Full Length Article

Onset of smoldering fires in storage silos: Susceptibility to design, scenario, and material parameters

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ABSTRACT

Biomass fuels in large storage units are prone to self-heating and ignition causing smoldering fires. Here, the susceptibility of such ignition processes to parameters is explored through small-scale experiments. In a silo geometry, wood pellets samples of size 0.75 to 1.5 kg were heated from below to initiate smoldering, while the top was open, allowing convective exchange of gases between the porous sample and the surroundings. The thermally insulated sidewalls reduce the heat flow in lateral direction in a similar way that additional pellets material would do in a larger set-up. Thus, the present experimental set-up mimics a much larger system in lateral direction. After heating was terminated, the procedure led to self-sustaining smoldering or spontaneous cooling, depending on parameters.

The transition zone between smoldering and non-smoldering was explored under variation in sample size, imposed heating, pellets type, and height of sample container. Logistic regression was applied to fit the experimental data to a model. The model predicted the probability of an experiment to result in either smoldering or non-smoldering under variation in parameters – and the parameters were sorted according to importance. The duration of the external heating was found to be the most influential parameter. For risk assessments in connection with large biomass fuel storage units, this result indicates that the temperature increase could be more important than the size and geometry of the storage unit and the stored material type.

1. Introduction

Solar energy may be harvested using a number of different technologies, each with advantages and disadvantages. Trees and plants collect solar energy as biomass: Even though the efficiency (stored bioenergy to incident solar energy) is low, usually below 1%, the production phase is technologically simple, and large areas may be exploited. Furthermore, a range of products may be obtained based on biomass: from various fuels to a number of chemicals, see, e.g., [1–4]. However, there are technological and economical challenges connected to transportation, production, and storage.

Whatever the final product, storage time is unavoidable both for semi-finished and finished biomass products. Storage usually occurs in large units, typically silos, which makes sense from an economical and practical perspective. However, when stored in large quantities, biomass products are prone to self-heating, spontaneous temperature increase that potentially lead to smoldering fires [5]. Whether such a

temperature increase will level out or increase till a smoldering fire is started depends on a number of parameters, most importantly the size of the stored material. There are calculation schemes where the maximal size for temperature stability (critical condition) can be determined [5]. From a practical point of view, one needs to keep the stored amount below this critical size – or, alternatively, keep storage times sufficiently short (typically, below 3 months for large silos) [6].

Smoldering fires evolve slowly, are flameless, occur at low temperatures, and are difficult to detect and to extinguish [7,8]. The oxidation occurs directly at the surface of a solid fuel and yields large amounts of toxic gases and volatiles compared with flaming fires [8]. A smoldering fire may reach a balance between heat production and heat losses to the surroundings. Under such conditions, the smoldering fire will last for hours, days, or months, and the fire is referred to as self-sustaining. The slow evolution of the smoldering process leads to fires that remain undetected, while the comparatively high concentrations of carbon monoxide cause a number of fire deaths every year [7]. Large

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greenhouse gas emissions are produced by smoldering fire in wildland and elsewhere [7–9].

A common biomass fuel used for heating of buildings is wood pellets. Wood pellets and similar biomass fuels have been reported to self-ignite and smolder during storage, handling and transportation [6]. Biomass fuels are often stored in silos, and a significant number of silo fires have been caused by self-ignition [6,10]. One example is the silo fire in Esbjerg, Denmark, in 1998 – 1999 [6]. The fire lasted for nine months before it was extinguished, with losses of 8 million euro [6]. In other cases, smoldering fires have evolved to flaming fires and even to gas explosions, causing large damages to the facilities [11,12]. Smoldering fires produce combustible gases, which may mix with air and ignite. Gas explosions initiated by smoldering fires occurred for instance in Stavanger, Norway in 1985 and Tomylovo, Russia in 1988–1989 [13]. The gas explosion in Stavanger was triggered by self-heating of rape-seed pellets stored in a silo. A week prior to the explosion, flames were observed in the silo. Attempts to extinguish the smoldering fire using foam were unsuccessful. In Tomylovo, Russia, several gas explosions occurred in a facility storing various products [13].

The accidents described above make obvious the risks connected to self-ignition and smoldering in wood pellets. There are, however, two main challenges. First, direct experimental study of initiation of smoldering fires through self-heating is impracticable, due to the significant time scales involved. In a direct experimental approach, one would need to follow the spontaneous temperature build-up in a large pile, one the other hand, a number of indirect approaches exists, like hot-storage tests. Second, both the initiation and the subsequent evolution and propagation of smoldering fires are influence by a series of factors connected to the fuel material as such, its surroundings (container, etc), and characteristics of the internal or imposed heating [5]. Among important material parameters are moisture content, porosity and particle size [6]. With high moisture content, spontaneous combustion may be preceded by exothermic bio-chemical reactions in the material. The permeability of a material and geometrical boundary conditions affect the supply of oxygen to the smoldering zone, and variations could enhance or suppress the smoldering process [13]. Hagen et al. [14] found that higher density in cotton samples resulted in reduced temperature for initiation of self-sustaining smoldering fires. Similarly, the heat flux scenario also influences the ignition temperature of cotton [15].

Sample size is another factor that affects the occurrence of smoldering, e.g., in dusts layers [5]. The thickness of a dust layer has an impact on the hot-plate ignition temperature: as the thickness increases, the ignition temperature decreases systematically [16]. Palmer [17] found that the minimum depth necessary to obtain self-sustaining smoldering is dependent on dust type and particle size. Hadden et al. [18] investigated the effect of sample size on thermal radiant ignition of smoldering in polyurethane foam, where the heat flux necessary to induce smoldering was found to decrease with increasing sample size. Krause and Schmidt [10] ignited dust deposits, by embedding glowing nests into the dust samples. The glowing nests need to have a critical dimension for dust to be ignited. The critical dimension depends on dust type and sample size. Larsson et al. conducted small and medium scale (1 m³) experiments and found that both energy production and gas emissions are dependent on pellet type [19,20]. Pauner et al. [21] predicted the critical ambient temperature and storage size for wood pellets and protein powders using a small-scale basket test.

In this contribution, we study experimentally initiation of self-sustaining smoldering in a (small-scale) silo geometry, for which we have previously reported results on smoldering under imposed cooling [22,23], smoldering in wood-based thermal insulation materials [24], and effects of varied air flows through the sample [25]. We do not use slow, homogeneous heating of the sample (with temperatures at all positions approximately equal at any point in time) to reach self-sustaining smoldering, but fast, inhomogeneous heating. We compare our results to the case of homogeneous, slow heating in Section 4.3.

Parameters associated with both sample and external conditions have been varied: sample size, pellets type, height of sample holder, and external heating. For each parameter combination, the temperature necessary to initiate self-sustaining smoldering has been determined. Obviously, the values found for this transition temperature reflect our choice of imposed, external heating. However, our focus is on how the determined temperatures *correlate* with the mentioned parameters, not the obtained temperature values as such. Our analysis reveals *the relative importance* of the parameters.

In Section 2 the experimental set-up and properties of the wood pellets will be described. The results are presented in Section 3, followed by discussion and statistical analysis in Section 4. The conclusion follows in Section 5.

2. Experimental set-up and procedure

2.1. Materials

Wood pellets from two different manufacturers were used in these experiments. The wood pellets (Class 1) were fabricated according to Norwegian (NS 3165) [26] and Swedish (SS 187120) [27] standards and the material properties are presented in Table 1.

2.2. Experimental set-up

The experimental set-up is shown in Fig. 1. An insulated steel pipe with an inner diameter of 15 cm was used to contain the wood pellets sample. Two different pipe heights were used in the experiments, a 33 cm pipe (referred to as "low pipe"), and pipes of 58 to 63 cm height (referred to as "high pipe"). The latter type consisted of two low pipes stacked on top of each other, the variation in heights was due to the way the pipes were stacked. The pipe was positioned vertically on top

Table 1

Material properties for the wood pellets and methods used.

