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Ignition temperatures of dust layers and bulk storages in hot environments

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Abstract

In many industrial installations, particulate solids (cereals, agri-food products, coal, plants, etc.) are stored or processed. Self-heating of these products, which can lead to fires and explosions, can occur in a variety of situations. Examples include large storage at room temperature, formation of a layer on a hot surface, layer deposited on a surface – insulating or conductive – in a hot environment or even storage of product exposed to heating on one side.

The main parameters that determine the occurrence of self-heating are the size of the container, the temperature, the residence time and the characteristics of the product. Depending on the type of situation encountered and these implementation conditions, the analysis of self-heating risks must be based on specific models and/or parameters.

This paper presents the different variants and combinations of the theoretical model from the theory of thermal runaway to represent self-heating, taking into account in particular the

symmetry or asymmetry of heating, reagent consumption and boundary conditions. It also discusses their adaptation to the previous identified industrial situations.

Nine products were chosen to be representative of those used in the different considered industrial situations. They were subjected to self-heating basket tests in isothermal ovens in order to determine the parameters for applying the described theoretical models. These results were compared with the results of self-heating tests in layers of different thicknesses in a hot environment, on an insulating or conductive plate, using a specially developed test protocol, as well as with the results of standardized tests of minimum ignition temperature in 5 mm layers.

This led to the proposal of the most appropriate theoretical model to represent the self-heating phenomenon for each of the four identified industrial situations.

This analysis can promote better design of industrial equipment and production conditions (temperatures, volumes or product flows...) in order to prevent fires and explosions.

Keywords: dust layer; bulk material storage; powder; auto-ignition; self-heating; thermal explosion

1. Introduction

Many industrial applications involve the generation, transport or storage of combustible divided solids. The risks associated with these activities are numerous. Health risks can result from exposure by inhalation or skin contact depending of the toxicity of the material. Risks of fouling of installations, fire and finally dust explosion when the particle size is sufficiently fine can also be generated. When warehousing is concerned or in case of dust deposited as layers on the floor, self-heating should also be considered and a thorough risk assessment

should imply the evaluation of the critical operating conditions (temperature, sizes and induction time) leading to a thermal explosion or thermal runaway. Various industrial situations can be identified and do not all require the same quantitative risk assessment approach. In each type of these situations, the safety parameters which have to be determined are specific:

a) The materials can be stored at room temperature, in bulk storage tanks, piles, silos, hoppers or can be accumulated in dust collectors. The materials are then subjected to a symmetrical heating and the self-ignition temperature and critical storage conditions can be determined using the European standard EN 15188 (ECS, 2007). This is the traditional case of grain silos or heaps of coal.

b) Bulk materials can also form layers on equipment with a high surface temperature. In such cases, a plane slab of combustible material is submitted to a unsymmetrical heating in a cold environment and its minimum ignition temperature has to be determined according to an experimental protocol described in the European standard EN 50281-2-1 (ECS, 1999). Such cases are encountered for dust deposits on pump bodies, hot engines or high temperature conveyor belts.

c) Another case can be envisaged when a dust layer is generated on a surface, in a hot environment. In this context, none of the standards mentioned above can accurately represent the thermal explosion phenomenon. Nevertheless, such situations can be practically encountered for deposits on the inner surface of a dryer or near the injection nozzles of an atomization tower.

d) Finally, a large quantity of dust can also be exposed to an unsymmetrical heating. It is the case when a silo or a pile of bulk solids is confronted with a fire directed towards one of its faces.

These four situations show that the variety of industrial cases is not entirely covered by the standards and that the only knowledge of the minimum ignition temperature in layer or of the self-heating temperature is sometimes not sufficient. This gap can be filled by both the development of new experimental protocols and the use of some of the numerous models formerly proposed to describe the self-heating phenomenon (Frank-Kamenetskii, 1938; Semenov, 1928; Thomas and Bowes, 1961).

Thermal explosion theory was first developed by Semenov (1928) and Frank-Kamenetskii (1938). These models are based on simplified heat balances applied to reacting materials at steady-state. Analytical treatments of such equations are usually focused on the calculation of a critical parameter, whose value determine if a steady-state can be reached for a given material, geometry and environment. Two extreme cases were largely commented (Bowes, 1984): Frank-Kamenetskii's formulation, corresponding to a Biot number approaching infinity (large temperature gradient in the material) and to systems governed by internal conductive heat transfer; and Semenov's case, related to very low Biot numbers (homogeneous temperature in the material) and adapted for reactive systems whose thermal conductivity is high compared to the Newtonian cooling (Boddington et al., 1971). Analytical solutions are thus available provided that some assumptions are made. For instance, by assuming a homogeneous reactive medium, its consumption is often neglected and the non-linearity of the heat generation term is overcome by using Frank-Kamenetskii exponential approximation. Depending on the industrial case and the operating conditions, by determining the boundary conditions, the critical parameter can be evaluated for simple geometric configurations (e.g. cube, slab, or cylinder).

However, despite the extent of the models describing thermal explosion phenomenon, their analytical resolution does generally not allow to take the complexity of the industrial situations into account. In addition to these approaches and with the increase of computer

resources, models integrating both heat and mass transfers in porous media and realistic chemical kinetics were developed. For instance, Krause et al. (2006) proposed a review of the numerous attempts made to describe self-ignition phenomenon for real-scale scenarios. They also developed a numerical model simulating smouldering fires of a single fuel by including the transport of seven chemical species and by taking into account the solid fuel decomposition, the char combustion and hydrogen and carbon monoxide reactions. Wu et al. (2017) developed a numerical model to predict the self-ignition temperature and concentration profiles of coal storages. They notably demonstrated that the influence of kinetic parameters was stronger than that of thermal parameters. In the framework of risk assessments in the nuclear industries, Bideau et al. (2011) proposed a model of self-ignition of dust mixtures, including the conductive, convective and radiative heat transfers as well as the chemical reactions of both solid compounds (Dufaud et al., 2014). Nevertheless, the precise knowledge of the physico-chemical properties of the materials layers or storages remains an important limitation. Some previous authors have also used models based on Arrhenius theory to compare results of ignition temperature of layers on a hot surface with results of self-heating basket tests in a hot environment. Bowes et al. (1962) worked principally on the influence of layer thickness, density and particle size of beech sawdust on ignition temperature on a heated surface. These results were modelled using Semenov and Frank-Kamenetskii theories, taking into consideration a zero-order reaction in an unsymmetrical heated slab and reactant consumption. These results were also discussed regarding to experimental basket tests in an oven results, on two materials including beech sawdust. The same comparison, using once again Semenov and Frank-Kamenetskii theories, were performed by Janes et al. (2008), on 14 different materials of various natures involving metal, natural organic, synthesis organic and vegetal.

The innovative contributions of this article concern the extension of the modeling to different variants of the theoretical model from the theory of thermal runaway to represent self-heating, taking into account and combining in particular the symmetry or asymmetry of heating, reagent consumption and boundary conditions. Its interest also lies in the discussion of the adaptation of various models presented to the previous identified industrial situations.

In order to challenge these various approaches, modeling results of self-heating behavior of the materials were compared to experimental results. More specifically, a new specific experimental protocol was adapted to measure self-ignition temperatures of dust layers in hot environments. The critical temperatures obtained for different materials were compared both with self-ignition temperatures determined in isothermal oven according to EN 15188 (ECS, 2007) standard, and with minimum ignition temperature of dust layers in cool environments, as described in EN 50281-2-1 standard (ECS, 1999). Using the kinetics parameters determined from cubic baskets tests and using Frank-Kamenetskii model, analytical solutions were applied to represent the self-heating behavior of the materials under various experimental conditions. Finally, operational guidelines will be proposed in light of the experimental results, and considering the advantages and limitations of the analytical solutions, for practical assessment of industrial self-heating risks.

2. Materials and methods

Three kinds of tests were performed on nine organic products. Tests to determine the self-heating behavior of bulk storages were carried out on baskets of various sizes using EN 15188 protocol (ECS, 2007). From these experiments, the critical operating conditions (i.e. self-ignition temperatures and critical sizes) can be determined and an estimation of the apparent activation energy of the combustion reaction can be derived from Frank-Kamenetskii theory (Bowes, 1984). In a second time, the minimum ignition temperatures in layer (MIT) were

characterized for 5 mm dust layers at ambient temperature as specified in EN 50281-2-1 standard (ECS, 1999). Finally, the self-ignition temperatures of 5 mm, 10 and 15 mm layers were determined for divided materials placed in hot environments. Comparative tests were carried out on a metal plate and on an insulating plate in the case of the 5 mm layer in hot environments. These results were then compared with the MIT in layer measured at ambient temperature in accordance with the international standards. Moreover, results obtained during self-heating tests in isothermal ovens in one hand and for dust layers in the other hand were compared and modelled.

