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Smoldering fire propagation in corn grain: an experimental study

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ABSTRACT

Smoldering fire exhibits low-velocity combustion rates and is a threat commonly presented in agriculture grain storages. Some physical and chemical properties of the grain, including particle size, porosity, moisture, and the environmental conditions influence the propagation of the smoldering process. Many works evaluate the conditions to develop smoldering combustion based on particulate size and granular materials. However, only a few studies focusing on smoldering propagation in cornflour and granular grains. In this work, different particle diameters (181.6 μm, 776.4 μm and 1411 μm) and moisture contents (0% and 15%) of corn are examined to understand the corresponding impact on the smoldering propagation. To achieve this, samples of corn were placed inside a cylindrical reactor with thermocouples distributed along the apparatus' central axis and heated by a hot plate, then the smoldering reaction with a rising heating transfer profile was analyzed. Our results demonstrate how changing storage conditions can profoundly influence the smoldering propagation within a pile of corn.

1. Introduction

Due to the elevated rate of crop production, grain materials are temporarily stored in warehouses, with the aim of preserving the quality of the products until their final destination reaches consumers. One of the common designed structures for grain storage are silos comprised of large vessels that are used for long-term storage, which have been constructed from different materials, including metal, concrete, masonry, and wood. Corn, rice, soy, wheat, are examples of grains commonly stored in silos. The behavior of these materials during storage is directly influenced by the chemical and physical properties of each grain, such as moisture, density, porosity, and particle size. For environmental safety, several hazards related to the combustion of the material may occur in silo installation, including fires and explosions.

Self-ignition is a common occurrence taking place in the porous storage of bulk materials such as powders and dust [1]; this, in turn, drives a smoldering combustion reaction. Smoldering may not be just an accidental event during grain storage. This process can also be triggered by an intentional ignition in the final stage of cultivation used in open-air burning of agricultural wastes [2]. Evaluation of this smoldering hazard, taking into account storage conditions, requires experimental determination of smoldering velocities. Previous works have examined the ignition characteristics of powders, thus determining the minimum temperature that sustains the burning reaction, while others studied the smoldering performance in larger sample materials. These works evaluate the behavior through upward and downward smoldering with an ignition source located at the sample's bottom or top [3–5].

Corn grains exhibit a high combustibility level and they are prone to smoldering combustion during storage [6]. The majority of published works are related to experimental investigations with peat, sawdust, biomass, and foam, yet there are no studies that address the upward spread of corn grains with different particle diameters and moistures. Experimental studies are of great value both for researchers and for the various industrial sectors, which can serve as a basis for decision making to optimize processes or improve the safety of a process or product. For example, Seco-Nicolás et al. [7] used the experimental calculation to determine the average temperature of flat plate solar thermal collectors, which is used to calculate the efficiency of the collector. Opoku and Kizito [8] investigated the characteristics of high heat applications experimentally. The work by Akbar et al. [9] presented an experimental study on the effects of quenching treatment on microstructure and hardness characteristics.

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In this work, the upward spread of self-sustained smoldering combustion was investigated in the laboratory-scale with cornflour and corn grain samples. Burning was initiated on the bottom with a hot plate, and the temperature and the smoldering spread rate were quantified. The novelty of this paper concentrates on the evaluation of the smoldering propagation rate and the temperature evolution of cornflour and corn grains with two typical levels of storage moisture. In the next section, a background to the problem will be presented, with a focus on findings from previous studies in the same field. Following that, the set up of the experimental design is then discussed. Later, results are discussed, where the results are also compared with the other dust and powder materials analyzed in other studies. Finally, a conclusion is presented to highlight key findings of the study.

2. Background of the study

According to the National Fire Protection Association - NFPA [10], agricultural materials have different levels of combustibility that lead to the development of various fire and explosion scenarios, as determined by the intrinsic properties of the stored material. Among the properties, the particle size of the grain is one of the factors with a great influence on the combustion rate. The smaller the grain size, the greater the surface area. Thus the higher the combustion rate will be [11,12].

One of the common fire hazards during grain storage is smoldering combustion. The stagnant material and physical conditions inside the silo structure are favorable conditions for self-heating to occur, which can lead to spontaneous ignition [13,14] and then trigger the smoldering combustion. Beyond spontaneous ignition, a possible type of ignition that can develop the smoldering combustion is an external source of heat. The glowing nests, also known as a burning char, may be generated inside large storage facilities and can sustain a smoldering reaction over weeks and are especially hard to detect [15]. Ohlemiller [16] defined the smoldering process as a slow, low temperature, and flameless form of combustion, fed by the heat resulting from the reaction when the oxygen attacks the surface of a condensed phase fuel.

Additionally, the smoldering hazard may evolve to open fires and even to dust or gas explosion [17]. The occurrence of a smoldering scenario exclusively requires the co-existence of four factors: i) porous solid material; ii) oxygen; iii) ignition source; and iv) a minimum layer of material, also known as smoldering combustion square (Fig. 1). As with any combustion process, the combustible material and oxygen must be in appropriate stoichiometric concentrations and the ignition must have the minimum energy required to start the combustion process, in this case, the smoldering process. The surface area influences the combustion rate directly; to promote the attack of oxygen at the char surface, a larger surface area is necessary. Both the particle diameter and the porosity can increase this surface area, developing a higher or lower rate of oxygen attack. The smaller the particle diameter and higher the porosity, the higher will be the oxygen attack and the combustion rate.

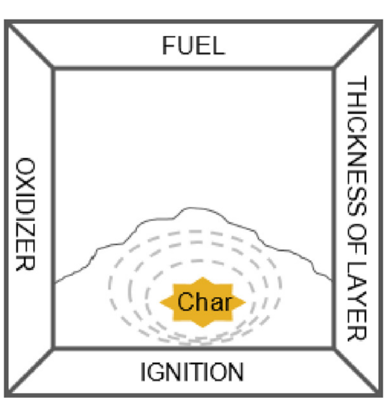


Fig. 1. Smoldering square.

Additionally, the particle diameter has an important impact on the level of heat transfer, which is responsible for supporting smoldering. From one particle to the next, the heat is mainly transferred by conduction, while within the voids between the heat occurs by convection. Related to the layer thickness of the stored material, it acts as an insulating barrier that concentrates the heat around the hot spot, which releases small quantities of heat by very slow oxidation at ambient temperatures. This heat accumulation results in a sustained increase in the temperature inside the barrier, which accelerates the oxidation rate.

Another characteristic of the grain that also plays an essential role in the ignitability and smoldering propagation of the grain is the level of moisture. According to Krause [18], some experiments show that organic material with a mass fraction of water above 16% can lead to a fermentation process developing inside the stored material within the silo, thus raising the temperature to as high as 70 °C. This reaction with the oxygen present inside the voids of the material represents a favorable condition for self-ignition, which will develop the smoldering combustion. Above a certain level, the moisture content may have an inhibiting influence on the propagation of smoldering.