Material properties	Pellet A (Norwegian)	Pellet B (Swedish)
Type of material	Wood (including bark) 20–50% pine 50–80% spruce	Wood 60% pine 40% spruce
Pellet diameter [mm]	8	8
Single-pellet density [kg/m³]	1020	1050
Bulk density [kg/m³]	710	730
Porosity [vol%]	30.4	30.3
Higher caloric value [kJ/kg][a]	18 834	17 453
Lower caloric value [kJ/kg][b]	17 433	15 931
Activation energy [kJ/mol][c]	91.4	107.1
Permeability [m²][d]	$\leq 2.4 \cdot 10^{-8}$	$\leq 2.1 \cdot 10^{-8}$
Moisture content [wt%]*[e]	**6.3** (6.8)	**7.7** (9.2)
Volatile compounds [wt%]*[f]	77 (82)	78 (85)
Ash content [wt%]*[f]	**0.46** (0.49)	**0.21** (0.23)
Elemental composition [wt%]*[g]		
Carbon (C)	**48** (48) *[52]*	**48** (47) *[52]*
Hydrogen (H)	**6** (6) *[6]*	**6** (6) *[7]*
Nitrogen (N)	**0** (0) *[0]*	**0** (0) *[0]*
Oxygen (O) & Others	**39** (39) *[42]*	**38** (37) *[41]*

*) Multi-valued material parameters are given for three conditions, denoted through boldface, (), and *[ ]* as follows: **As received** (Air dried) *[Water and ash free]*.
a) Determined using bomb calorimetry.
b) Determined using bomb calorimetry.
c) Determined using an adiabatic test [28].
d) Determined using a constant flow rate of air in a self-made experimental set-up with 0.16 m diameter and 0.6 m length.
e) Determined using Thermogravimetric Analyzer and Moisture Analyzer.
f) Determined using Thermogravimetric Analyzer. Defined as the amount of the material which undergoes pyrolysis and primary oxidation to form char.
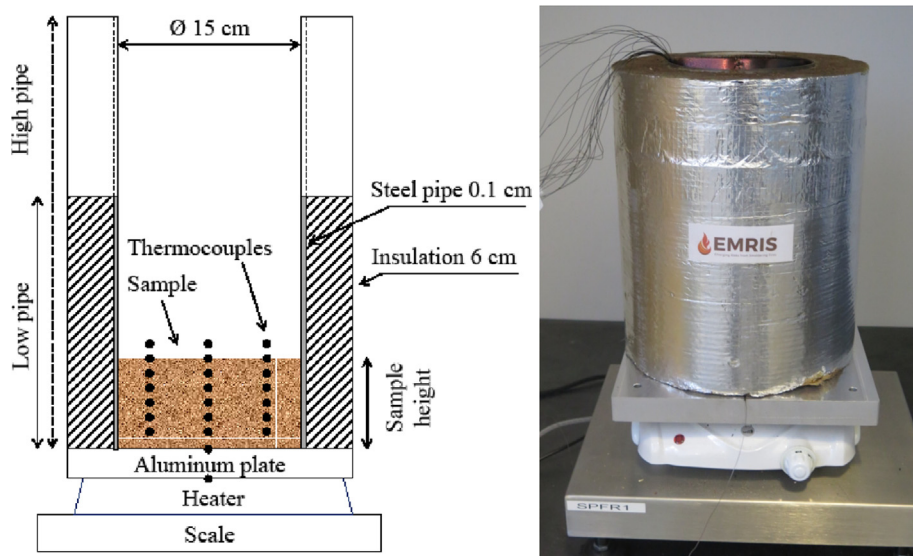g) Determined using Thermogravimetric Analyzer and Carbon/Hydrogen/Nitrogen determinator.

Fig. 1. Experimental set-up: sketch (left, not to scale) and photo of a low pipe (right). Thermocouple positions (●), differences in pipe sizes and sample height are indicated in the figure. See main text for further details.

of an aluminum plate. The sidewall of the pipe was insulated with 6 cm of insulating material with conductivity 0.085 W/(m·K). The heat loss through the sidewall of the pipe during the external heating period was estimated to less than 32 W for a temperature of 300 °C in a 12 cm high sample. The sample was exposed to ambient air at the top, but not at the bottom, where the aluminum plate blocked air exchange. Four sample heights (see Fig. 1) were studied: 6, 8, 10 and 12 cm.

The wood pellets were heated from below using a 2 kW electrical heater placed under the aluminum plate below the sample. The aluminum plate was used to obtain an even temperature across the surface in contact with the wood pellets. The temperature between the aluminum plate and the electrical heater was regulated in an on–off mode using a digital thermostat, with set-point temperature 370 °C. For each run, the heating rate was nearly constant from 2 h after start, but varied from 500 to 1000 W between runs. The temperature at the top of the aluminum plate (referred to as height 0 cm later in this article) stabilized around 360 °C during heating periods. The temperature on top of the aluminum plate was lower than the reference temperature due to heat losses through the aluminum plate and the sample. Alternatively, the temperature on top of the aluminum plate could have been used as set-point temperature, but preliminary experiments showed that this alternative led to larger temperature variations on top of the aluminum plate than the current ± 3 K.

Temperatures were measured using 0.5 mm encapsulated type-K thermocouples (the diameter given includes the outer shield). The thermocouples were positioned below and on top of the aluminum plate, as well as at selected positions inside and above the sample, as marked with filled circles in Fig. 1. The thermocouples were placed at levels with vertical spacing 2 cm, with three thermocouples at each level, one at the center and two at distance 3.75 cm from the center. A small ladder-like stainless-steel structure was used to keep the thermocouples in position as the sample height decreased due to mass consumption during an experiment. The components described above were placed on top of a scale. Mass and temperature values were recorded every 5 s.

2.3. Procedure

The scale was placed on a horizontal surface, the electrical heater with the aluminum plate were placed on the scale, and the insulated pipe was placed on top of the aluminum plate. The thermocouples and the wood pellets were then placed inside the pipe. Data acquisition was

started two minutes before the electrical heater was switched on. By design, the lower part of the sample was heated first, and it was necessary to pre-define a target condition for the lower part of the sample. The sample was heated from ambient temperature to the selected target condition. The target condition was fixed using a predetermined reference temperature, called the cut-off temperature The cut-off temperature was considered reached when two of three thermocouples located 2 cm above the aluminum plate had reached this temperature (see Section 2.4). The electrical heater was then disconnected. Different cut-off temperatures were selected to obtain at least two experiments for each sample height, one with self-sustaining smoldering and one merely with weak smoldering that readily self-extinguish (from now on referred to as non-smoldering).

An experiment was terminated when all temperatures inside the sample were below 50 °C. Experiments with and without self-sustaining smoldering were easily distinguished based on visual observations, and mass and temperature measurements (see Section 3.1). The fuel residue after each experiment was sorted into five categories according to color and appearance (see Ref. [29] for details).

2.4. Experimental method

The criterion for terminating the imposed heating of the sample, see Section 2.3, was chosen to obtain similar conditions in the lower part of the sample for a given cut-off temperature and for different sample sizes and pipe heights. Using this method, the onset of self-sustaining smoldering could be correlated to the typical temperatures (cut-off temperature) in the lower part of the sample. The time to reach a certain temperature varied with sample size. Even though it might take longer to heat a large sample to a given cut-off temperature compared with smaller samples, the temperatures in the hot lower part of the sample (close to the aluminum plate) would be the same at the time the external heater was switched off. Such a systematic approach is advantageous since the way a sample is heated as a whole, affects the onset of smoldering combustion [30].