2.1. Description of the tested materials

Nine organic products, often used in industrial dryers, were tested. These materials are presented in Figure 1. It should be underlined that the beechwood powder had already undergone a torrefaction stage before testing, which explains its aspect. Torrefaction is notably used to increase the energy content and density of biomass (Ohliger et al., 2013).

Their particle size distribution was measured either by mechanical sieving, in this case expressed in mass percentage (wt%) or by liquid phase granulometric analysis. In the latter case, the value indicated in Table 1 corresponds to a volume mean diameter. The moisture content was measured by differential weighing before and after drying at 105°C for 30 min or longer, i.e. when the measured mass no longer changed over time (Table 1).



Figure 1: Physical appearances of the various products tested.

#	Material	Particle size (μm)	Moisture content (%)
1	Ground beechwood	24*	4.3
2	Wheat flour	62 wt% > 315	10.3
3	“Lucy” coal	29*	1.4
4	Wood	33 wt% > 315	9.1
5	Alfalfa	100 wt% > 315	7.9
6	Hemp hurds	55 wt% > 315	8.3
7	Cocoa	8*	2.9
8	Pea fibres	55*	7.6
9	Rice husks	100 wt% > 315	9.3

Table 1: Characteristic particle size and moisture content of the tested materials. * Laser diffraction measurements

2.2. Self-ignition temperature of bulk storage

In order to determine the self-ignition behavior of the selected materials, experiments were carried out in isothermal ovens according to EN 15188 (ECS, 2007). The experimental set-up and procedure have already been described by (Janes et al., 2008). Cubic baskets of volume

ranging from 8 to 2744 cm³ were used in this study and at least three different sizes were tested for each temperature. Their critical sizes, or half-edges of the cube, are 1, 2.5, 3.5, 5 and 7 cm. The ignition criterion is validated if the temperature at cube center, i.e. the position of the thermocouple, exceeds the oven temperature by 60 K. In the absence of self-ignition, the test is stopped after 24 hours. The determination of an experimental relationship between the self-ignition temperature and the critical size of the storage allows the application of some models derived from Frank-Kamenetskii theory (see section 3).

2.3. Minimum ignition temperature of a layer

The MIT of a 5 mm dust layer was measured according to the standard method EN 50281-2-1 (ECS, 1999). This parameter is defined as the lowest temperature of a hot surface at which the ignition of a dust layer of a specified thickness occurs. The ignition criterion is validated if, during the test, a visible flaming or glowing is observed, a temperature rise of more than 250 K is reached or if the tested material temperature exceeds 450 °C. If no ignition occurs, the test is stopped after 30 minutes. More details on the apparatus and procedure have notably been provided by (Janes et al., 2008). This technique is useful to determine ignition characteristics of powdered materials (Chin et al., 2016; Turekova et al., 2013) but can also be implemented to estimate thermal and kinetic parameters (El-Sayed et al., 2016; 2017).

According to the IEC 60079-14 international standard, the maximum allowable surface temperature of an equipment installed in 20, 21 and 22 dust hazardous area as defined in the European directive 99/92/CE should be lower than the MIT of the tested material minus 75 K (IEC, 2014). However, it should be stressed that this criterion is only valid for industrial installations at room temperature.

2.4. Self-ignition temperature of a layer

In the case of layers exposed to a hot environment, the previous standard procedures are not relevant. Tests on dust layers were then performed in a 125 L isothermal oven to determine their self-ignition temperature. The material is placed in a metal ring of 100 mm diameter, which is consistent with EN 50281-2-1 standard (ECS, 1999). Both the oven and the layer temperature are recorded. As it is not possible to observe directly the ignition of the material, the ignition criteria chosen for such tests are those based on temperature measurements, i.e. a layer temperature greater than or equal to 450 °C and a temperature increase greater than 250 K, with respect to the oven temperature. As the nature of the supporting plate can impact the self-heating behavior, tests were performed on a conductive metal plate or on a ceramic insulating plate (Figure 2), which will modify the thermal transfers at the system's boundaries.



Figure 2: Insulated oven of 125L (left), ceramic insulating plate (middle) and metal plate (right)

3. Self-heating models

Numerous theoretical models of self-heating systems have been developed since the first approach proposed by (Semenov, 1928). In this article, a specific attention will be given to steady-state models and to their applications to powder layer or storage subjected to symmetrical or unsymmetrical heating.

3.1. Theoretical basis: Frank-Kamenetskii's approach

The classic approach of Frank-Kamenetskii will be briefly described hereafter, as some of its approximations and assumptions will be discussed along this article. A slab of thickness $2r$ is considered, where r is the distance from the symmetry center and the system boundary. The self-heating phenomenon of the slab, exposed to a constant temperature T_A , can be represented by the following differential equation, in the specific case of a steady state:

$$-\lambda \frac{d^2T}{dx^2} = q(T) \quad (1)$$

where λ , T , x and q are respectively the effective thermal conductivity of the medium, the temperature, the distance and the volume rate of heat generation. It is then assumed that the heat generated within the reactive system will be transferred by conduction across the volume. By considering a zero-order reaction and assuming an Arrhenius law, the rate of heat generation per unit volume $q(T)$ is defined by:

$$q(T) = \Delta H \rho A e^{-E_a/RT} \quad (2)$$

where ρ is the system bulk density, ΔH , the enthalpy per unit mass, A , the pre-exponential factor of Arrhenius law, R , the gas constant and E_a , the activation energy. The assumption of an unlimited amount of reactant will be discussed later on.

The resolution of equation (1) was possible by the use of an approximation, proposed by (Frank-Kamenetskii, 1938), who assumed that, provided $RT_A/E_a \ll 1$:

$$-\frac{E_a}{RT} \approx -\frac{E_a}{RT_A} + \frac{E_a}{RT_A^2} (T - T_A) \quad (3)$$

By introducing the following dimensionless parameters:

$$\theta = \frac{E}{RT_A^2}(T - T_A) \quad \text{and} \quad z = \frac{x}{r} \quad (4)$$

And by using the ‘exponential approximation’, equation (1) becomes:

$$\frac{d^2\theta}{dz^2} = -\delta e^\theta \quad (5)$$

where the commonly used Frank-Kamenetskii dimensionless parameter is described as follows:

$$\delta = \frac{E_a}{RT_A^2} \left(\frac{r^2 \Delta H \cdot \rho \cdot A}{\lambda} \right) e^{-E_a/RT} \quad (6)$$

The maximum value of this parameter δ_c will define the limits between the steady-state and the thermal explosion, i.e. for δ greater than δ_c , no steady-state will exist and a thermal explosion will occur in the reactive system.

By rearranging equation (6), the critical parameter δ_c can be linearized as:

$$\ln \left(\frac{T_A^2 \delta_c}{r^2} \right) = M - \frac{N}{T_A} \quad (7)$$

where T_A is here the critical ambient temperature which will lead to the thermal explosion and M and N are defined as:

$$M = \ln \left(\frac{E_a}{R} \frac{\Delta H \cdot \rho \cdot A}{\lambda} \right) \quad \text{and} \quad N = \frac{E_a}{R} \quad (8)$$

Then, by tracing $\ln \left(\frac{T_A^2 \delta_c}{r^2} \right)$ as a function of $1/T_A$ for various sizes of powder piles or deposits, the apparent activation energy E_a can notably be estimated. This method will specifically be used to analyze the results obtained for isothermal tests carried out in baskets.

Various assumptions have been made in developing such a model: an unlimited amount of reactant, a symmetrical heating, no detailed considerations of the boundary conditions and the ‘exponential approximation’. In the following sections, models providing solutions to one or other of these reductive hypotheses will be presented and applied to our experimental cases.