The smoldering process presents some basic events responsible for the structure of smoldering fires, which can be described as four distinct chemical and thermal sub-fronts formed by preheating, drying, pyrolysis, and oxidation [19]. The pyrolysis step transforms grain into char and generates ash as a product. Then the oxidation sub front consumes char and oxygen, releasing heat to continue feeding the pyrolysis step and maintain the process already started. Fig. 2 exemplifies a one-dimensional upward smoldering process where the heat transfer occurs on the sample bottom. As heat is being transferred by the hot plate and between material particles, the column of grain material goes through all the steps. After some time, the moist material is almost dry due to the heat transfer inside the column, leading to the fundamental steps of pyrolysis and oxidation. At the end of the process, if the combustion is total, only ashes and oxidized material will remain. On the other hand, if the combustion is partial, there will also be unburnt material. The smoldering reaction can be described as incomplete combustion, during its progress beyond the production of CO_2 and H_2O, there is a significant production of CO and CH_4 [20]. The CO/CO_2 rate that can be considered as an index of the incompleteness of combustion is approximately 0.4 for smoldering reactions [19], which indicates a significant production of this flammable gas.

Several studies examined the smoldering behavior of solid materials promoted by an external source of heat, via investigating material properties and behavior in a minimum ignition temperature [21,22]. Once the ignition temperature is reached, the stored material may or may

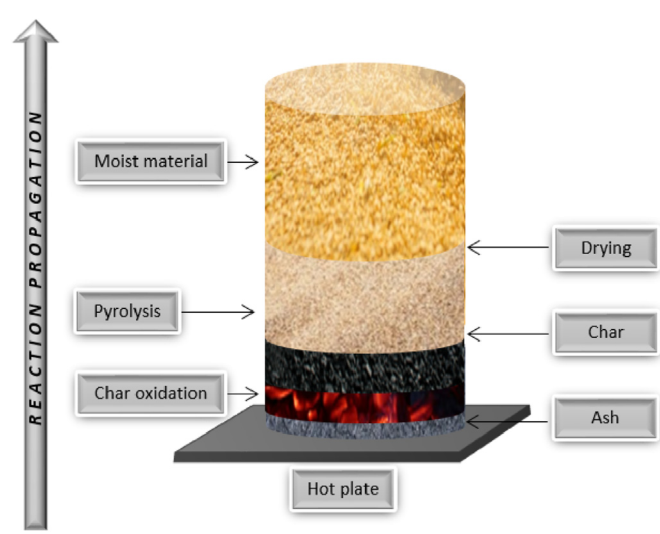


Fig. 2. Smoldering propagation in corn grains.

not continue the burning reaction if it can keep the generated heat trapped. If the heat generated is sufficient, it will feed the process of smoldering combustion until the combustible material runs out or until the stored energy is insufficient to continue the process. Investigations focusing on the smoldering process have been reported for several years with different types of materials - wood, biomass, coal, magnesium, and agricultural products [23–26].

Since the smoldering process is dependent on four factors, any change in one of them can influence the combustion propagation rate. Most works evaluate the ignition temperature of dust or bulk varying some factors related to the characteristics of the grain, the depth of the layer, type of ignition, and variations on the level of oxygen. El-Sayed and Abdel-Latif [27] studied the smoldering of a dust layer on a hot plate. They examined the influence of dust particle size, sample sizes (height and diameter), and a mixture of two combustible dust materials on the ignition time and the critical temperature.

Regarding the analysis of large amounts of combustible materials and the influence of material properties on the burning rate of smoldering, several studies that have looked at upward and/or downward smoldering spread. Palmer was one of the first authors to study the sustained smoldering inside deposits of dust up to 850 mm deep with small trains of cork dust, beech and deal sawdust, and grass dust [28]. Additionally, his work indicated that the propagation time of the smoldering from the base to the top of the deposit was about the square of the depth of the dust. Torero and Fernandez-Pello performed upward smoldering experiments with foam samples of heights of 150 mm, 175 mm, 200 mm, and 300 mm [3]. Hagen et al. pointed out that the upward smoldering rate inside cotton samples increased as the fuel density decreased [29]. Huang and Rein investigated the upward spread of peat fire from the underground to the surface after forced ignition [30].

A lack in the literature yet exists on examining smoldering velocity in grains and the influence of their chemical and physical characteristics. There is still a need for deeper studies for a full understanding of how the chemical and physical characteristics of each material influence the smoldering reaction rate.

As previously mentioned, smoldering combustion's investigation is directly connected to self-heating and self-ignition topics. For this reason, many works evaluate the materials' ignition temperature since these steps will generate enough energy to ignite the material and continue to fuel the smoldering reaction. Several authors have been studying smoldering in powder and dust and assessing the influence of materials properties for years. Intending to find out experiments related to smoldering a cluster of experiments performed was developed based in the Elsevier Scopus database.

For the initial research, a set of keywords was adopted with the following string: "smoldering OR smouldering OR self-heating OR self-ignition AND experimental OR laboratory OR test AND dust OR grain OR powder". After analyzing the titles and abstracts of all generated articles, all that was not related to the keywords and those that did not present all the information in the database were eliminated. Thus, only 66 articles were listed from 2009 to 2019.

The software VOS viewer was used to materialize the smoldering experiments cluster with the documents published in the Scopus database since 2009. For the cluster evaluation, the words in titles and abstracts were analyzed. Fig. 3 shows the cluster with network visualization of text data, which indicates four main clusters group connected with thirty-four terms. It is possible to observe that many smoldering studies start their research with the evaluation of the ignition characteristics. The reason is that many researchers evaluate the ignition in different scenarios to determine the minimum temperature or the minimum time that the studied material can maintain the burning process after the heat source is turned off. That is, they define the characteristics of the ignition that allow the material to undergo the smoldering process by itself.

The yellow cluster, consisting of five terms, encompasses experimental investigations relating the particle size to the ignition time. Another topic also addressed are the experiments with biomass, a reactive fuel, which presents a fire hazard during the stages of its production, handling, and usage. Jones et al. [31] performed some tests with single-particle measurements, thermal analysis, dust layer, and basket ignition tests. Other work investigated the effects of fuel type, moisture content, and particle size of biomass [4].