The sample height affected the time to reach the cut-off temperature, therefore the total heat input to the sample varied between experiments. The measurement points at 2 cm did not necessarily coincide with the hottest regions. This is somewhat surprising, but may be understood as a consequence of asymmetric heating of the sample (along the horizontal direction). Different heating duration for samples of equal size may result from such an asymmetry (as further discussed in
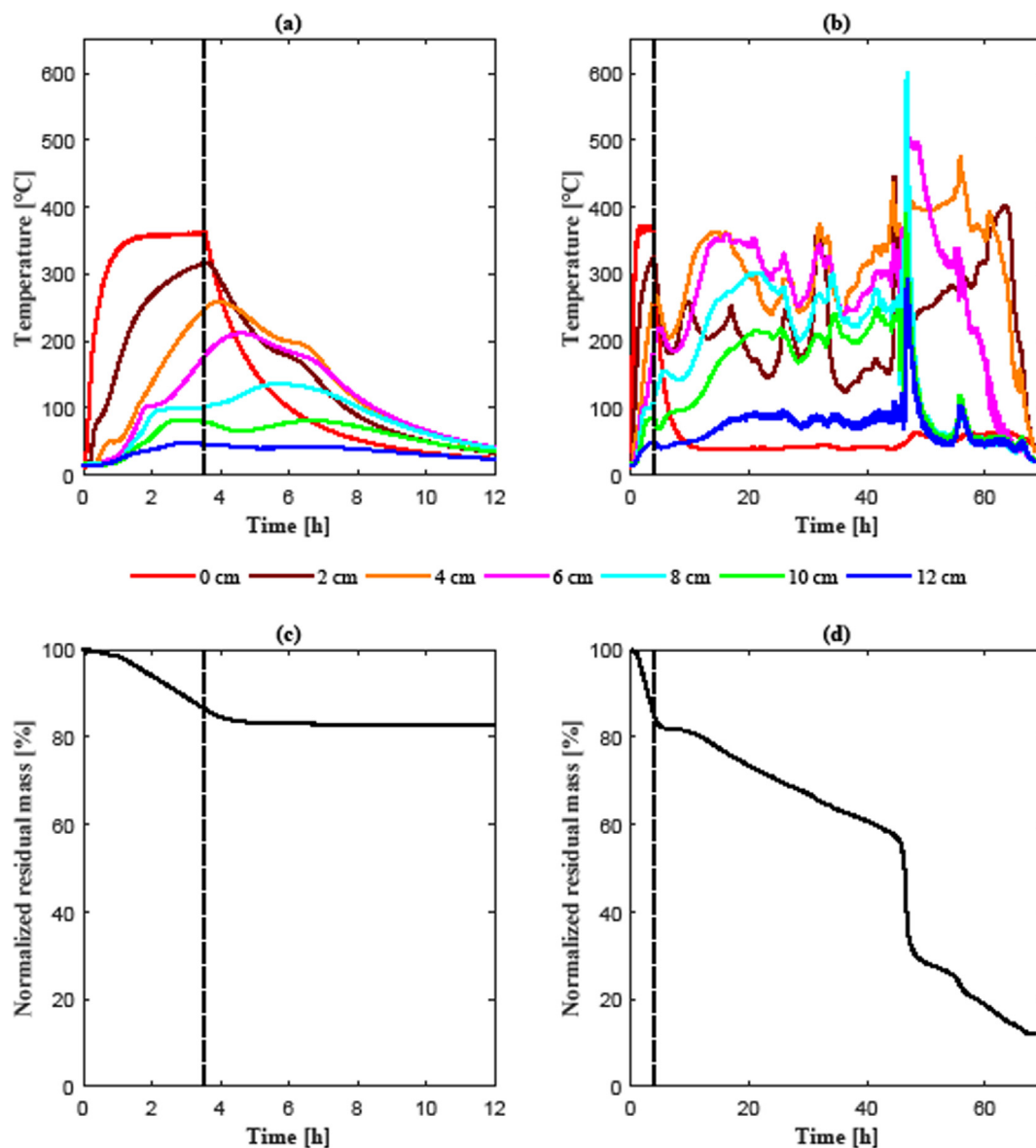
Fig. 2. Temperature and normalized residual mass as function of time. Left (parts a and c): Non-smoldering experiment with wood pellets type A, sample height 12 cm (1.484 kg), cut-off temperature 310 °C, total duration 12 h. Right (parts b and d): Smoldering experiment with wood pellets type A, sample height 12 cm (1.484 kg), cut-off temperature 320 °C, total duration 70 h. Parts a and b: Temperatures along the centerline of the sample as a function of time. Parts c and d: Normalized sample mass as function of time. Vertical dashed lines indicate the time the external heating was switched off. Only temperatures at the center of the sample, at different heights, are displayed in parts a and b (for clarity). The complete data sets contain three temperature measurements at each height level, as illustrated in Fig. 1 and Fig. 6. In part c, the mass loss was 14% during external heating, and 3% during the subsequent cooling. In part d, the mass loss was 14% during external heating and 74% during the self-sustaining smoldering.

Section 4.2). In connection with this set-up-specific mechanism, one may add that while there in general are many theories where the time to thermal runaway can be calculated, their predictions differ [5].

3. Results

3.1. Temperature and mass loss

Typical temperatures and sample mass profiles as function of time are shown in Fig. 2. Non-smoldering behavior is strikingly different compared with smoldering behavior (left vs right side of Fig. 2). The examples shown in this section are representative for both types of pellets used in the experiments.

Fig. 2a and c shows a representative non-smoldering experiment, where the sample cooled down towards ambient temperature - after the

external heater had been switched off at 3.5 h, indicated by the vertical dashed line. As shown in Fig. 2c, only a small fraction of the mass, approximately 17%, was lost during the heating and the cooling process. The cooling process is slow, compared with the heating process, and the experiment lasted 12 h. 6–9% of the mass loss is due to evaporation of moisture (see Table 1). The remaining 8–11% is an irreversible mass loss due to decomposition of the wood pellets. The decomposition can be visually observed for 20–45% of the pellets in a sample as permanent discoloration (black or brown pellets, see Ref. [29]).

Fig. 2b and d shows a representative evolution of temperatures and mass during a smoldering experiment. After the initial heating (3.8 h), indicated by the vertical dashed line, the sample cooled down. Approximately 1.8 h later, there was a significant increase in the sample temperature, which indicates self-sustaining smoldering, with a heat
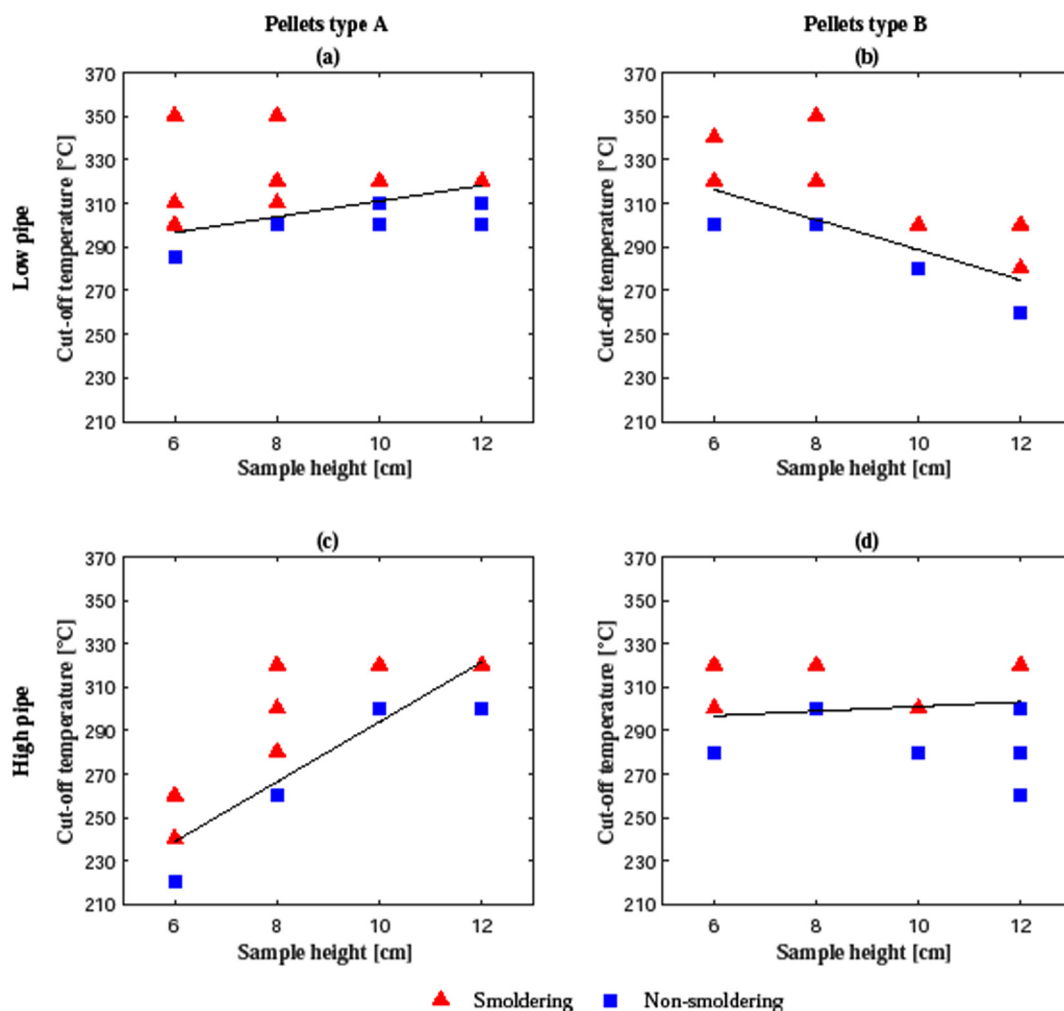
Fig. 3. The occurrence of smoldering and non-smoldering experiments as cut-off temperature and sample height were varied for two different types of wood pellets (columns) and two different pipe heights (rows).