3.2. Symmetrical heating

3.2.1. Symmetrical heating without reactant consumption

If a symmetrical heating is assumed and if the conductive heat transfer at the powder surface is removed by convection and radiation, the boundary conditions can be expressed as follows by using the definition of Biot number Bi for the Newtonian boundary condition:

$$\frac{d\theta}{dz} = 0 \quad \text{at } z = 0 \quad (9)$$

$$\text{and } -\frac{d\theta}{dz} = Bi \theta_s \quad \text{at } z = \pm 1 \quad (10) \quad \text{with } Bi = \frac{h.r}{\lambda}$$

where h is the global heat transfer coefficient. For a given value of Bi , a steady-state can only be achieved if a solution is defined for equation (5), whose general solution is:

$$\theta = \ln(C_2) - 2 \ln \left[\cosh \left(z \sqrt{\frac{\delta C_2}{2}} + C_1 \right) \right] \quad (11)$$

The values of the constants C_1 and C_2 depend on the choice of the boundary conditions applied to the powder. If the previously defined boundary conditions (9) and (10) are applied, the following solution is obtained (Thomas, 1958):

$$\ln(\delta) = \ln \left(\frac{2D^2}{\cosh^2 D} \right) - \frac{2D \tanh D}{Bi} \quad (12)$$

$$\text{where } D = \sqrt{\frac{\delta e^{\theta_0}}{2}} \quad \text{and } \theta_0 = \frac{E}{RT_A^2} (T_0 - T_A) \quad (13)$$

Analytical solutions can be obtained and simple values of δ_c can be deduced as a function of the Biot number. For instance, when Bi tends to zero, then δ_c tends to Bi/e for a slab (Semenov limit), and when Bi increases infinitely, a commonly value of 0.878 is used for δ_c (Bowes, 1984). If the exponential approximation is not used, δ_c reaches a critical value of 0.857, with $Bi = \infty$ and by using the concept of equivalent sphere (Boddington et al., 1971). For a cube, the critical parameter δ_c tends to 2.569 when Bi increases infinitely. Such model can be used to represent piles or storages of powdered fuels at room temperature, but also layers exposed uniformly to a hot environment.

3.2.2. Symmetrical heating with reactant consumption

The previous approach considers that the effect of reactant consumption can be negligible, which is potentially valid for large storages, but leads to significant errors in the case of small powder samples. Barzykin et al. (1964) notably developed the previous analysis by including the effect of the reactant consumption taking into account a dimensionless adiabatic temperature rise called 'B' related to the powder consumption:

$$B = \frac{E_a}{R.T_A^2} \frac{\Delta H.C_0}{\rho.C_p} \quad (14)$$

where C_0 and C_p are the initial fuel concentration and the heat capacity of the reactive system. By introducing $\epsilon = \frac{R.T_A}{E_a}$, they proposed a new formulation of the critical parameter:

$$\delta_c(Bi, B, \epsilon) = \delta_c(\infty) \varphi(Bi) \varphi(B) \varphi(\epsilon) \quad (15)$$

where $\delta_c(\infty)$ is the limit of δ_c when Bi tends to infinity and with the following corrections:

$$\varphi(Bi) = \frac{Bi}{2} (\sqrt{Bi^2 + 4} - Bi) \exp\left(\frac{\sqrt{Bi^2 + 4} - Bi - 2}{Bi}\right) \quad (16)$$

$$\varphi(B) = 1 + \frac{2.4}{B^{2/3}} \quad (17)$$

$$\varphi(\epsilon) = 1 + \epsilon \quad (18)$$

This model, which can be applied to a cube shape (storage) as well as to a slab (powder deposit), is only valid for systems presenting a significant temperature rise (i.e. for $B > 100$) and for $\epsilon < 0.05$ (Bowes, 1984). $\delta_c(\infty)$ is chosen amongst the limits given in subsection 3.2.2.

3.3. Unsymmetrical heating of a slab

Several authors have dealt with self-ignition of an unsymmetrically heated deposit (Clemmow and Huffington, 1956). The model, chosen to represent the ignition of a powder layer with one of its face is exposed to a high temperature whereas the other remains at ambient temperature, was developed by Thomas and Bowes (1961). In order to ensure the validity of Frank-Kamenetskii's approximation, the temperature of the hot face T_p is taken as reference and equation (4) becomes:

$$\theta = \frac{E}{RT_p^2} (T - T_p) \quad (19)$$

Applied at the coolest surface of the powder, the parameter θ_0 can be defined as:

$$\theta_0 = \frac{E}{RT_p^2} (T_A - T_p) \quad (20)$$

The maximum value of δ can then be found by solving the following system of equations (21) and (22) for given values of the Biot number and θ_0 (Thomas and Bowes, 1961):

$$\delta = \frac{P^2}{\sinh^2 P} \exp\left(-\frac{P}{2Bi} - \frac{\theta_0}{2}\right) \left[\cosh P - \cosh\left(\frac{P}{2Bi} + \frac{\theta_0}{2}\right) \right] \quad (21)$$

$$2(1 - P) \left[\cosh P - \cosh\left(\frac{P}{2Bi} + \frac{\theta_0}{2}\right) \right] + \frac{P}{2Bi} \exp\left(-\frac{P}{2Bi} - \frac{\theta_0}{2}\right) + \left(1 - \frac{P}{2Bi}\right) P \sinh P = 0 \quad (22)$$

When δ_c is determined, the approach described in 3.1 can be applied to assess the critical conditions of the thermal explosion. Nevertheless, it should be stressed that the previous model was developed for a hot surface on a perfect thermal conductor, i.e. the case of a powder layer on a metal plate in a hot environment. A peculiar case is found when the temperature of the hot plate is equal to the ambient temperature, i.e. $\theta_0 = 0$. Then, solving the equations (21) and (22) leads to $\delta_c(\infty)$ tending logically to 0.878.

In order to take the reactant consumption into account, it is possible to combine the previous approach, to determine $\delta_c(\infty)$ for a given θ_0 , with Barzykin's model (Barzykin et al., 1964).

A complementary case, consisting of combustible layer in contact with a perfect insulator, was also studied by Thomas and Bowes (1961). It corresponds to the experiment performed with a ceramic insulating plate placed in the isothermal oven. The boundary condition at $z=0$ becomes:

$$\frac{d\theta_0}{dz} = 0 \quad (23)$$

Then, the critical parameter δ_c can be extracted from the following equation (Thomas and Bowes, 1961):

$$\sqrt{2\delta_c} \tanh(\sqrt{2\delta_c}) + 2Bi \ln[\cosh(\sqrt{2\delta_c})] = -Bi \theta_0 \quad (24)$$

The various configurations which will be studied in this article are associated to their model in the Table 2. Letters A to H in Table 2 are used in Figures 3 to 11 to name the results of various modelling approaches.

Contrary to the other models for which T_A corresponds to the ambient temperature, G and H were applied with the parameter T_A corresponding to the oven temperature.

Self-heating model	Symmetrical heating	Cube	With reactant consumption	Finite Biot	δ_c corrected from (Eq.15) by $\delta_c(\infty)=2.569$		
	Symmetrical heating	Cube	No reactant consumption	Finite Biot	As Biot > 10, $\delta_c = 2.569$		
				Infinite Biot	(A) $\delta_c = 2.569$		
		Slab	No reactant consumption	Infinite Biot	(B) $\delta_c = 0.857$ or 0.878		
			With reactant consumption	Finite Biot	(C) δ_c from (Eq. 12)		
	Unsymmetrical heating	Slab	With reactant consumption	Finite Biot	(D) δ_c corrected from (Eq. 15) with $\delta_c(\infty)=0.878$		
			No reactant consumption	Finite Biot	Metal plate	(E) δ_c from (Eq. 21) and Eq. (22)	(G) oven temperature
				Finite Biot	Insulating plate	(H) δ_c from (Eq. 24) Oven temperature	
			With reactant consumption	Finite Biot	Metal plate	(F) δ_c from (Eq. 21) and (Eq. 22) and corrected by (Eq. 15)	
					Insulating plate	δ_c from (Eq. 24) and corrected by (Eq. 15)	

Table 2: Case studies and associated models for the determination of δ_c

4. Experimental results

4.1. Thermal conductivity and Biot number

The thermal conductivity of the powders was determined by using a TCI Thermal Conductivity Analyzer (CTherm). Each thermal conductivity given in Table 3 corresponds to the mean value obtain for three replicates. Unfortunately, due to a lack of product, the measurement of the thermal conductivity of alfalfa was not possible. The Biot number was then calculated, for a 5 mm dust layer (2.5 mm half-thickness), from the following relationship estimating the global heat transfer coefficient h_r :

$$h_r = \frac{4 \varepsilon_1 \varepsilon_2 \sigma T_A^3}{\varepsilon_1 + \varepsilon_2 - \varepsilon_1 \varepsilon_2} \quad (25)$$

where ε_1 and ε_2 are the emissivity of the powder and of the environment (laboratory hood or oven) and σ is the Stefan-Boltzmann constant. An average convection coefficient of $25 \text{ W}\cdot\text{m}^{-2}\cdot\text{K}^{-1}$ was estimated and added to h_r (Bideau et al., 2011). It can be seen that the

approximation of an infinite Biot number is not applicable for a 5 mm thickness layer. However, for a cube of 2.5 cm half-edge, Bi is obviously ten times larger than the values presented in Table 3, which will justify the use of $\delta_c(\infty)$ for such configuration (Thomas, 1958).

