluation of the results showed how reliably the activation energy and frequency factor can be determined from a single adiabatic hot storage test. It showed the deviations that can be expected and the effect that these deviations can have on the SIT and the length of safe storage. A mathematical model developed by Krause *et al.* [3] is used for this purpose. It uses joint differential heat and mass transportation equations to predict the spontaneous ignition behaviour of any bulk material.

Literature

- [1] DIN EN 15188: 2007: Bestimmung des Selbstentzündungsverhaltens von Staubschüttungen, Beuth Verlag, Berlin, 2007
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- [3] Krause, U.; Schmidt, M.; Lohrer, C.; A numerical model to simulate smouldering fires in bulk materials and dust deposits, Journal of Loss Prevention in the Process Industries 19 (2006) pp. 218-226

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