The blue cluster with nine terms contains some experiments of spontaneous ignition of the dust layer on hot surfaces. The ignition of a thin dust layer is very common in industries producing or handling dust. The work of Dufaud et al. [32] presented a model to the ignition characteristics of metal powders on a hot surface. It investigated the

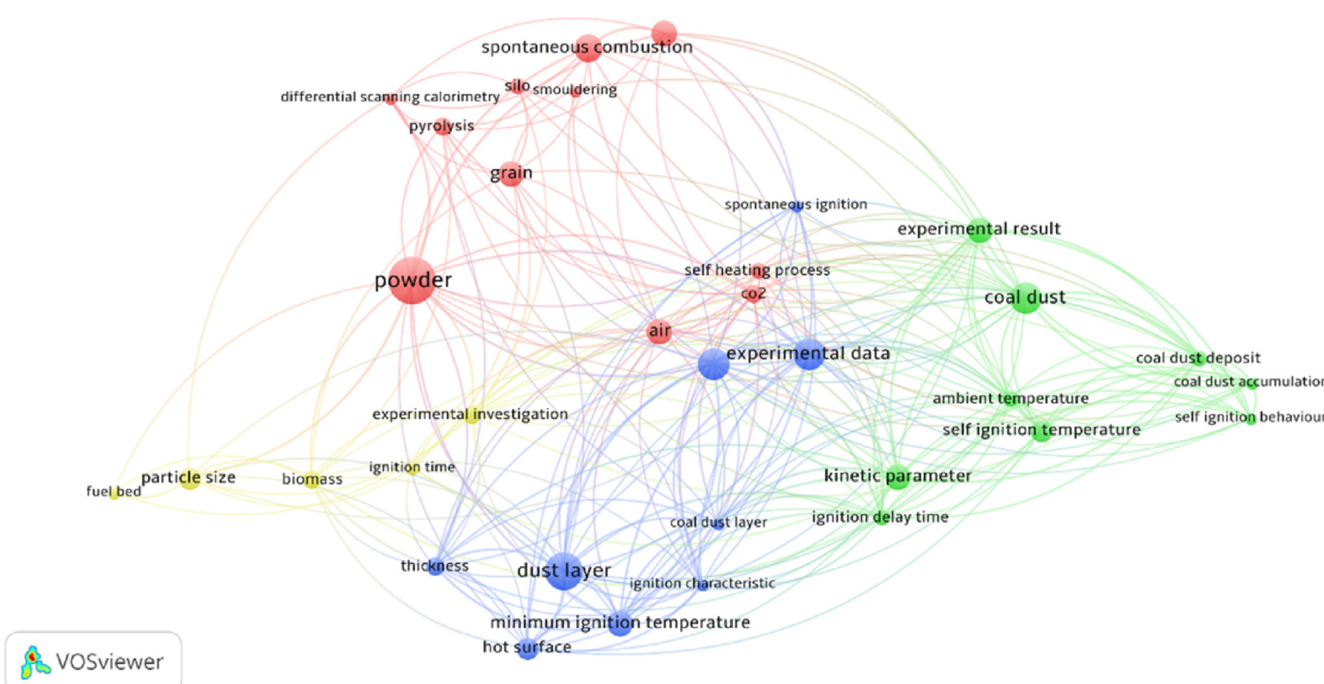


Fig. 3. A cluster of smoldering experiments (network visualization).

minimum ignition temperatures and oxidation behavior of zirconium, tantalum, and the mixture of both metals. Chunmiao et al. [22] performed experiments with four different particle sizes of magnesium powder layers and determined the minimum ignition temperature. A model describing temperature distribution overtime was also developed.

The green cluster exhibits nine terms related to studies on kinetic parameters and self-ignition temperature of some dust, such as coal dust. In addition to laboratory experiments, numerical modeling was also performed and showed good agreement with the experimental data. Wu et al. [33] experimentally investigated layers of coal dust with different oxygen concentrations and concluded that oxygen has a significant influence on the minimum ignition temperature, where it decreases when oxygen concentration increases. Other work from Wu et al. [34] studied the self-ignition behavior of three bituminous coal dust theoretically through a numerical model and the influences of ambient temperature and moisture content that indicated delays in the ignition time. Another numerical method was developed to investigate the self-ignition behavior of coal dust, varying the gas atmosphere. It was found that the inhibiting effect of carbon dioxide is relatively small, and oxygen consumption increases radically after ignition [35].

The red cluster with 11 terms encompasses experiments connecting a smoldering process, pyrolysis reaction, and spontaneous combustion in grain, the main issue under examination in this work. Handling and storing grain in silos pose a higher risk of fires, including smoldering. The experiments focus on how the self-heating develops inside the powder or dust material. Fernadez Anez et al. [36] proposed an experimental method to detect self-ignition in solid fuels (wood chips, sewage sludge, and coal) with different grain sizes. The results achieved for these materials showed the possibility to detect the spontaneous combustion process using measurements of CO and CO_2 emissions. The authors indicated an earlier detection compared with results from conventional thermogravimetry and differential scanning calorimetry tests. He et al. [37] investigated the reaction heat of the smoldering of some agro-stalks (wheat straw, corn stalk, cotton stalk, millet straw, sorghum stalk, and sweet potato rattan powder). These materials were smoldered and pyrolyzed in a simultaneous thermal analyzer (STA) and showed that the heat emission characteristic between the smoldering process and pyrolysis process was notably different. The work performed by Engel et al. [38] developed a simulation to determine the smolder temperature under different ambient conditions and the smolder temperature, according to DIN EN 50281-2-1, was compared with the simulation for validation.

The four clusters have terms in common, which allow the connection between them. One common objective of the experiments carried out in the literature was to promote better knowledge about the phenomenon of smoldering. As previously mentioned, the smoldering process can be started either with the self-ignition step of the material itself or with the ignition of a heat source. Therefore, the methodology proposed herein connects experiments performed on hot plates to evaluate the ignition temperature and the smoldering process in grain.

3. Experimental method

The experimental method utilized is set up in a way to allow the examination of certain smoldering parameters. These include the velocity of propagation and the temperature variations inside a corn pile as a function of the distance from the ignition source. The tests were performed twice to assure the representativeness of the experimental method.

3.1. Schematic laboratory-scale smoldering set-up

The main experimental set up adopted herein is derived from the literature to investigate the onset smoldering of corn granules. The experimental configuration is sketched in Fig. 4. Other authors have simulated experiments with other types of combustible materials on a similar experimental set-up to examine smoldering propagation of peat and wood char [30,39] upward.

As shown in Fig. 4, the cornflour and granular corn were filled into a cylindrical reactor. The reactor consisted of a perforated cylindrical structure made of steel and aluminum, 80 mm in diameter D and 120 mm high L that was positioned on the center of a hot plate. Then, the smoldering reactor was gently filled with the corn samples, trying to avoid their compaction.

The cornflour and granular corn were ignited from the bottom using a hot plate of 1500 W that achieved temperatures higher than 400 °C. The plate was connected and controlled by an electric panel, which was set to achieve 550 W of power to reduce any temperature variations during the experiment. This value of power can reach approximately 350 °C and guarantee that the temperature will stay consistent during the experiment. Other experiments performed without accounting for varying hot plate temperature indicated that results could be impacted. Before the final experiments, some preliminary experiments were carried out and the temperature evolution was evaluated. We observed if more than half of the sample with cornflour was able to keep the heat trapped and continue to feed the smoldering reaction. El-Sayed and Abdel-Latif [27] carried out experiments to determine the ignition temperature of a thick sample of cornflour with 10 mm layer depth on a hot plate set at 270 °C. However, as pointed out by Park [39], the larger the sample, the higher the ignition temperature of the material. The preliminary experiments of this work showed that temperatures below 350 °C did not allow burning the whole pile of cornflour with 120 mm deep with the methodology used. Right after the hot plate was turned off, the thermocouples indicated that the temperature dropped to room temperature. The 350 °C temperature allowed the heat to be stored in the sample, ensuring that