Tested Material	Beechwood	Wheat flour	“Lucy” Coal	Wood	Hemp hurds	Cocoa	Pea fibres	Rice husks
λ (W.m ⁻¹ .K ⁻¹)	0.064	0.049	0.071	0.057	0.055	0.071	0.077	0.044
Bi (-) for 2.5 mm	1.13	1.48	1.03	1.28	1.32	1.03	0.95	1.66

Table 3. Thermal conductivity and Biot number for some of the materials tested

4.2. Self-ignition characteristics of bulk material storages

The self-ignition characteristics, i.e. critical temperature and critical size, were determined for at least 3 basket sizes (Table 4). As expected, the self-ignition temperature decreases as the basket size increases, except for the cocoa powder for half-edges from 3.5 to 5 cm. It can be noted that rice husks are less prone to self-ignition whereas “Lucy” coal has the lowest self-ignition temperature, i.e. down to 385K (112°C) for cubes of 10 cm edge.

By using equation (7) and tracing $\ln\left(\frac{T_A^2 \delta_c}{r^2}\right)$ as a function of $1/T_A$, the apparent activation energy of the combustion reaction was estimated for each material with an average error of 5% (Table 4). Given the values of Bi, the critical parameter δ_c was set at 2.569 (Bowes, 1984). The activation energies are consistent with those found in the literature, notably for wood for which an activation energy of 102 kJ.mol⁻¹ was quoted by Bowes (1984). Moreover,

activation energies of 100 to 110 kJ.mol⁻¹ were found for wheat dust (El-Sayed and El-Sayed, 2018) and mean values of 80 kJ.mol⁻¹ were notably obtained for coal in the case of spontaneous combustion (Wang et al., 2009), which is consistent with our results. Obviously, the kinetics parameters related to the combustion of such materials will vary as a function of its rate-limiting step (volatilization, oxygen diffusion...). In the case of lignocellulosic fuels, Branca and Di Blasi (2013) showed that the activation energy related to the char devolatilization step can be estimated at 113 kJ.mol⁻¹, whereas Ea associated with the actual char combustion, is of the order of 183 kJ.mol⁻¹ for each studied product. However, the activation energies associated with the decomposition of hemicellulose, cellulose and lignin varies from 110 to 200 kJ.mol⁻¹, notably for beech, fir and wheat straw.

Basket sizes – Half-edge	Beech- wood	Wheat flour	Lucy coal	Wood	Hemp hurds	Cocoa	Pea fibres	Rice husks	Alfalfa
1 cm	-	495	440	495	480	450	466	530	-
2.5 cm	449	463	405	460	455	420	445	490	-
3.5 cm	432	453	395	450	445	405	439	470	455
5 cm	420	443	385	435	430	405	429	460	445
7 cm	411	433	-	-	-	-	424	-	435
Ea (kJ/mol)	89.8	120.0	88.7	105.2	119.7	103.5	154.6	98.7	121.4

Table 4. Self-ignition temperatures (in K) for various basket sizes and activation energy for the 9 materials tested.

4.3. Minimum ignition temperatures and self-ignition temperatures of dust layers

As described in sections 2.3 and 2.4, the ignition behavior of dust layers, exposed to an unsymmetrical heating over a hot plate or a symmetrical heating in an isothermal oven, was studied. The measured minimum ignition temperatures of dust layers and self-ignition temperatures of the materials are given in Table 5.

Material	MIT (°C)	Self-ignition temperature (°C)			
	5 mm	5 mm	5 mm	10 mm	15 mm
	Hot plate	Metal plate	Insulating plate		
Beechwood	290	220	215	190	-
Wheat flour	350	240	250	225	210
“Lucy” coal	330	210	210	175	165
Wood	330	270	265	235	215
Alfalfa	380	260	270	250	-
Hemp hurds	300	230	240	215	-
Cocoa	260	200	200	185	170
Pea fibre	270	200	215	195	185
Rice husks	> 400*	> 300*	> 300*	> 300*	-

*Table 5: Minimum ignition temperature of dust layers and self-ignition temperature of various thicknesses layers of material tested. * No ignition during test at 300 °C*

It appears that, as expected, the minimum ignition temperature of a 5 mm thick layer is always greater than its self-ignition temperature. The results show that the critical ignition temperature in an oven is 60 to 120 °C lower than the MIT on the hot plate. Such differences

are explained by the fact that the heat generated by the oxidation reaction is more difficult to evacuate when the temperature difference between the material and the environment is low. Table 5 show that materials of high particle size such as alfalfa or wheat dust, forming more heterogeneous layers, are generally characterized by the highest ignition temperatures. This could be related to a lower apparent density and average heat capacity of such beds.

The tests in isothermal ovens were conducted on metal or ceramic plates. For most of the materials, the self-ignition temperature is 10 to 15 °C lower with a conductive plate than with an insulating one (Table 6). However, for products such as wood and beech dust, the critical ignition temperature is lower with an insulating plate. On one hand, the insulating plate hinder the heat transfer from the oven to the bottom of the layer. On the other hand, it could also promote heat accumulation within the layer during the self-heating process. As a consequence, the ignition delay time may be either shortened or lengthened depending on the combustion kinetics and the nature of the plate. By extending these statements, it can also be assumed that products having combustion kinetics driven by a high activation energy will self-ignite at a higher oven temperature when an insulating plate is used (lower average temperature in the layer compared to the oven temperature) rather a metal plate. By following, products having combustion kinetics driven by a low activation energy will ignite more easily with an insulating plate rather than a metal plate. From a practical point of view, it seems that products with activation energies lower than approximately 105 kJ may ignite with an insulating plate at a lower temperature than with a metal plate (Beechwood, "Lucy" coal, wood, cocoa). On the contrary, products with a higher activation energy may ignite at a higher temperature when an insulating plate is used (Wheat flour, Alfalfa, Hemp hurds, Pea fibres).

Material	Self-ignition temperature (in 5 mm layer in oven) difference between metal and insulating plates	Activation Energy
	K	kJ.mol ⁻¹
Beechwood	5	89.8
Wood	5	105.2
“Lucy” coal	0	88.7
Cocoa	0	103.5
Hemp hurds	-10	119.2
Wheat flour	-10	120.0
Alfalfa	-10	121.4
Pea fibres	-15	154.6

Table 6: Comparison between the self-ignition temperature difference between metal and insulating plates and the activation energy

The experimental uncertainties should also be kept in mind, critical temperatures being measured with a 10°C accuracy with the metal plate and 5 °C with the insulating plate. As a consequence, the nature of the plate must therefore be adapted to the industrial situation to be studied. Indeed, an insulating plate will for example be representative of a material stored on the ground, while a metal plate is more representative of a dust accumulating on a dryer wall.

It is also necessary to consider the effect of thermal properties of the supporting plate on the ignition temperature measured. Indeed, the heat capacity of the plate has an influence on the absorption of certain amount of the energy released by the self-heating reaction. This is likely to change, at least, the induction time before the self-ignition phenomenon occurs, and may

also modify the critical self-ignition temperature. This influence, combined to that of the thermal conductivity of the supporting plate, necessarily occurs in a transient regime of establishing a thermal equilibrium on either side of the plate. This is probably always the case for layers of a few millimeters thickness tested in this work. It would be necessary to model these phenomena to highlight the real influence of the thermal properties of the supporting plate on self-ignition temperature results, in order to differentiate it from the influence of the thermal and reactivity characteristics of the tested materials.