generation from internal processes that was larger than the heat loss to the surroundings. Relatively high temperatures were observed over a significant time period (55 h, see Fig. 2b). The sample did not cool down until most of the mass was consumed, as shown in Fig. 2d. Characteristic features of the smoldering experiment in Fig. 2b are high temperatures, with repeated periods of erratically increasing and decreasing temperatures. Similar behavior was reported by Lohrer, Krause and Steinbach [31], where fast reactions consume the oxygen inside the sample and the reaction rate decreases. Then, fresh oxygen migrates into the sample and the reaction rate increases again. A smoldering experiment may last for many hours; the experiment shown in Fig. 2b and d lasted nearly 66 h after the external heater had been turned off.

The strikingly different evolution in the two experiments shown in Fig. 2 arise as a consequence of an apparently small change in the imposed external heating, with only a 10 °C increase in cut-off temperature.

Prior to the experiments, it was assumed that the heat transfer upwards along the pipe would be one- dimensional (1D). However, it was observed that the three temperatures measured at a given height inside the sample differed both during and after the external heating period. In some instances, the highest temperature was recorded in the center of the sample, while in others the highest temperature was between the center and the pipe wall. During the external-heating period, the temperature variation at a given height was no more than 5–10 K between center and positions 3.75 cm from the center. On the other hand, during self-sustaining smoldering, the temperature difference at a certain

height could be as large as 50 K. The asymmetric temperature distributions suggest that the heat transfer through the sample was not purely one-dimensional.

Further information on the smoldering process can be extracted from Fig. 2b. After the external heater was switched off, the samples cooled down over a significant period before most of the temperatures again increased. The temperature increase occurred without any changes in external conditions. In Fig. 2b, the temperatures 2 cm above the aluminum plate decreased for 1.8 h after the external heater was switched off and then increased again. This indicates a smoldering process in the lower part of the sample (0 – 2 cm). The fact that there was a group of measurement points where the temperatures increased in a correlated way indicates an active heat-producing zone that spreads through the sample. The highest temperatures, with intense combustion (44.7–48.6 h), led to a significant mass loss where the mass decreased from 57% to 30% of the initial value. This corresponds to a rate of 1.7 g/min, which is much higher than the average mass loss rate of 0.3 g/min over the duration of the experiment.

As explained above, our procedure in most cases led to a period of spontaneous cooling after the external heating unit had been switched off. Only after that cooling period, the sample displayed either smoldering (increasing temperatures) or continued cooling (decreasing temperatures). Thus, the sample (in some of the cases) evolve into smoldering from a very weak starting condition, which could be called a *marginal condition*, resulting from *weak forcing*.

3.2. Visual changes during experiments

During the very early phases of a smoldering experiment, there were no visible indications of smoldering at the top layer of the sample, and no smoke was observed. As the temperatures in the sample increased during external heating, smoke was observed together with some discoloration of the pellets grains at the top of the sample. In some cases, single grains expanded. As the experiment continued, the height of the sample decreased, and the pellets on the top became increasingly discolored. After the peak temperatures (at around 48 h in Fig. 2b and d), the pellets on top of the sample had become predominantly black and the height and the mass of the sample were significantly reduced. For the remainder of the experiment, the visual appearance of the sample did not change. After the experiments, the sample consisted of black pellets and gray ash.

For the non-smoldering cases, some smoke was observed during the external heating of the sample. The top of the sample had discoloration and expanded grains in some cases, and in others appeared unaffected. After the end of the experiment, the sample contained both unaffected and discolored pellets, but no ash. More details on residue composition can be found in reference [29].

3.3. Onset of smoldering

Experiments were carried out with several sample heights (sample sizes), several cut-off temperatures, two pipe heights (see Section 2.2), and two different types of wood pellets (see Table 1). A total of 47 experiments were carried out. The occurrence of smoldering and non-smoldering in terms of these parameters is summarized in Fig. 3. Smoldering occurred when the lower part of the sample reached a sufficiently high cut-off temperature during the initial external heating period. The transition region from non-smoldering to smoldering was found using logistic regression (see Section 4.1). The p50-line (solid line) denotes an estimated 50% probability that an experiment will result in smoldering for a given combination of sample height and cut-off temperature.

The same experimental data set as in Fig. 3 is replotted in Fig. 4 with cut-off temperatures replaced by the total duration of the external heating period, which is the time to reach the cut-off temperature. Smoldering occurs more easily with longer duration of external heating and smaller sample size. When these two parameters are taken into account, pellets type and pipe height do not improve the differentiation between smoldering and non-smoldering (see further explanation in Section 4.1). The p50 lines shown in Fig. 4 were therefore obtained by fitting simultaneously all data (from all the four figure parts). In Fig. 3, on the other hand, all four parameters were taken into account when producing the fitted lines and only the data corresponding to each figure part was included in the fit – leading to slopes that are markedly different[1].

Assuming that heat propagates uniformly from the hot aluminum plate and upwards through the sample and that the heat loss through the side of the sample can be ignored, all iso-temperature surfaces should be horizontal planes at all times. One would then expect duration to be a unique function of cut-off temperature. The differences between Figs. 3 and 4 indicate that such a simplified assumption cannot be made. This issue is further discussed in Section 4.2. Figs. 3 and 4 display the obtained data with the variation in pellets type and pipe height in separate plots. A more comprehensive data analysis is given in Section 4.1 below, with all data points included simultaneously.

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[1] Note that there is not a sufficient number of experiments to warrant that all four parameters are significant – the different slopes of the separation lines could be a spurious effect. See further explanation in Section 4.1.

3.4. Smoldering velocity

The vertical velocity, with which the smoldering reaction front moves upwards, can only be estimated indirectly. The smoldering propagation is most likely influenced by the degree of asymmetry in the heat transfer during the early phases of the experiment (see Section 3.1). Smoldering velocities were estimated by considering the temperatures after the external heater was switched off (after the vertical line in Fig. 2). The first minimum (turnaround point) in a set of temperature curves occurring after the electrical heater was switched off was used as a reference, assuming that the corresponding thermocouple was located near the starting point of the self-sustaining smoldering. The vertical velocity was determined using the vertical distance between two thermocouples and the time interval between occurrences of the reference temperature. The smoldering velocities were in the range 8 to 16 mm/hrs (or 0.1 – 0.3 mm/min), similar to values for other cellulose-based materials in packed fuel beds [8,32]. These values apply to the early, weak phases of self-sustaining smoldering, with very low mass-loss rate.