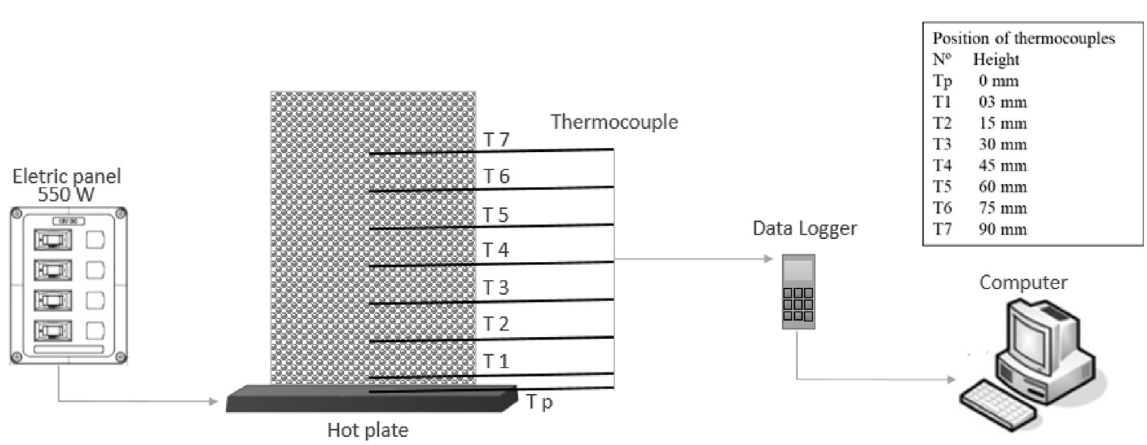


Fig. 4. Schematic of the experimental set-up.

the heat was used to continue the progress of the burning reaction.

To initiate an upward fire spread inside the samples, the hotplate was heated with an electric panel until it achieved a temperature of 350 °C, and this was then maintained for 4 h. If the pre-determined temperature and the heating time are sufficient, smoldering combustion will initiate, and the tests continue until the char and ash residues have cooled to room temperature. Initially, some preliminary experiments were conducted with lower temperatures and shorter heating time, but these did not present a long-smoldering propagation.

During the smoldering process, temperatures at different heights in the cylindrical reactor were measured using seven type K thermocouples that were placed along the vertical centerline of the reactor. Thermocouples were positioned 3, 15, 30, 45, 60, 75, and 90 mm in height from the hot surface and one on the direct contact with the hot plate.

The temperature in each thermocouple was recorded every minute with the support of a data logger. The recording of the experiments continued until the temperatures on all thermocouples dropped to room temperature. Subsequently, the samples smoldering velocities and the maximum temperature achieved in the smoldering process were determined.

3.2. Sample preparation

To obtain a better understanding of the smoldering process, two large samples of corn from a grain producer in Spain were obtained. One of the samples was cornflour and the other sample was grounded corn grains. To obtain particle diameter smaller than the grounded corn grains, the grain was crushed in a grinder and later sieved in different diameters, from 2.0 mm to 0.63 μm. Most parts of the grain exhibited particle sizes of 0.5 mm and 1.0 mm. Therefore, three-particle diameters of corn material were used in this research project as shown in Fig. 5. A small sample of each corn material was analyzed by Beckman Coulter LS Particle Size Analyzer to determine the particle distribution and estimate the particle diameter. The cornflour (F), the sieved grain 0.5 mm in diameter – small grain (GS), and the sieved grain 1.0 mm in diameter – large grain (GL) indicate the results for three percentile diameters (d10, d50, and d90) obtained from the cumulative size-frequency curves (Table 1).

Besides the particle diameter, the material moisture is another factor that influences the smoldering process. Aiming to obtain different moistures levels, the samples were separated into two environments. The first one was a climatic chamber with humidity of 60% and a temperature of 16 °C, which allowed a moisture content of 15% (m/m). The other was a desiccator with a temperature of 100 °C that eliminated the moisture content of cornflour and corn grain. All samples were left for at least three days in these environments to ensure the same moisture and, in the case of the desiccator, to ensure that the samples were totally dry, exhibiting a moisture level around 0% (m/m).

The environmental conditions of the two environments specified for the storage of the samples was controlled, which guaranteed the same final moisture value. Before each experiment, we separated and weighed a small part of the sample was for comparison and verification of the existing water content, which showed similar moisture content. While the rest of the sample protected in containers went directly to the immediate realization of the experiment, avoiding direct contact with air and the probability of adsorption of water in the material.

Table 1
Particle distribution of the corn materials.

SAMPLE	d10 (μm)	d50 (μm)	d90 (μm)
Cornflour (F)	18.62	181.6	408.7
Grain sieved 0.5 mm (GS)	253.3	776.4	1199
Grain sieved 1.0 mm (GL)	964.1	1411	1855

3.3. Experiment combinations

This work encompassed six combinations of experiments, obtained by varying particle diameter and moisture content: each combination was then performed twice, resulting in a total of 12 experiments. When the cornflour and granular corn were ignited from the bottom surface by the hot plate, smoldering propagated upward.

The experiments were performed, varying the two influencing factors chosen. For each particle diameter, two moisture conditions were evaluated, namely dried and wet. Table 2 shows the experiments conducted.

4. Results and discussion

4.1. Smoldering combustion analysis

As previously mentioned, this work aims to evaluate the influence of particle diameter and moisture in smoldering propagation of corn materials. Since all the experiments conducted exhibited some level of smoldering, it was possible to determine the smoldering velocities with the graphs of temperature evolution.

During the experiments, specific features were observed in the smoldering combustion, specifically related to the char production, glowing char, shrinkage of the sample, and smoke emissions. Fig. 6 displays a sequence of images reproduced during a test with a dried corn flour sample. The sample was carefully deposited inside the smoldering reactor, which is located just above the hot plate (A) and after a few hours of heating the samples with cornflour presented some cracks at the top of sample (B); these cracks indicate the drying step and the shrinkage of the

Table 2
Experiments performed.

CODE	SAMPLE	d50 (μm)	MOISTURE (%m/m)
F-Dried	Cornflour	181.6	0
GS-Dried	Grain sieved 0.5 mm	776.4	0
GL-Dried	Grain sieved 1.0 mm	1411	0
F-Wet	Cornflour	181.6	15
GS-Wet	Grain sieved 0.5 mm	776.4	15
GL-Wet	Grain sieved 1.0 mm	1411	15

Fig. 5. Samples of the corn materials.