Moreover, the self-ignition temperature decreases when the layer thickness increases, which is consistent with the fact that self-heating occurs more easily for great storage dimensions.

Rice husks have higher ignition temperatures than other products, which may be due to their large grain size. It should be noted that MIT of dust layers greater than 400°C were also recorded by Polka et al. (2012) under similar conditions for rice flakes. On the contrary, cocoa powder and pea fibres are more prone to ignition than the other materials. With regard to the two wood powders, the MIT of dust layers and self-ignition temperatures of 'wood' (undetermined species) are significantly higher than those of beech. This difference can be related to a greater particle size but also to greater moisture content. Moreover, wood dust exhibits also a similar trend for similar reasons. Finally, the thermal behavior of coal appears to be particular as its MIT of dust layer is greater than 300 °C whereas its self-ignition temperatures are relatively low in comparison with the other materials. Nevertheless, similar results were quoted by Nagy (1965) for charcoal (MIT of dust layer of 340°C), but lower values, i.e. 233°C, were obtained by Babrauskas (2003) for Pittsburgh coal.

5. Comparison between models and experiments

Figures 3 to 11 present the applications of the models developed in section 3 to the various materials. As previously shown, the characteristics and thermal behavior of these products are greatly different; it is then obviously difficult to define a single model for all the products studied.

First of all, the application of Frank-Kamenetskii's approximation has to be discussed. Indeed, equation (3) is only valid if $RT_A/E_a \ll 1$. With regard to the activation energies given in Table 4 and considering a temperature range of 300 to 650K, the maximum value of RT_A/E_a is 0.06, which remains lower than unity and validates the previous approximation.

For a slab subjected to a symmetrical heating, models (C) and (D), both corresponding to a finite Biot with respectively an infinite or finite amount of reactant, give similar results regardless of the powder nature. However, the application of Thomas' model (Thomas, 1958) leads always to critical dimensions slightly greater than for model (D) - equation (15), but the difference is hardly noticeable on the graphs. The latter model leads usually to the minimum critical dimensions, except for beech (Figure 3) for which the model (H) - unsymmetrical heating over an insulating plate – presents lower values. On the contrary, the maximum critical dimensions are obtained by applying the model corresponding to an unsymmetrical heating of a slab over a metal plate (E).

The minimum ignition temperature (MIT) is often well represented by the previous model (E). However, it is useful to take the reactant consumption into account, i.e. model (F), in order to predict the MIT of beech-wood. For coal, alfalfa and, to a lesser extent, wheat dust, the various models failed to represent the MIT of a 5 mm layer. Indeed, they tend to be conservative and underestimate the critical dimensions for which a thermal ignition will occur. Models (G) and (C) give rather similar results, which can be explained by the fact that a layer placed on a metal plate in an isothermal oven is nearly subjected to a symmetrical heating (low θ_0), due to the high thermal conductivity of the metal support on which the layer

rests. It should be stressed that the behavior of the layer placed on an insulating plate is always well represented by model (H).

In the case of unsymmetrical models, it can be noticed that the difference ($T_p - T_A$) between the temperature of the hot face and the temperature of the hot environment (Eq. 20) is directly correlated to the thermal properties of the powder (Eq. 14) for most of the materials studied (Figure 12). The specific behavior of cocoa is underlined, which can be due to its low melting point and porosity of the dust layer.

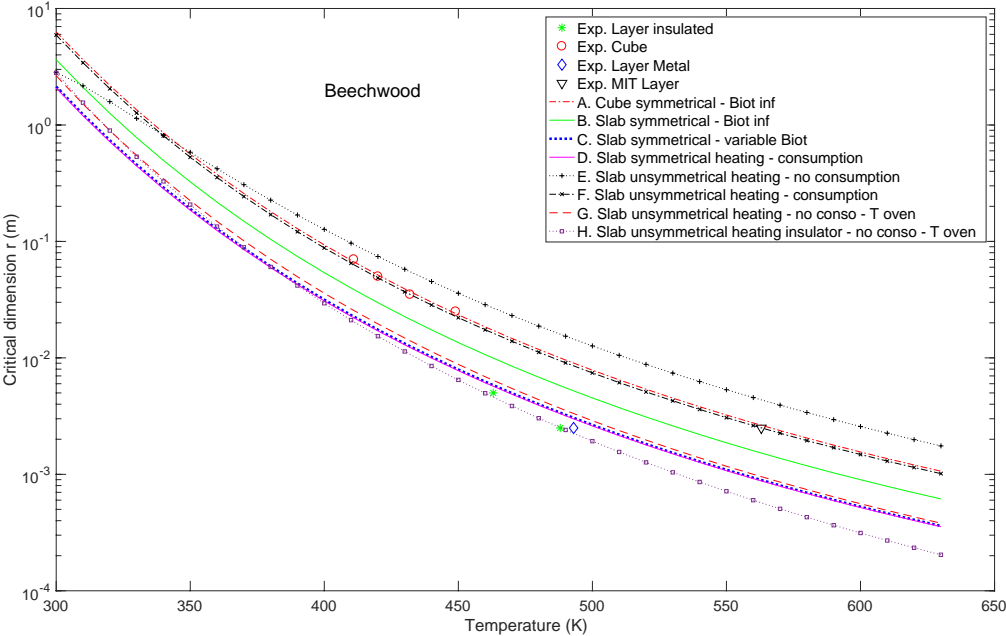


Figure 3: Temperature dependence of the critical dimension of ground beechwood layers and storages: comparison between experimental results and various models

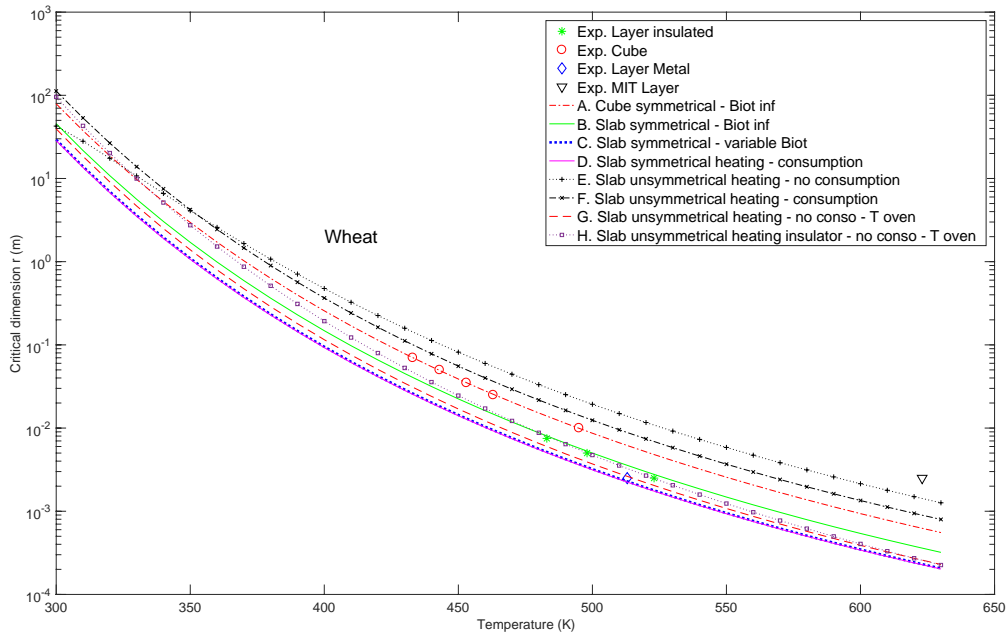


Figure 4: Temperature dependence of the critical dimension of wheat flour layers and storages: comparison between experimental results and various models