4. Discussion

4.1. Quantification of the transition region using logistic regression

The transition regions in Figs. 3 and 4 were estimated by logistic regression. This is the most used statistical tool for identification and quantification of governing parameters for an experiment with only two possible outcomes [33]. In this article, the outcomes are smoldering and non-smoldering, and the parameters are cut-off temperature, sample height, pellets type, pipe height, and duration of external heating. The probability for onset of smoldering as a function of e.g., cut-off temperature T can be modelled using Eq. (1).

$$p = Prob\{\text{Smoldering }|\text{Cut-off} = T\} = \frac{e^{\alpha_1(T-T_{p50})}}{1 + e^{\alpha_1(T-T_{p50})}} \tag{1}$$

T_{p50} and α_1 are coefficients that are chosen such that they maximize the total likelihood of all the experimental data. The function has the following interpretation: If $T = T_{p50}$, then $p = 50\%$, and it is consequently equally likely that the experiment will end up with smoldering or non-smoldering as outcome. The probability p increases towards 100% as T increases, and decreases towards zero as T decreases (for $\alpha_1 > 0$). For large α_1, the increase is more rapid, meaning that the interval of cut-off temperatures T where the outcome of the experiment is uncertain, becomes narrower. This interval is referred to as the transition region.

The onset of smoldering could, however, depend on a number of different parameters, $x_1, x_2, \cdots, x_n$. Eq. (1) can then be generalized to Eq. (2):

$$p = \frac{e^{\alpha_1(x_1-x_{1,p50})+\cdots+\alpha_n(x_n-x_{n,p50})}}{1 + e^{\alpha_1(x_1-x_{1,p50})+\cdots+\alpha_n(x_n-x_{n,p50})}} = \frac{e^{\alpha_0+\alpha_1 x_1+\alpha_2 x_2+\cdots+\alpha_n x_n}}{1 + e^{\alpha_0+\alpha_1 x_1+\alpha_2 x_2+\cdots+\alpha_n x_n}} \tag{2}$$

All the constants $x_{1,p50}, \cdots, x_{n,p50}$ are lumped into α_0 in the last expression. The coefficients $\alpha_0, \alpha_1, \cdots, \alpha_n$ are chosen to maximize the likelihood of the experimental data. A parameter $(x_1, x_2, \cdots, x_n)$ is called significant if - when the value of its corresponding coefficient $(\alpha_1, \alpha_2, \cdots, \alpha_n)$ is set to zero - there is less than 5% probability that the observed effect was produced by chance alone. A significant parameter is likely to have an effect on the outcome of the experiment.

How well a model like the one in Eq. (2) performs, is measured through its ability to reproduce the data. Visually, a good model would separate well the smoldering and non-smoldering points with the p50-lines. In Fig. 4, part b is an example with clear separation, while part d is less convincing. However, as few parameters as possible should be included in the model not to over-fit the data, in particular when the number of experiments is small.

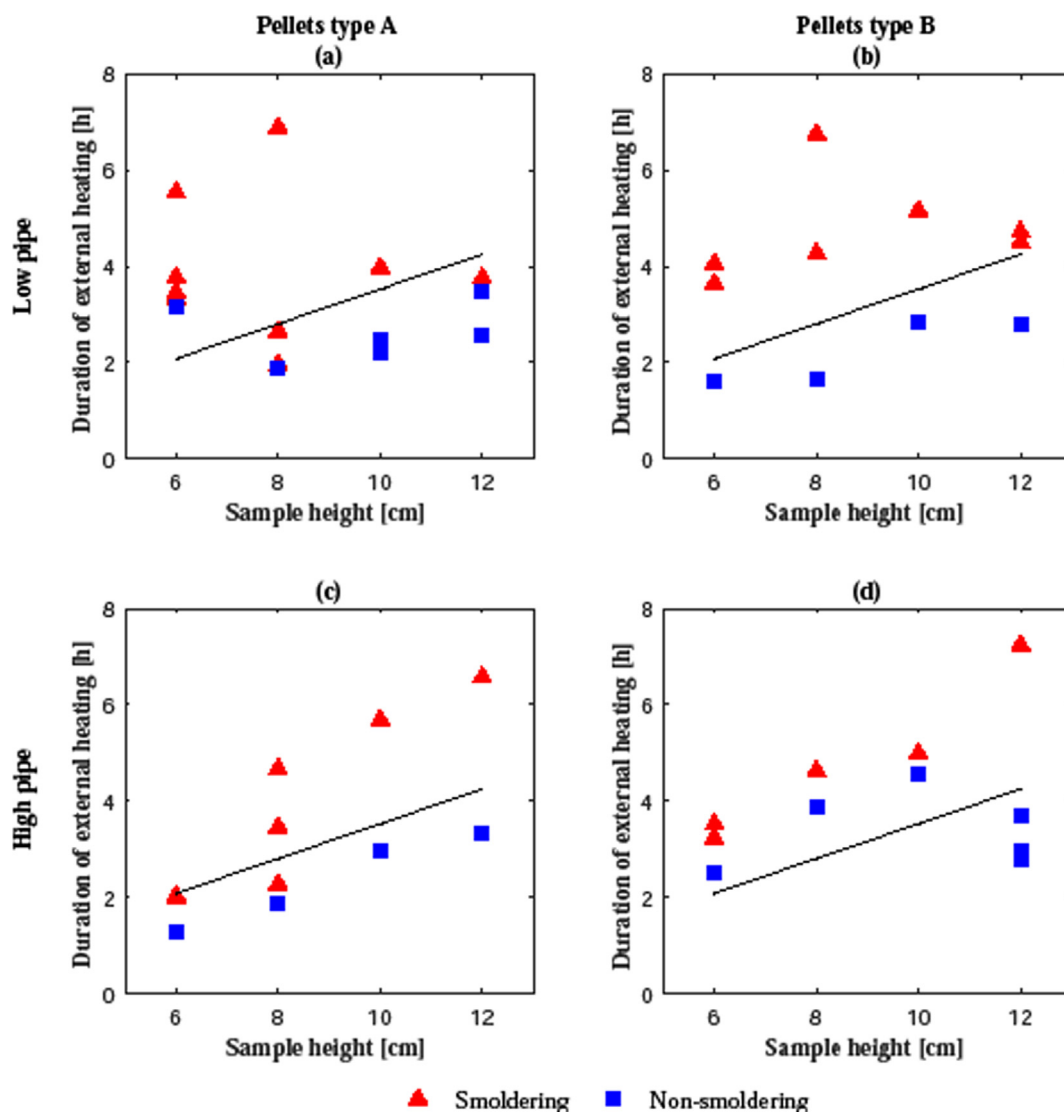Analysis of the model in Fig. 3 with four different p50-lines gives

Fig. 4. The occurrence of smoldering and non-smoldering experiments as duration of external heating and sample height were varied for two different types of wood pellets (columns) and two different pipe heights (rows).

that all the included parameters (x_1= sample height, x_2= cut-off temperature, x_3= pellets type, and x_4= pipe height) are significant when standard statistical assumptions are applied[2]. However, the number of coefficients (including combinations of coefficients) is high compared to the number of experiments, and the results should be treated with caution. Moreover, in the experimental procedure (see Section 2.3), the cut-off temperatures were chosen in order to minimize the number of experiments and still being able to detect the threshold between smoldering and non-smoldering. A statistical side-effect of this is that parameters that appear to be significant might not be significant after all.

A better approach is to suggest a model with fewer parameters. A reasonable model can be developed using x_1= sample height and x_2= cut-off temperature, and three coefficients (α_0, α_1, α_2). Cut-off temperature is then still found to be highly significant, while sample height is only close to being significant.

A similar model with a better fit to all the observed data is defined by two parameters: x_1= sample height and x_2= duration of external

heating and three coefficients (α_0, α_1, α_2), where both parameters are found to be highly significant. The results are shown in Fig. 5a, which contains all data points in Fig. 4a-d. In addition to the p50 line, the figure shows p10 and p90 lines (lower and upper dotted line, respectively) corresponding to 10% and 90% probabilities. This means that it is, e.g., more than 90% probability of onset of smoldering in the region above the p90-lines. As in the model for cut-off temperatures described in the preceding paragraph, one model covers all combinations of pipe-height and type of pellets. Cut-off temperature, pipe-height and type of pellets are not found to be significant if they are added to the model in Fig. 5a. Since cut-off temperature is correlated with duration of external heating, only one of them is significant in the same model. Interestingly, the pellets type for the model in Fig. 5a is almost significant if added to the model. This can be seen visually in Fig. 4d; if the transition region lines were shifted upwards, the lines would fit the data for pellets type B better. Different models are not mutually exclusive, since, as mentioned, the different factors tend to depend on each other.