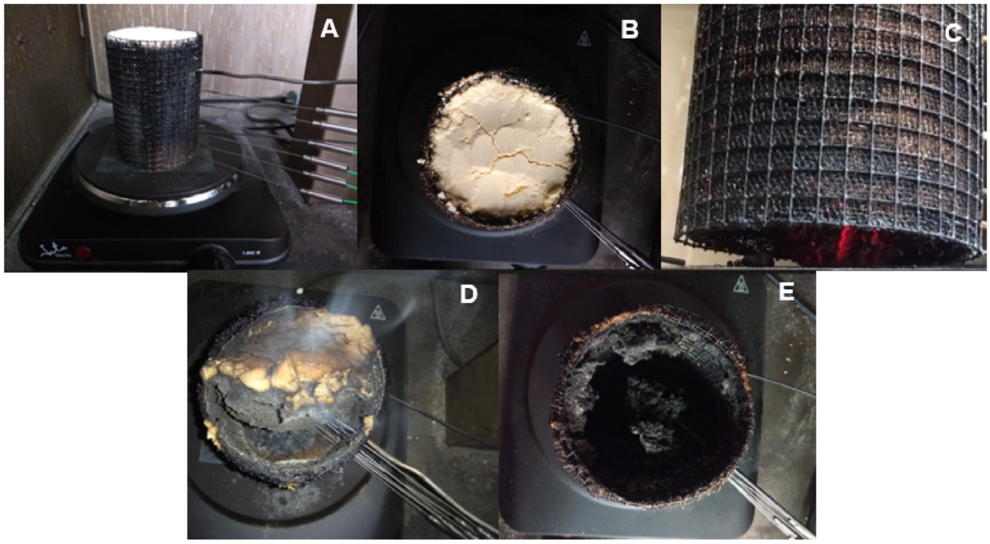


Fig. 6. The sequence of images took during the experiments.

material. However, it needs to be pointed out that such cracks were not too evident in larger grain sizes. During the tests, the samples with cornflour produced much smoke (D), and the appearance of a glowing nest was also visibly detected in some cases even with the hot plate switched off (C). At the end of the tests, in the case with cornflour, there were many ashes (E), and in the case of the two-grain types, there were ash, char residues, and unburned material.

At the end of each experiment, the data recorded by the data logger were plotted on graphs showing the temperature evolution in each thermocouple over time.

With the temperatures recorded on data loggers, it was possible to plot graphs indicating the temporal variation of the thermocouple temperatures along the central axis of the corn samples. All graphs indicate the temperatures of the eight thermocouples in each experiment, which are shown in Figs. 7-9.

Fig. 7 (A) shows the temporal evolutions of the temperature of the thermocouple obtained in an experiment with dried cornflour, and with 181.6 μm of particle diameter (F-Dried). The preheating zone in F-dried was very short as can be noticed. Then, the onset of pyrolysis appeared and the temperatures in all thermocouples indicated an increase of about 100 °C for every 20 min, approximately. The point located 60 mm from the plate (thermocouple T5 in Fig. 7 (A)) needed approximately 2 h and 20 min to increase its temperature significantly. Once the process of smoldering starts, the temperature continues to increase until it reaches a maximum of 786 °C at approximately 5 h and the smoldering process ended after 7 h of the experiment.

Fig. 7 (B) shows the result for the case of wet cornflour with 181.6 μm of particle diameter (F-Wet). In the F-Wet sample graph, it can be noted that the material remained for a long time within the pré-heating zone. It was also possible to observe that it took more time to the last three thermocouples of F-Wet to initiate the pyrolysis step. The thermocouple T5, located in the middle of the sample, required more than 2 h and 40 min to reach the hot plate temperature. The two results of cornflour exhibit some divergence, in the maximum temperature achieved and in the extension of the smoldering process. The higher moisture case has lower temperatures on most thermocouples and the temperature remained high until 7 h of the experiment after which it drops down over a 1-h duration. After the shutdown of the hot plate, it can be noticed that smoldering propagation continued until the top of the both samples, which represents that all the fuel material was consumed.

Fig. 8 (A) shows the temperature variation for the case of dried small grain with 776.4 μm of particle diameter (GS-Dried). The granular corn of GS-Dried continued the step of drying until 3 h of experiment, which implied that the thermocouples required relatively a long time to reach the temperature of the heating plate. The thermocouples T5 and T6 exceeded the hot plate temperature for nearly three and 4 h of experiment, respectively. Moreover, the process duration of smoldering was superior to the other cases performed with cornflour. A longer smoldering time was observed predominantly in thermocouples T5 and T6, which completed the process after 11 h and the sample around the thermocouple T7 was unable to sustain the heat to maintain the smoldering.

The case of small wet grain with 776.4 μm of particle diameter (GS-Wet) indicated lower temperatures than the other cases involving F-

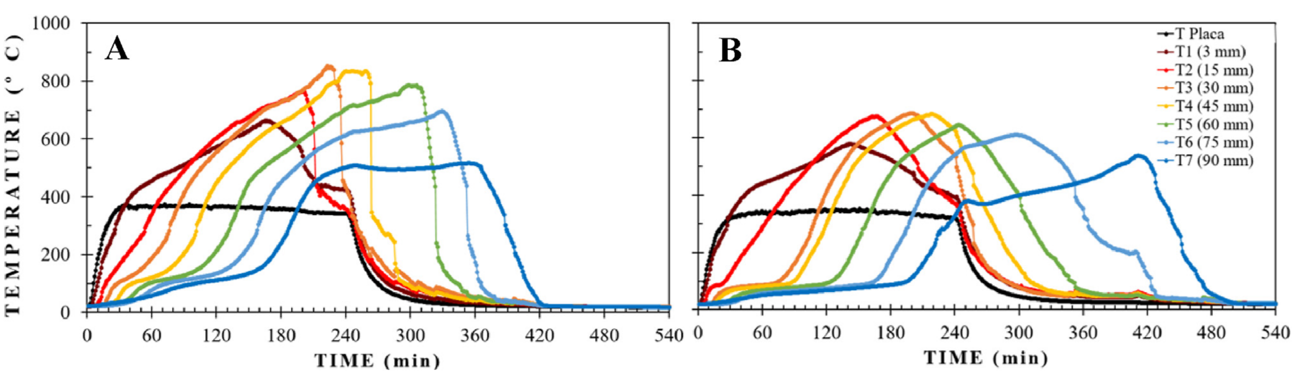


Fig. 7. Temperature evolution of F-Dried (A) and F-Wet (B).

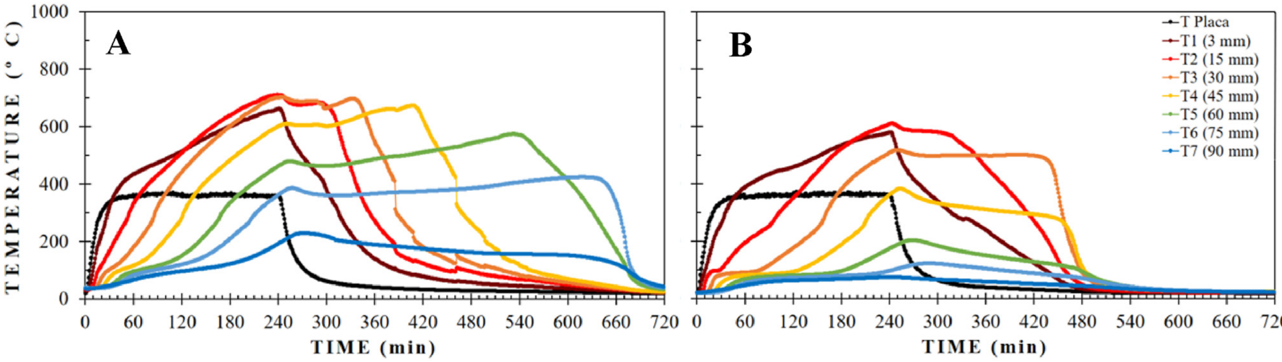


Fig. 8. Temperature evolution of GS-Dried (A) and GS-Wet (B).