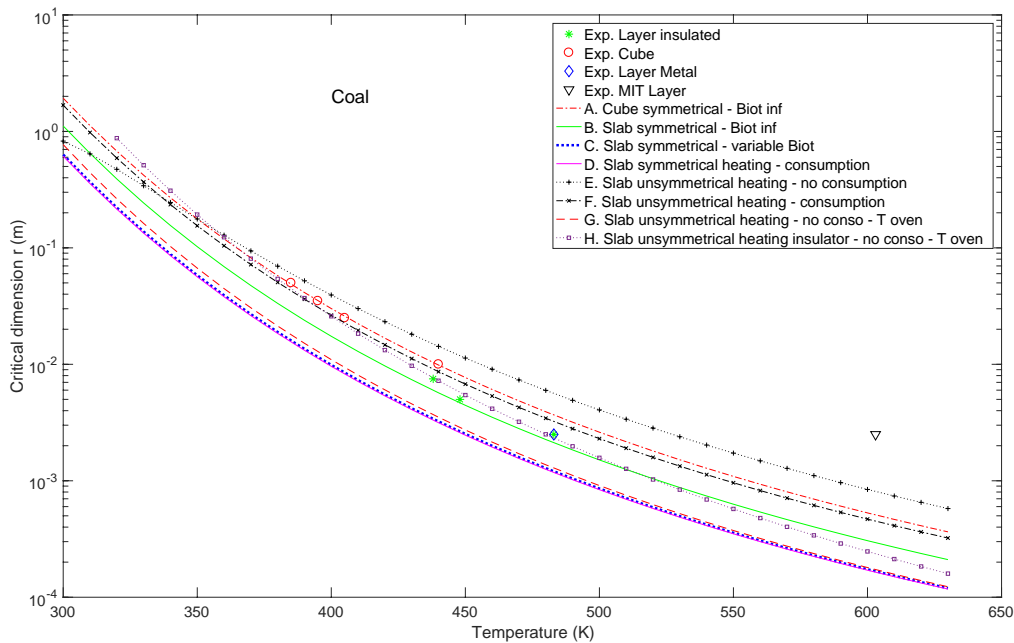


Figure 5: Temperature dependence of the critical dimension of "Lucy" coal layers and storages: comparison between experimental results and various models

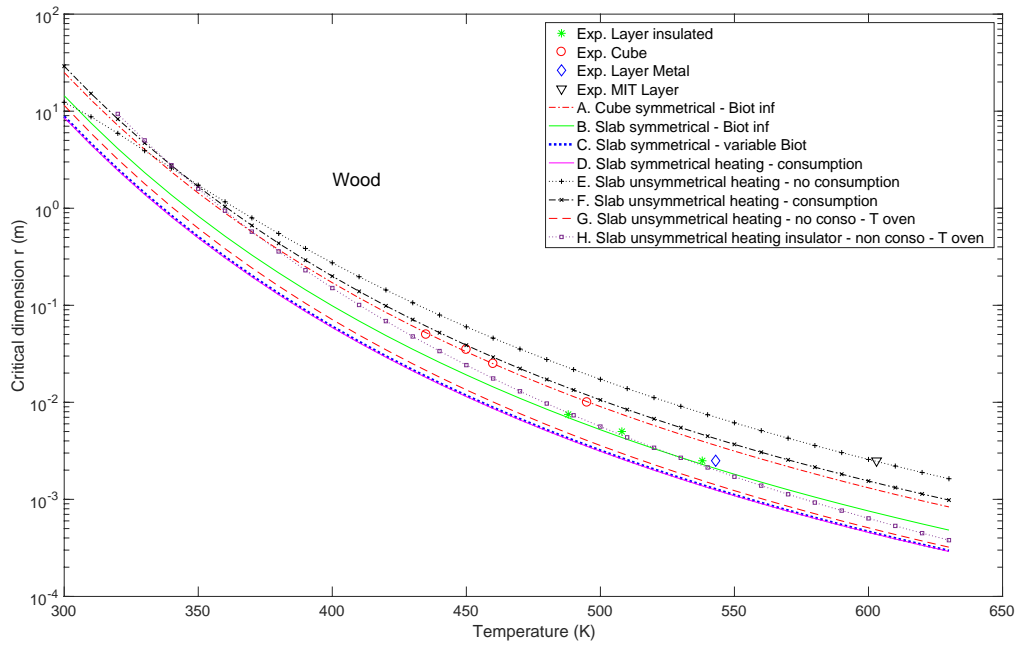


Figure 6: Temperature dependence of the critical dimension of wood layers and storages: comparison between experimental results and various models

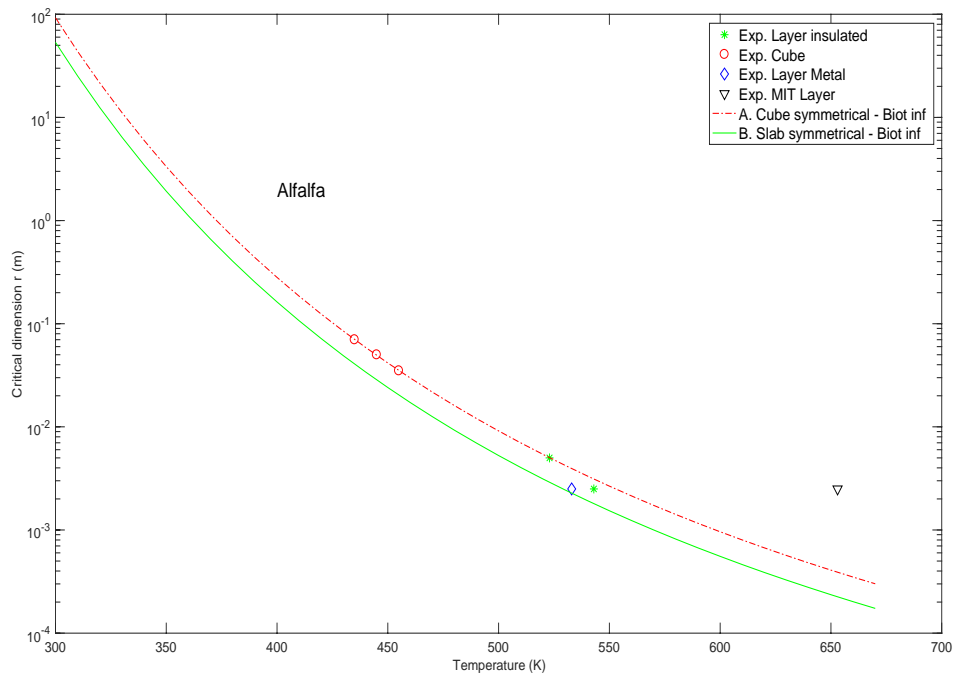


Figure 7: Temperature dependence of the critical dimension of alfalfa layers and storages: comparison between experimental results and various models

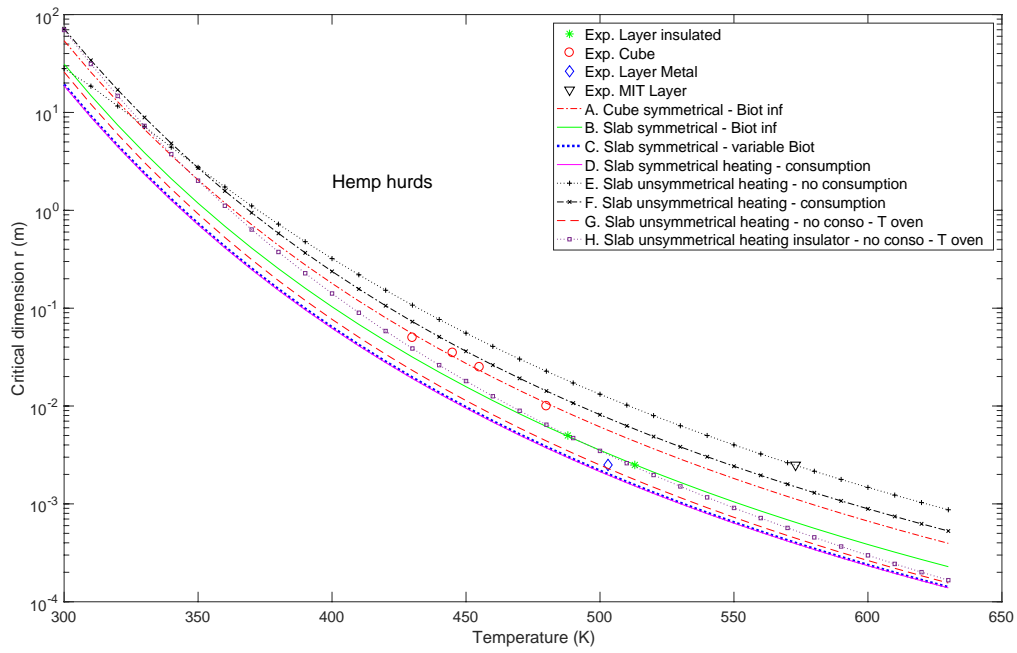


Figure 8: Temperature dependence of the critical dimension of hemp hurds layers and storages: comparison between experimental results and various models