Another model with an even better fit to all the observed data and where both parameters are significant, is given by the parameters: x_1= sample height and x_2= (sample height) × (duration of external heating). The results are shown in Fig. 5b. In this model the effect of sample height on the onset of smoldering is reduced as the sample

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[2] The transition regions in the model all have different slopes and intercepts, but a common coefficient determining the width of the transition regions. A total of 9 coefficients ($\alpha_0, \cdots, \alpha_8$) are needed for this.
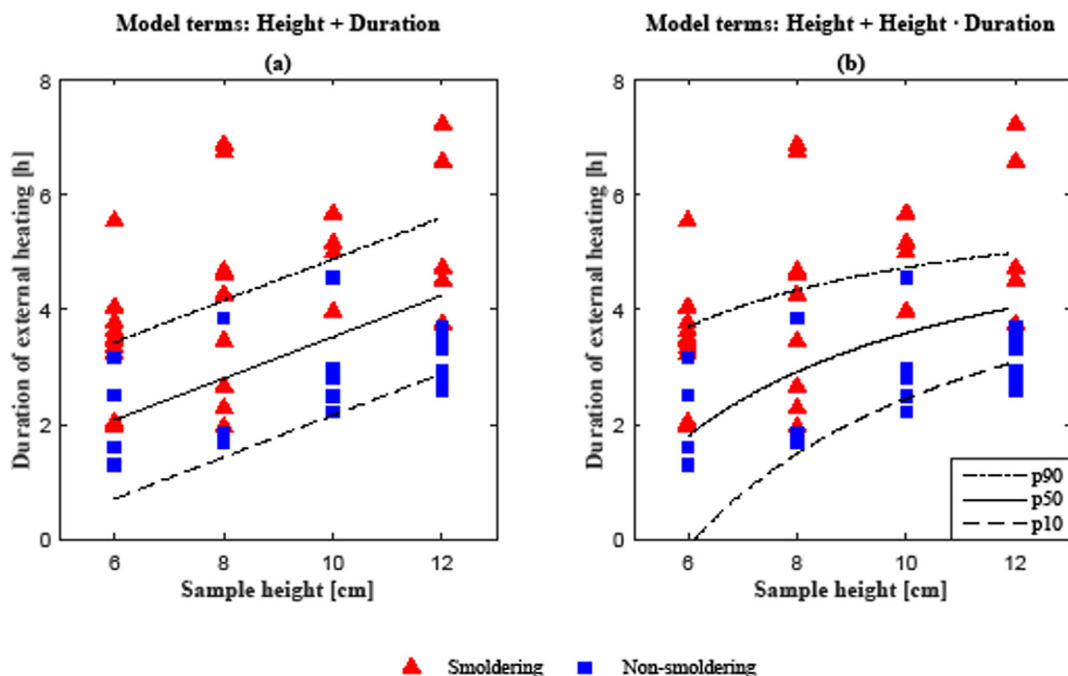
Fig. 5. Estimated transition region for onset of smoldering based on two different models, both based on two parameters: Sample height and duration of external heating. The p10 and p90 lines corresponds to 10% and 90% probabilities, respectively.

height increases: When the sample height is increased from 6 to 8 cm, the duration must be increased with 1 h, while it is sufficient to increase the duration with only 0.5 h as the sample height is increased from 10 to 12 cm. This is in accordance with the decrease in the free surface to volume ratio as the sample height increases: a lower increase in the heating duration is needed to have the same temperature build-up in the lower parts of the sample, due to the thermal insulation from the upper parts.

4.2. Cut-off temperature and duration of external heating

The cut-off temperatures and the duration of the external heating have been shown to be important parameters. In Section 4.1, the best fit to the data was obtained when duration of the external heating was used as one of the main parameters. A reasonable interpretation is that the total amount of energy delivered to the sample during the external heating determines whether smoldering will occur or not.

On the other hand, using the cut-off temperature (see Sections 2.3 and 2.4), one focuses on an assumed hot zone in the lower part of the sample as decisive for ignition of self-sustaining smoldering. Moreover, the experimental procedure used assumes equal temperatures horizontally (horizontal iso-temperature planes). As pointed out in Section 3.3, duration does not seem to be a unique function of cut-off temperature as expected for horizontal iso-temperature planes. Thus, heat propagation during external heating of the sample is probably more complex. The hotter regions could for example extend along parts of the sidewall.

The recorded temperature data have been studied in detail for such deviations. If the assumption of horizontal iso-temperature planes holds, all thermocouples 2 cm above the aluminum plate (used to determine the cut-off temperature, see Section 2.3) should reach the selected temperature simultaneously. The data demonstrate that this was never the case. An example is shown in Fig. 6. The temperature distribution for the same experiment is shown in Fig. 7.

Specifically, the very shape of the 2-cm curves in Fig. 6 shows that small variations in these curves may lead to significant variations in duration, for the same criteria in terms of cut-off temperatures for halting the external heating. This indicates that duration is the more
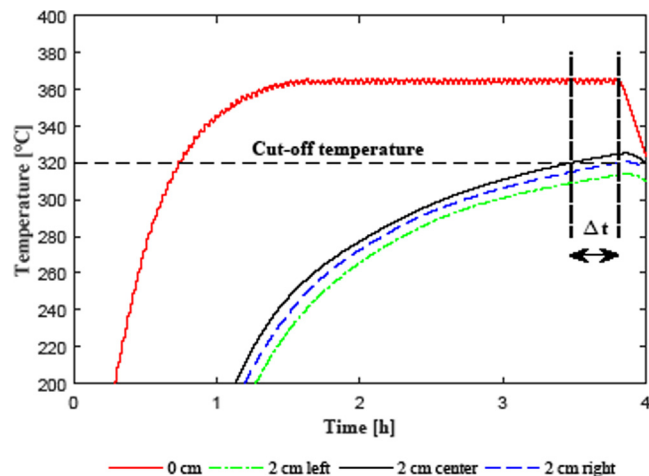

Fig. 6. Temperature as function of time during external heating, for the same experiment as shown in Fig. 2b and d. The temperature curves demonstrate that iso-temperature planes are not horizontal. The time interval (Δt) (see main text) between the times where the first and the second thermocouple reached the cut-off temperature was 0.36 h or 21 min in this experiment.

stable parameter for distinguishing between smoldering and non-smoldering cases, the same conclusion as was reached in Section 4.1.