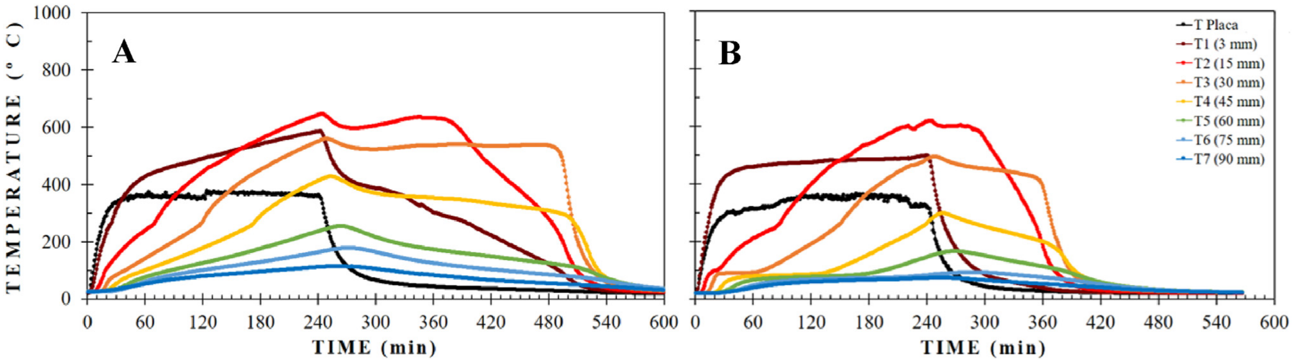


Fig. 9. Temperature evolution of GL-Dried (A) and GL-Wet (B).

Dried, F-Wet, and GS-Dried (Fig. 8 (B)). The GS-Wet experiment indicated that the corn material above the thermocouple T4 increased the drying time compared to the three other samples. Comparing the pyrolysis step of cornflour and granular grain samples, GS-Dried and GS-Wet took more time to achieve higher temperatures. The smoldering process was only kept in the sample closest to the plate. After switching off the heating source, the temperature of the thermocouples T3 and T4 remained elevated for more than 3 h, while the other temperatures were dropping down.

Both experiments with GS-Dried and GS-Wet only exhibit a partial smoldering process. The samples had not been sufficiently heated to develop the smoldering combustion. The dried case stopped approximately 75 mm away from the hot plate, whereas the wet case stopped nearly 45 mm away from the plate. Another important observation was that the case GS-Dried sustained higher temperatures within almost the entire sample, which better represents the heat transfer between the pyrolysis and oxidation step. The case of GS-Wet also exhibited this continuous heat exchange, but only from the bottom to the height of 45 mm. The reason is the lack of sufficient energy to feed the pyrolysis reaction from the height of 45 mm to the sample top.

The evolution of the smoldering in the dried and wet cases with large grain (GL) presents a similar behavior to small grain (GS), as shown respectively in Fig. 9 (A) and 9 (B). Related to the drying step, GL-Dried showed a short time to dry the sample. Nevertheless, GL-Wet with 4 h of heating the corn material was still drying above 60 height mm. Due to the greater particle diameter and smaller surface area, the onset of smoldering was more significant than the other cases, and the samples could not keep the heat generated to develop the oxidation reaction. Therefore, both tests indicated a partial smoldering process. The GL-Dried case showed higher temperatures only in the first four thermocouples closest to the hot plate. The experiment maintained elevated temperatures in T2, T3, and T4 for at least 4 h after shutting off the plate. There was no significant increase in temperature above 60 mm of the grain sample that promoted the continuation of the smoldering reaction.

The GL-Wet experiment showed lower temperatures in all thermocouples. The first three thermocouples indicated elevated temperatures for 2 h after the turning off of the equipment, which sustained the smoldering until 30 mm away from the plate. Above 45 mm of the grain sample, all the heat was dissipated and the process stopped.

As the smoldering process is slow, compared to flaming combustion, the temperatures along the sample took a long time to achieve the hot plate temperature and then sustained the smoldering propagation process, which can be noticed in all graphs. They are quite similar to those obtained by Hagen et al. [29], where their graphical results indicated an extended period for the development of smoldering in cotton samples to begin.

In upward smoldering experiments, the heat generated by the hot plate heats the bottom material until the energy is sufficient to feed the pyrolysis reaction (first step of the smoldering process) to produce char. Then the char oxidation step releases more heat, and the smoldering reaction front goes up along the sample. The seven thermocouples allocated in the center axis showed the temperature evolution as the reaction advanced. The graphs showed, in the first minutes of the experiments, a drying step with little change in temperature, followed by the pyrolysis and oxidation step with a rapid temperature increase that was sustained even after the hot plate shutdown. The temperature decay finally indicates the termination of the char oxidation.

Comparing the particle sizes, it is possible to note that the temperature profiles within the samples with larger particle diameters develop more slowly than in the smaller grains, which is totally in agreement with other experiments that have been performed in the literature [24,27]. The reason for the slower development of the temperature profile was the smaller surface area of the larger grains, which provides a lower amount of char to be oxidized. Analyzing the moisture content, the experiments showed that the pre-dried samples did not need to absorb energy from the oxidation reaction to heat and evaporate the water inside the particles. Consequently, all the heat generated by the hot plate was absorbed by the pyrolysis step, and higher temperatures were achieved.

Evaluating the temperature recorded by the T5 (located in the middle of the sample), F-Dried and F-Wet required more than 2 h to surpass the hot plate temperature. In the case with GS-Dried, the temperature of T5 took more than 3 h to raise its temperature, while for the GS-Wet sample, it did not achieve the hot plate temperature hindering the maintenance of the smoldering process. For the samples of GL, the temperatures were higher only near the heat source, approximately 45 mm (GL-Dried) and 30 mm (GL-Wet) away from the hot plate. Although the dried samples of GL and GS showed a slower evolution in temperature increase, the smoldering reaction time was maintained for two and 4 h, respectively.