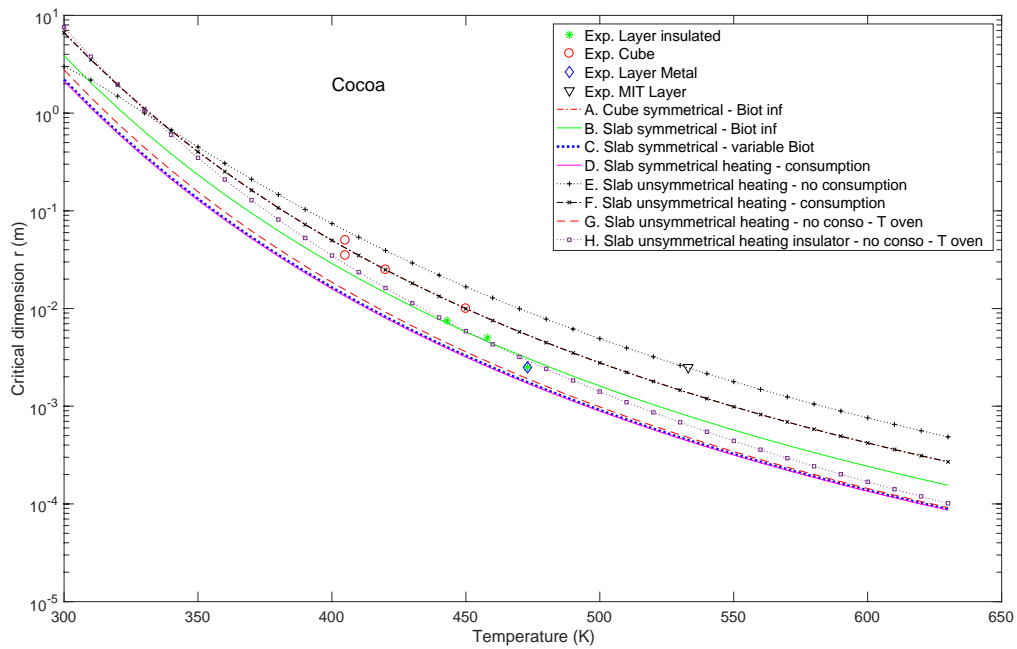


Figure 9: Temperature dependence of the critical dimension of cocoa layers and storages: comparison between experimental results and various models

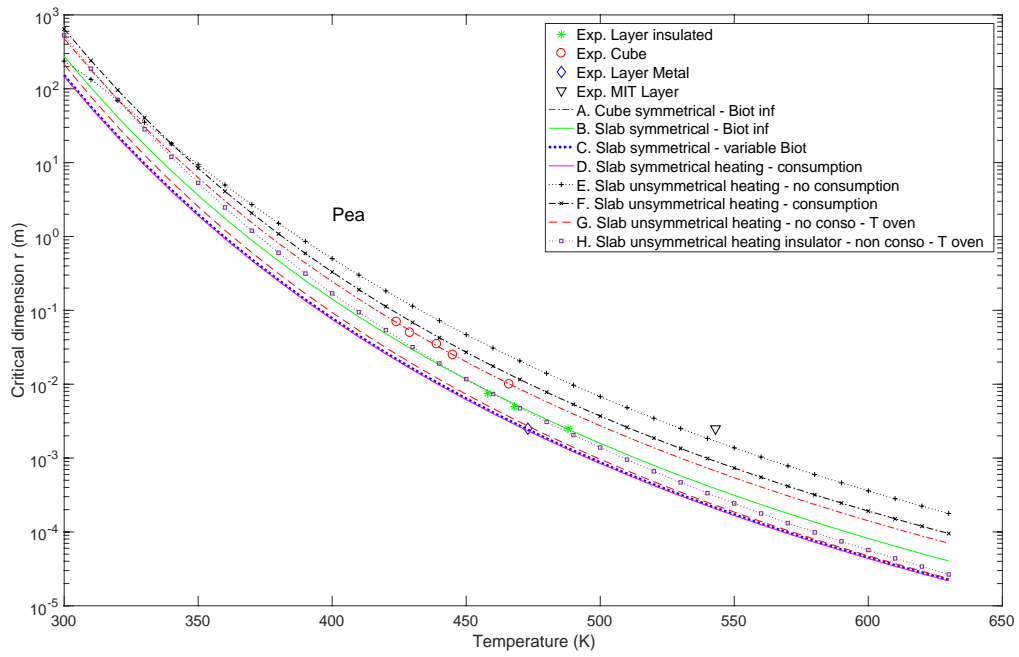


Figure 10: Temperature dependence of the critical dimension of pea fibres layers and storages: comparison between experimental results and various models

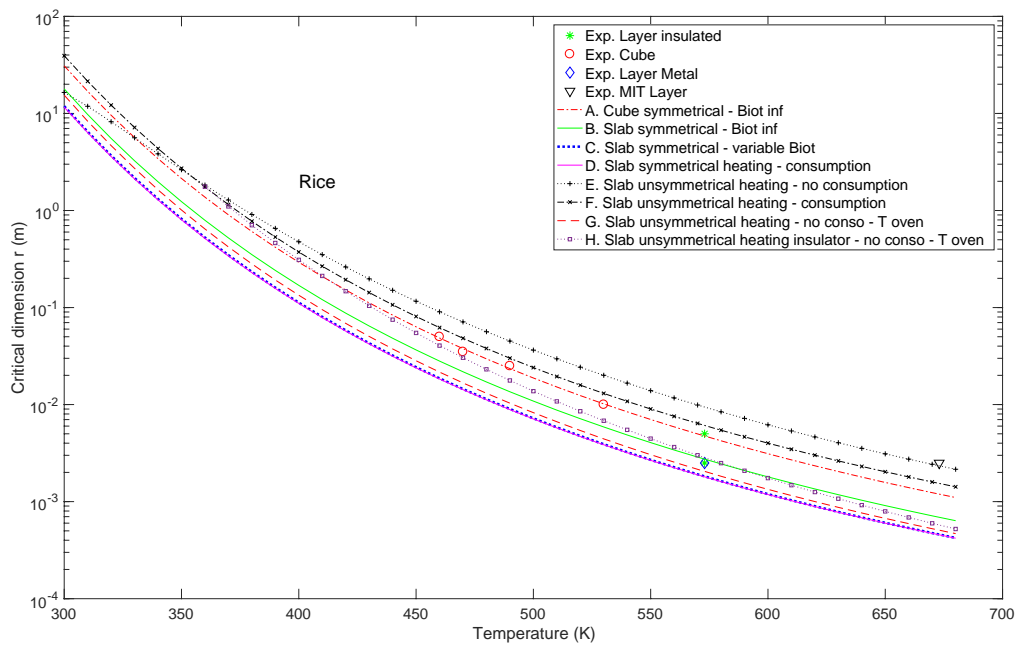


Figure 11: Temperature dependence of the critical dimension of rice husks layers and storages: comparison between experimental results and various models

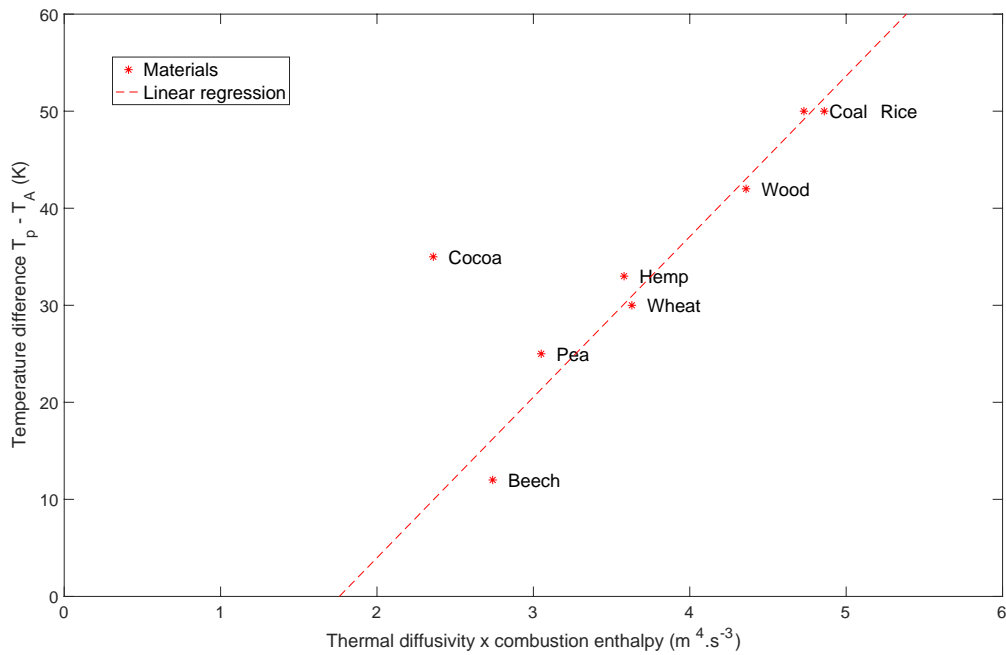


Figure 12: Correlation between the temperature difference between the temperature of the hot face and the ambient temperature and the thermal properties of the powder