A measure for the temperature deviations in the sample was obtained using the interval (Δt) between the times when the first and the second thermocouple at height 2 cm reached the preselected temperature (cut-off temperature). For the experiment in Fig. 6, the preselected temperature was 320 °C, and the interval (Δt) between the times when the first and the second thermocouple reached this temperature was 0.36 h. Fig. 8 shows how the time interval (Δt) varies with pellets type, pipe height and sample height. There is only small variation in the time interval Δt with sample height for the experiments with low pipe and pellets A (Fig. 8a), but an increase for the experiments with high pipe (Fig. 8c and d). The trend in Fig. 8b is more unclear. There are strong indications that the outlier in Fig. 8b resulted from a displaced thermocouple.
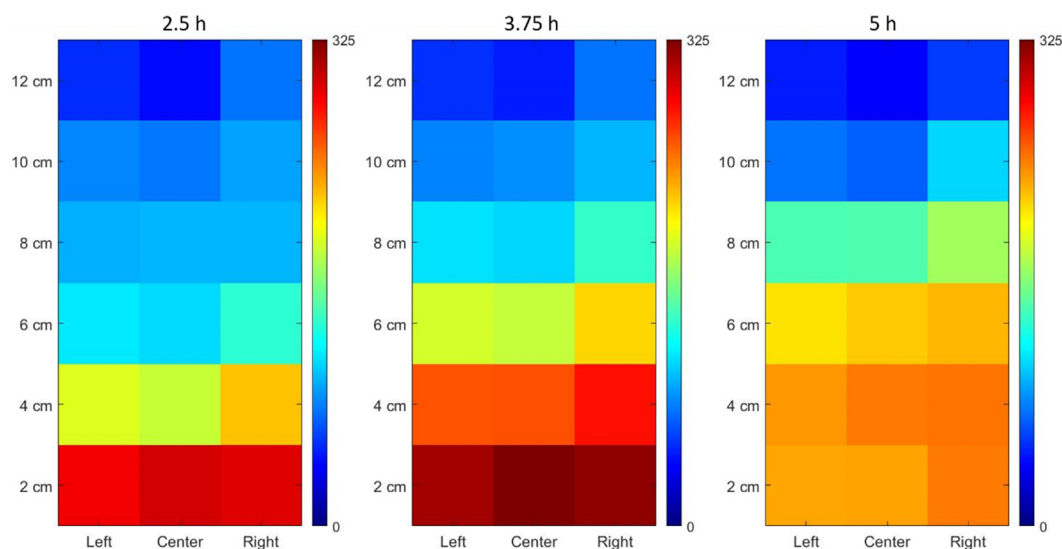
Fig. 7. Temperature distribution in a vertical section of the sample, for the same experiment as shown in Fig. 2b and d and 6. Each square corresponds to the temperature measured by one thermocouple (the positions of the thermocouples inside the sample are shown in Fig. 1). The temperature distribution is displayed at three points in time, as shown above each diagram, corresponding to during, at the end of, and after the heating period. Note that the last of these diagrams is taken at a time after the time range shown in Fig. 6. In all diagrams, the temperature distribution is asymmetrical with respect to the vertical direction, with the hottest region to the right. Surprisingly, this asymmetry still remains more than an hour after the external heating was terminated (this observation is further discussed in the main text, at the end of Section 4.2). This was after the entire sample to some extent has cooled down, and shortly before temperatures increased spontaneously.

The Δt values in Fig. 8 are of the order 1 h. Thus, the figure demonstrates that the assumption that there are horizontal iso-temperature planes during all stages of the heat transfer from the aluminum plate to the sample – is wrong. Moreover, the deviations vary significantly with the parameters. Therefore, the procedure using cut-off temperatures to determine the amount of heating necessary for

smoldering to occur may give unreliable results, since the procedure tacitly assumes such horizontal iso-temperature planes.

Fig. 8 demonstrates that the asymmetrical temperature distribution depends on the parameters. Still, when interpreting the data in the figure, one should bear in mind that temperatures are only measured at three points at the 2-cm-level. It is likely that asymmetrical temperature
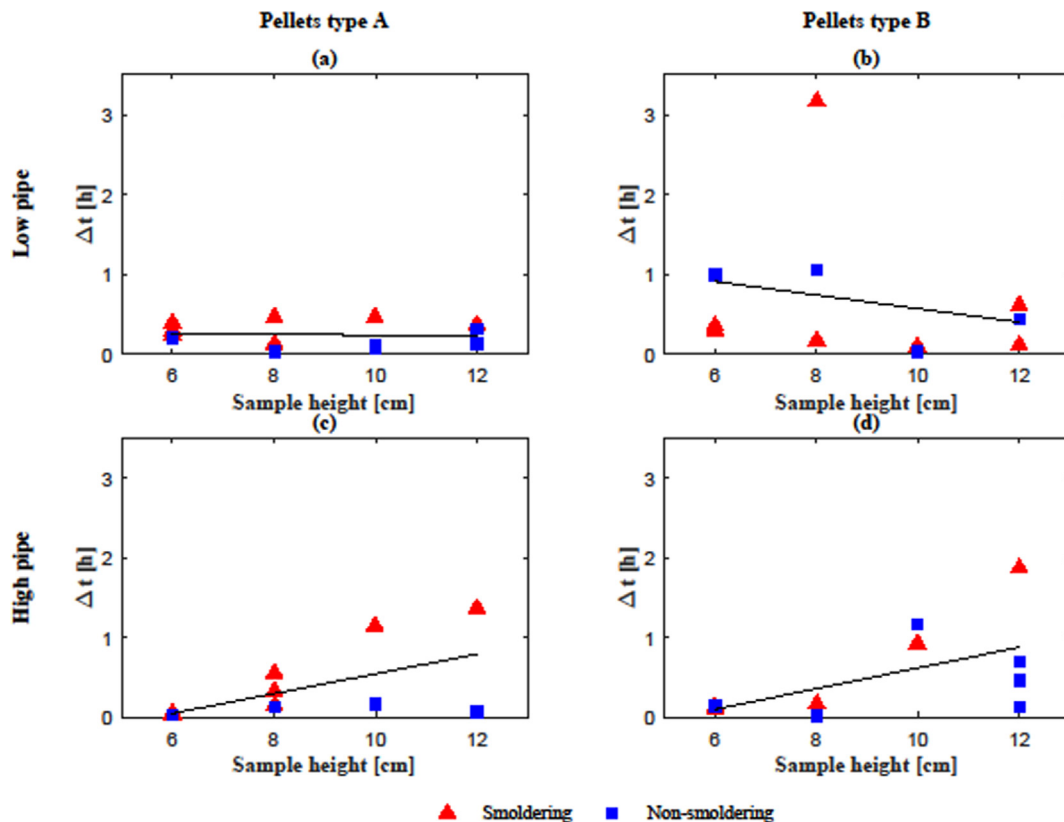

Fig. 8. Interval (Δt) between the time when the first and the second thermocouple at height 2 cm reached a preselected temperature as a function of sample height. Intervals for high pipe have a pronounced increase with sample height. The black lines in the plots are trend lines.

Table 2
Tentative results from additional experiments using the set-up in Fig. 1. In these additional experiments, the hotplate was on throughout each run, but had a lower temperature compared with the main experimental series. See further explanation in the main text.

Sample height (cm)	Time until peak (h)	Peak temperature (°C)
10	28	664
12	34	842
14	54	864
16	208	750

distributions also occur in other parts along this level, without being recorded. The fact that Δt increases systematically with sample height for high pipe may reflect more persistent convection patterns than for low pipe: hot air rises along the wall with a hot region, cool air enters at the opposite side – stabilizing or even enhancing an asymmetry in temperatures. Such convective flow patterns could comprise both the sample and the space above it in the pipe – due to the high permeability of the sample (see Table 1).

The pipe height potentially influences processes leading to (a possible) onset of smoldering through the convective flow of air and gas between sample and surroundings. A higher pipe could have a larger chimney effect and enhance these convective flows more than a lower pipe. As explained in the previous paragraph, the results in Fig. 7 indicate that there is a measurable influence from the pipe height. On the other hand, in the statistical analysis (Section 4.1), pipe height was found not to be a significant parameter in explaining the entire data set. Thus, pipe height is a parameter that should be considered for design of laboratory experiments and industrial installations. However, it is among the less important in the selection of parameters studied in this article.

As was stated in the caption of Fig. 7, the asymmetric temperature distribution shown in the figure is similar through overall heating and cooling of the sample. One possible explanation is that temperature distributions in the sample and spontaneous convective air flows between sample and surroundings are coupled and stabilize each other: Hot air and gas will rise from hot parts of the sample. Thus, cooler air will have to enter the sample from above through parts of the sample that are less hot, contributing to keeping these parts at lower temperature. Therefore, replacement air that reaches the hot parts of the sample has been preheated as it passes other parts of the sample, and the hot parts will more easily maintain combustion at a noticeable level and stay hot.

4.3. Heating scenarios

Prolonged heating increases the likelihood for smoldering. This can be related to the drying, preheating and partial combustion occurring during external heating. Longer duration of the external heating gives dryer and more decomposed material prone to smoldering.