Despite the lack of studies evaluating upward development with particulate materials of corn grain, analogous works with other materials help as a comparison to the smoldering process performed in this work. The samples used in the experiments of other authors presented different fuel bed heights, ranging from 150 mm to 500 mm. Huang and Rein [30] found that the ignition of peat was not possible, if the ignition source was more profound than 150 mm below the top surface, regardless of its moisture content. As stated by He and Behrendt [40], an upward smoldering is affected by the following factors, listed in decreasing order of importance: height of the fuel bed, particle size, ignition conditions, ambient temperature, and homogeneity of the fuel bed. Its work elucidated the idealization of a homogeneous sample with a flat surface at the top. However, in reality, many arcs inside the fuel bed and wrinkles on the top surface were present. In this work, we also observed an arc at the bottom of the samples near the heating plate and some wrinkles at the top of the samples with cornflour. He and Behrendt [40] evaluated four particle sizes with an almost dry moisture content of wood char granules that were dried in an oven at 398 K for 24 h. While Huang and Rein [30] investigated dried peat samples, that were obtained by drying at 90 °C for 48 h, and wet samples with 35%, 70%, and 100% of moisture content. Here, the samples were dried in an oven at 100 °C for at least 72 h and wet samples with 15% m/m of moisture content. The experiments performed showed that it is not easy to ignite corn material with a height of 120 mm, nevertheless the maximum temperature inside the piled varied between 600 °C and 850 °C. Similar as the experiments performed by He and Behrendt [4] that the piled char was only ignited after the heater has been switched on for 2 h and 40 min, and the maximum temperature near the top surface was about 1000 K (727 °C) at 10 h after the ignition onset. Other works indicated temperatures around 600 K (327 °C) and 800 K (527 °C) with wood and peat samples. According to Rein [19], typical peak temperatures for smoldering are in the range from 450 °C to 700 °C, and fuels as coal can rise up to 1000 °C. Like the work done by Huang and Rein [30], to ensure the repeatability, two experiments were conducted at each condition in this work.

Fig. 10 shows the average maximum temperature achieved in each thermocouple for the six tests varying the influence factors. Usually, the highest temperatures achieved in samples with F-Dried and F-Wet were the thermocouples T3 and T4. It can be noticed that temperatures along the central axis of the cornflour experiment were kept extremely high, indicating that the smoldering propagation achieved all heights of the cornflour samples. On the other hand, for samples with the grain (GS and GL), the highest temperatures were obtained in thermocouples T2 and T3, indicating that the smoldering hot spot was localized between 15 mm and 30 mm away from the hot plate.

An important point observed in the graph of Fig. 11 is that the value registered from thermocouple T1 for the GS-Dried sample is relatively higher than the temperature of the F-Wet sample and the values of T2 and T3 are very close. Although GS-Dried has a smaller surface than the cornflour (F-Wet), the GS-Dried behaves like the F-Wet sample up to the height of 30 mm from the hot plate. As observed in the individual graphs of the temperature evolution, these graphs indicate that the maximum temperatures in samples with cornflour are higher than in grain samples and the maximum temperatures in all thermocouples remained high enough to sustain the smoldering. The transferred heat between the pyrolysis and oxidation reactions was sufficient to develop the upward smoldering process. However, in samples with the two sizes of grain, the temperatures begin to decrease from thermocouple T3, indicating a lack of heat with sufficient energy to initiate the pyrolysis reaction, which hindered the propagation of the smoldering.

4.2. Smoldering velocity analysis

From the temperature evolutions graphs, the speed at which smoldering occurs can be determined. The time taken to reach a temperature of 350 °C is measured in each thermocouple, located at a certain distance from the hot plate. Then, a graph is plotted with the time values of each thermocouple for all experiment tests, and the smoldering velocity is calculated as the slope of the regression line. Fig. 11 shows the linear regressions and their equations for the tests with the three particle sizes for the dried and wet samples, which indicates that the slope is higher for

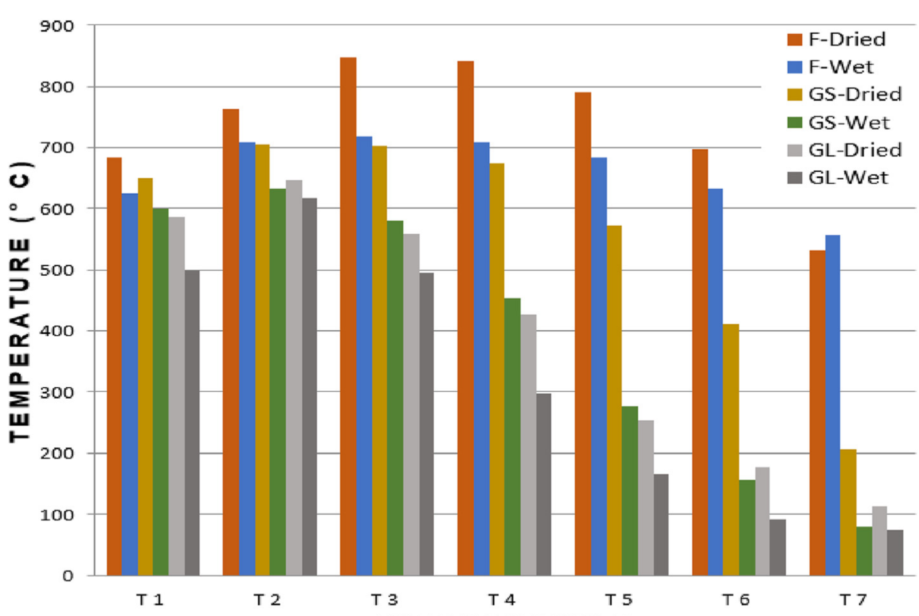


Fig. 10. Maximum temperature in each thermocouple for all cases performed.