6. Operational guidelines for self-heating prevention in industrial processes

In the case of a layer deposited on a hot surface, the confrontation between models and experiments shows that the model (E), corresponding to an unsymmetrical heating of a slab over a metal plate, could be representative but does not describe all situations, especially for wheat flour and “Lucy” coal. It is why the experimental protocol described in EN 50281-2-1 (ECS, 1999) is recommended to determine the minimum ignition temperature (MIT) of a 5 mm layer of a tested material. The application of this standard promotes the prevention of ignition sources, by limitation of maximum surface temperature of electrical apparatuses used in presence of combustible dust. It is important to note that, if the predictable thickness of the dust layer on the apparatus is more than 5 mm, it is possible to determine the MIT of a 12.5 or a 15 mm dust layer, as a function of envisaged thickness. Nevertheless, in this case, the best preventive measure is to regularly remove dust deposits, to limit their thickness as low as

possible. The industrial configurations concerned are for example dust deposits on high temperature equipment as conveyor belts or hydraulic presses, or on hot devices like pumps or engines.

The storage of bulk material at room temperature in storage tanks, silos, piles, hoppers... or the accumulation of bulk materials in dust collectors systems are well modelled by the application of Frank-Kamenetskii theory (A), as showed by this study. This model is representative of a symmetrical heating without reactant consumption and assuming the “exponential approximation”. In these industrial situations, it is recommended to use the experimental protocol prescribed in EN 15188 standard (ECS, 2007) using cubic or cylindrical baskets, as described.

When a bulk material is deposited as a layer on an insulating surface in a hot environment, the comparison between experimental results and modelling indicates that the best correlation is obtained for the model (H), representative of unsymmetrical heating over an insulating plate. If the plate is thermally conductive, such as a metallic plate, the more appropriate correlation is difficult to deduce from Figures 3 to 11, due to experimental uncertainties. Experimental results show that the difference between the self-ignition temperature on the metallic plate and on the insulated plate seems to be related to the activation energy. For most of the material tested, the self-ignition temperature on the metallic plate is 10 to 15 K lower than that obtained on an insulating one. It could be noted that except in the case of beechwood, experimental self-heating temperatures are framed for the minimum values by model (D) – slab symmetrical heating with reactant consumption – and for the maximum values by model (E) – slab unsymmetrical heating without reactant consumption. These industrial configurations correspond to bulk material accumulated on the inner surface of a dryer or near the injection nozzles of an atomization tower, for which the surface is most of the time metallic. As an example, spray drying is often used as an encapsulation technique, notably by

the food industry, for instance in the production of amorphous starch powder (Niazi, 2012). A substance to be encapsulated (the load) and an amphipathic carrier (usually some sort of modified starch) are homogenized as a suspension in water (the slurry). Then, after dispersion on this solution in the spray dryer, a co-current or a counter current heated air flow is blown into the equipment. If a heap of dried material sticks to the wall inside the equipment, near the injection point of the solution, it will gradually thicken until it is cleaned. If the temperature of the drying air is too high, this amount of powder heats itself up and becomes able to ignite the explosive powder/air atmosphere that is formed in the spray drier. It is therefore particularly important to adjust the temperature of the drying air on the basis of the self-ignition temperature of a layer of realistic thickness in a hot environment and on a metal supporting plate, measured or calculated using basket test in oven results. As an example, for wheat flour the self-ignition temperature of a 5 mm layer on a metal plate was measured at 240 °C. The dimensioning of the drying temperature on the basis of the standard minimum ignition test, which corresponds to 350 °C for the same wheat flour in this work, can lead to an explosion.

An insulating surface can be encountered for example in the case of the material layer on a belt dryer, or for a layer deposited on the soil submitted to a hot environment. Finally, the heating of a wall (conductive or insulating) of storage by an external heat flow such as a fire also corresponds to these types of situation. In practice, to model the cases of layers deposited on a surface in a hot environment, different possibilities could be considered:

- Using of a specific experimental protocol based on the EN 15188 standard, with layers of specified thickness on conductive or insulating plate, as a function of industrial situation to be considered.
- Modelling the material behavior using the model (H), representative of unsymmetrical heating over an insulating plate, in the case of an insulating plate and if essential parameters needed are known, such as the temperature of the hot surface.

- Modelling the lower bound of the material behavior using the model (D), representative of slab symmetrical heating with reactant consumption, in the case of a conductive plate and if essential parameters needed are known, such as the heat capacity or combustion heat of the powder.
- Applying the experimental protocol of EN 15188 standard, using cubic or cylindrical baskets as described to obtain experimental self-heating temperatures, and during the modelling step, replacing the δ_c parameter by those defined for a slab. A safety margin of about 20 K should be subtracted from the calculated self-heating temperatures, as shown by the confrontation between results and modelling from this study.

7. Conclusions

Risk analysis related to self-heating phenomena of dust layers or bulk storages is essential and therefore, the determination of self-ignition and minimum ignition temperatures is compulsory. However, if the use of standardized tests is necessary, their operating conditions must always be compared with actual industrial conditions. In particular, this study urges to proscribe the use of the minimum ignition temperature of a dust layer obtained by application of EN 50281-2-1 standard to represent the behavior of layers deposited on a surface in a hot environment. This can lead to misconceptions and thus to fires and dust explosions. The use of a specific experimental procedure leading to the determination of the self-ignition temperature of a powder layer is then recommended. Thomas and Bowes' model (1961) appears suitable to represent such industrial situations. More generally, several models can be applied to represent the self-ignition behavior of bulk storages or dust layers under various heating conditions; however, their application greatly depends on the knowledge of the

powder thermal properties. As a consequence, standardized experiments applied to industrial powders appear to be the best solution to assess the self-ignition risk, but protocols still need to be developed.

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Nomenclature

A	pre-exponential factor of Arrhenius law (-)
B	dimensionless adiabatic temperature rise (-)
Bi	Biot number (-)
C_1, C_2 and D	constants used for modelling the symmetrical heating without reactant consumption
C_0	initial fuel concentration (kg.m^{-3})
C_p	heat capacity ($\text{J.kg}^{-1}.\text{K}^{-1}$)
E_a	activation energy (J.mol^{-1})
h_r	global heat transfer coefficient ($\text{W.m}^{-2}.\text{K}^{-1}$)
P	$\sqrt{2\delta e^{\theta_m}}$ where θ_m is the maximum value of θ
R	gas constant ($\text{J.K}^{-1}.\text{mol}^{-1}$)
T	temperature (K)
T_A	ambient temperature (K)
T_p	temperature of the hot face
q	volume rate of heat generation (W.m^{-3})
r	distance for the symmetry center and the system boundary (m)
x	distance (m)
z	dimensionless parameter (-)
δ	Frank-Kamenetskii dimensionless parameter (-)
δ_c	Frank-Kamenetskii critical dimensionless parameter (-)
$\delta_c(\infty)$	limit of δ_c when Bi tends to infinity (-)

ΔH	enthalpy per unit mass (J.kg^{-1})
ε_1	emissivity of the powder (-)
ε_2	emissivity of the environment (-)
ϵ, φ	parameters used to model the symmetrical heating with reactant consumption
λ	thermal conductivity ($\text{W.m}^{-1}.\text{K}^{-1}$)
ρ	system bulk density (kg.m^{-3})
σ	Stefan-Boltzmann constant ($\text{W.m}^{-2}.\text{K}^{-4}$)
θ	dimensionless parameter (-)
θ_0	dimensionless parameter at the boundary condition $z = 0$ (-)