The shifts in transition temperature (the borderline temperature between smoldering and non-smoldering behavior) can be large when heating scenario, sample size, and sample geometry are varied. To illustrate, results from three types of additional experiments will be briefly mentioned. In the first type, the samples were heated slowly, resulting in close to identical temperatures throughout the sample at any stage (isoperibolic hot storage). Pellets A (see Table 1) was used in these experiments. The general trend is that the transition temperature for smoldering to occur decreases with an increasing volume to surface ratio. Geometries with similar *V/A ratios* will have comparable transition temperatures [34], whereas an exact extrapolation according to the theory of Frank-Kamenetzkii is valid for *similar geometries* only [35,36]. With V/A ratios corresponding to sample heights (in the main experiments reported in Section 3) 8 cm and 12 cm, the transition temperature was determined to be 130 °C and 120 °C, respectively.

The second type of additional experiments were carried out in a cylinder-shaped sample holder with inner diameter 15 cm and height 1.6 m. The sample holder was thermally insulated and kept in vertical position. The sample was heated over the entire cylindrical wall, while air was flushed from top to bottom. Note that the inner diameter was the same as in the main experiments reported in this paper, see Section 2.2. Using this experimental configuration and Pellets A, a transition temperature of 130 °C was found [34–36].

Thus, experiments with slow heating and a homogeneous temperature distribution through the samples have transition temperatures that are much lower than those found for fast heating and a spatially inhomogeneous temperature distribution in the sample in the main experiments (Section 3). This is quite as one would expect.

A third type of experiments further illustrate the interdependency between heating temperature, heating duration, and the occurrence of smoldering. These experiments were carried out using the set-up shown in Fig. 1. However, instead of a short heating period with the hotplate at around 360 °C as in the main experiments (as described in Section 2.3), we used in these additional experiments a hotplate temperature around 310 °C and continual heating throughout the experiment. Additional wood pellets was repeatedly added to keep the sample mass approximately constant. This led to a very stable temperature distribution in the sample, with decreasing temperatures from bottom to top, in contrast to the erratic behavior seen in Fig. 2b. These long, quiet periods, with all sample temperatures below hotplate temperature, were interrupted by short smoldering periods with temperatures above hotplate temperature. As indicated by the preliminary results given in Table 2, as sample size increases, both the time till smoldering occurred and the peak temperature increased. Further details will be given elsewhere [37].

4.4. Sample height

In addition to the cut-off temperatures and the duration of the external heating, the height of the sample is a parameter that affects the onset of smoldering, as indicated by the positive slopes of the p50-lines in Fig. 5. Thus, the larger sample, the longer the period with external heating has to be in order to reach a state with self-sustaining smoldering. A reasonable interpretation is that for higher samples, larger volumes need to be heated. However, as indicated in Fig. 5b, the influence of the sample height diminishes as sample height increases.

For some of the experiments with sample height 6 cm, the residue after smoldering experiments contained noticeably more uncharred pellets than for higher samples. The differences could possibly be explained by a lower thermal insulation of the reaction zone and higher heat losses to the surroundings due to the higher free surface to volume ratio. There may be a critical sample height in this range for the experimental setup and protocol we have used in the main experiments (see Section 2). This is in accordance with Rein [8], who describes a minimal depth for spread of a smoldering fire.

4.5. Pellet type

The two types of wood pellets in this study differ in the measured activation energies, bark contents, production site, storage time, and differences in the amount of pine and spruce in the composition, as shown in Table 1. Despite the differences, the pellets are classified within the same pellet class (Class 1, see Section 2.1), and are similar when compared with the wide range of pellet types and classes available on the market [38]. The effect of pellet type is very close to being significant in both models in Fig. 5. The experiments with the two different types of pellets were done at two different locations. The same experimental set-up and procedure were used at both locations, and control experiments were performed to ensure reproducibility of the heating process. Still, there could be site-specific unknown variables that influenced the experiments.
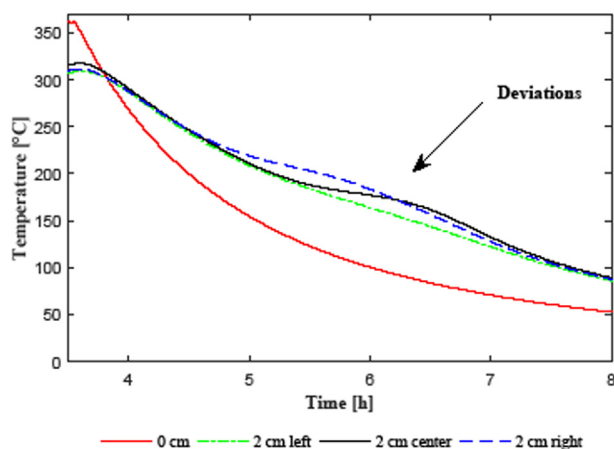
Fig. 9. Details from the non-smoldering experiment in Fig. 2. The temperatures at 2 cm height deviate from the pure cooling behavior at 0 cm (top of the aluminum plate).

4.6. Near-self-sustaining smoldering

The temperature evolution in the non-smoldering cases shows that there were processes within the sample that generated heat, but the heat production was not sufficient to establish self-sustaining smoldering. These heat generating-processes are revealed by deviations from pure cooling behavior, in the period after the electrical heater was switched off. An example is shown in Fig. 9. Here, the top of the aluminum plate (at 0 cm) displays a pure cooling behavior, while the temperature measurements higher in the sample indicate heat-generating processes through deviations from pure cooling. All non-smoldering experiments had similar temperature deviations; the extent of the deviations increased with increasing cut-off temperature. Thus, for a non-smoldering case, such details in the temperature evolution could indicate how close a sample was to self-sustaining smoldering.

4.7. Relevance for applications

In this study, a small-scale set-up was used, with sample volume up to 2 dm^3. Doubling the sample height from 6 to 12 cm gave a threefold increase in the duration of smoldering experiments, typically from 15–20 h to 60–80 h. Previous research with pellets in large-scale experiments (3.7 m^3) showed that after combustion times of about 30 h, only a small part in the center of the sample was pyrolyzed [8,39].

In Section 4.1, the best fit to the data set was obtained with the following two parameters: duration of external heating – and (sample height) × (duration of external heating), see Fig. 5b. This figure also demonstrates that the influence of the sample height diminishes as sample height increases. Thus, for much larger samples, like in industrial storage units, one would expect the temperature build-up, which in most cases stems from self-heating, to be the most important parameter.

Our main result may be useful for risk assessments of biomass fuels stored in large amounts: it points to the heating scenario as a main concern – and most decisive for whether a smoldering fire will be initiated or not. The type of biomass fuel, and the size and geometry of the storage unit are less important. Thus, one should focus on potential drivers for temperature increase: self-heating, electrical or mechanical malfunction, radiative heating.

5. Conclusion

The onset of smoldering in wood pellets has been investigated using a vertical steel pipe heated from below, with free exchange of gases at the top of the sample and thermally insulated side walls. Parameters

that may affect the onset have been investigated: pellets type, sample height, cut-off temperature, duration of external heating and pipe height. Logistic regression has been used to quantify the transition region from non-smoldering to smoldering.

Further work on the impact of varying pellet type, sample size, heating scenario, and air flow through the sample would be of interest. An upscaling of the set-up could lead to a better understanding of the scalability of the present results. Furthermore, the deviations from pure cooling observed during the non-smoldering experiments deserve a more detailed study. One would expect these deviations to grow as the onset of smoldering is approached. Finally, the asymmetrical distribution of temperatures in the sample during external heating, and the associated convective flow patterns through the sample should be further studied.

CRediT authorship contribution statement

Edmundo Villacorta: Methodology, Investigation, Writing - original draft, Writing - review & editing. **Ingunn Haraldseid:** Methodology, Investigation, Writing - original draft, Writing - review & editing. **Ragni Fjellgaard Mikalsen:** Methodology, Investigation, Writing - original draft, Writing - review & editing. **Bjarne Christian Hagen:** Conceptualization, Methodology, Writing - original draft, Writing - review & editing, Supervision, Project administration. **Sveinung Erland:** Methodology, Formal analysis, Writing - original draft, Writing - review & editing. **Gisle Kleppe:** Methodology, Software, Writing - original draft. **Ulrich Krause:** Writing - original draft, Supervision. **Vidar Frette:** Conceptualization, Methodology, Writing - original draft, Writing - review & editing, Supervision, Project administration.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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