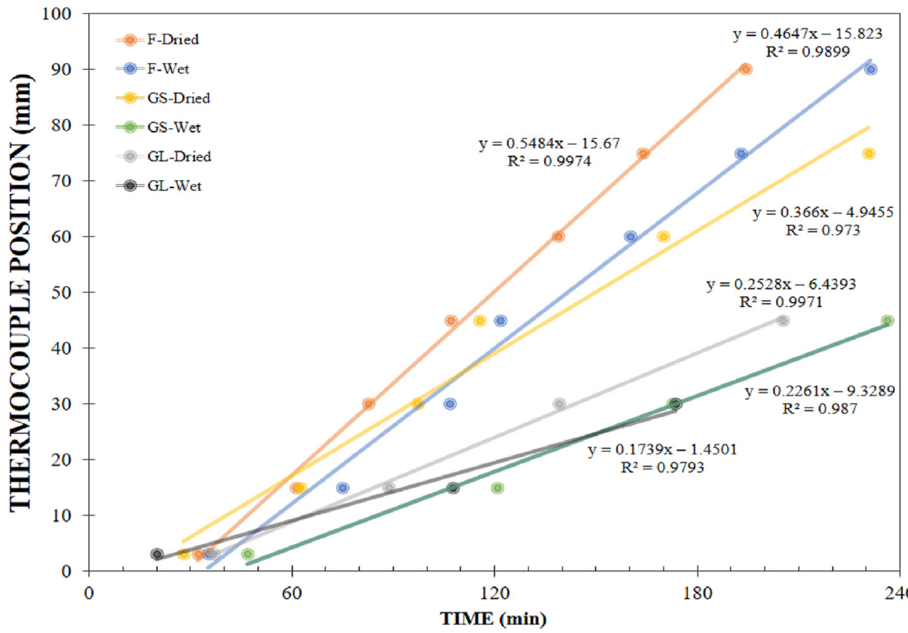


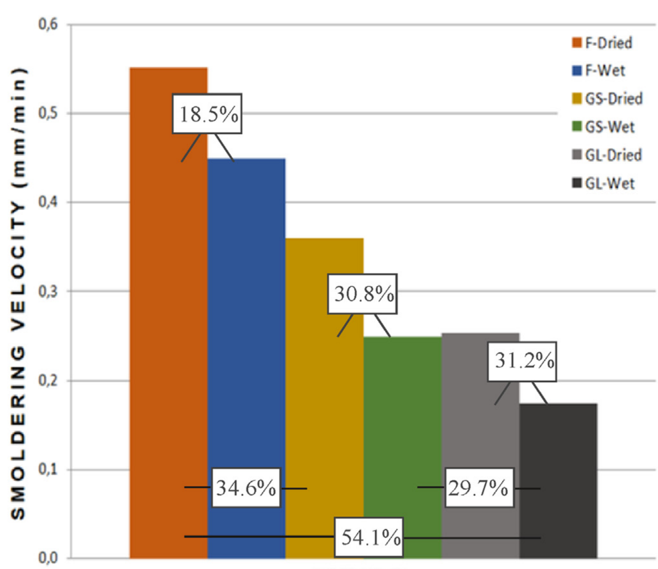
Fig. 11. Linear regression of particle diameter.

smaller particles. Analyzing the temperature development of each thermocouple, different smoldering rates were verified in each test. The smaller the particle size of the grain material, the higher the speed of smoldering propagation.

Comparing the smoldering velocities obtained for different corn material sizes and two levels of moisture, it is observed that the smaller the particle size, the higher the speed as indicated in Fig. 12. Concerning the dried material, for both grain sizes (GS-Dried and GL-Dried), the speed reached was 0.25 and 0.36 mm/min, respectively, while for the tests with cornflour, the value was 0.55 mm/min. By varying only the particle size, it is possible to verify the speed reduction. Between F-Dried and GS-Dried the reduction was 34.6%. GS-Dried and GL-Dried indicated a reduction of 29.7%. The most considerable reduction was achieved between F-Dried and GL-Dried was 54.1%.

The moisture also influenced the speed of propagation of the smoldering. It can also be verified that, as expected, the product with higher moisture produced a slower smoldering velocity rate. Comparing the samples with F-Dried and F-Wet, there was a reduction of 18.5% when increasing the level of moisture of the sample. While in the experiments with GS and GL, there was a reduction of 30.8% and 31.2%, respectively. Therefore, one can conclude that the moisture content had more influence in larger particles than in smaller particles.

Related to the smoldering velocity, Rein [19] explained that despite the considerable variation in the chemical properties of smoldering fuels, burning typically spreads around 1 mm/min, slower than flame spread. The spread rate is directly influenced by the material combustibility and it will fluctuate. Torero and Fernandez Pello's experiments evaluated foam samples and exhibited a spread between 6 mm/min and 18 mm/min [3], this value is relatively higher than that defined by Rein. Nevertheless, this elevation can be understood due to the high combustibility of the foam, since the experiment kept on by approximately 1h. Conversely, the experiments of this study showed slower smoldering velocities between 0.17 mm/min and 0.55 mm/min. Despite the slower values, the smoldering combustion in corn materials' storage is still a



Samples	Velocity (mm/min)	Smoldering
F-Dried	0.551	Total
F-Wet	0.458	Total
GS-Dried	0.358	Partial
GS-Wet	0.229	Partial
GL-Dried	0.253	Partial
GL-Wet	0.174	Partial

Fig. 12. Smoldering velocity compared to particle diameter.

present hazard, the burning process can be extended for long periods, such as weeks, and its suppression is harder to combat compared to flaming combustion.

4.3. Repeatability

The theoretical analysis of smoldering process points out that many factors can affect the combustion rate. Since these factors (external and internal) influence the process considerably, the operational conditions must be severely controlled to achieve a high level of repeatability. Our experiments intended to control the material properties and external conditions. Comparing the maximum temperatures reached along the central axis of the samples, the experiments performed with cornflour showed deviations around 0.2% and 5% between the thermocouples located until 75 mm of height and less than 6.5% near 90 mm of height. However, both diameters of granular corn exhibited significant variations in the maximum temperatures. Until the second thermocouple located at 15 mm of height, the deviations are less than 5%. Between thermocouples in 30 mm and 45 mm, the temperatures varied less than 10%. Above 60 mm, the deviations were around 10% and 33%. Regarding the smoldering velocities, the experiments for all samples showed small deviations fluctuating around 1% and 3%. Although the maximum temperatures have some significant discrepancy, the smoldering velocities rates did not seem to be affected.

5. Conclusions

This paper examined the conditions that enhanced the combustibility of grain particles stored in silos. An experimental method was utilized, where moisture content and particle size were controlled to understand the impacts on smoldering combustion in cornflour and granular grains. The upward smoldering propagation succeeded vertically from the sample's bottom until the point where the heat exchange was not sufficient to sustain the process. Samples with small particle diameter showed a higher smoldering evolution compared to samples with larger diameters. It was possible to observe that the most critical case, with higher speed and higher temperatures, was the test with dried cornflour with the smoldering velocity of 0.55 mm/min and maximum temperature achieved of 846 °C. Between the dried cases of cornflour (F) and the larger granular grain (GL), the reduction in smoldering velocity was 54.1%. One of the reasons is its relatively greater surface area compared to that of the grains. The smaller the grain size, the higher is the surface area for the oxygen attack on the particle, thus promoting a higher rate of smoldering and a greater capacity to sustain the combustion reaction. Comparing the moisture factor, it had more influence in the velocities of granular grains than in cornflour. The cornflour (F) samples indicated a velocity reduction of 18.5% when increasing the level of the sample moisture. While the experiments with granular grains (GS and GL) exhibited a reduction of 30.8% and 31.2%, respectively.

Therefore, the storage of extremely dry particulate or granular materials in structures such as silos presents a worse fire scenario due to this characteristic of remaining longer in combustion. Thus, it is possible to conclude that dried small-sized corn materials, when susceptible to the smoldering reaction, can present very high temperatures and can compromise the integrity of the structures that store these materials.

Credit authorship contribution statement

Ana Rosa: Formal analysis, Investigation, Validation, Data curation, Writing - original draft. Ahmed WA Hammad: Supervision, Validation, Writing - original draft, Writing - review & editing. Eduardo Qualharini: Supervision, Writing - review & editing, Project administration. Elaine Vazquez: Supervision, Writing - review & editing, Project administration, Funding acquisition. Assed Haddad: Conceptualization, Methodology, Writing - original draft, Writing - review & editing, Project administration, Funding acquisition.

Declaration of competing interest

The authors declare no conflict of interest. The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript, or in the decision to publish the results.

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References

[1] A. Janes, D. Carson, A. Accorsi, J. Chaineaux, B. Tribouilloy, D. Morainvillers, Correlation between self-ignition of a dust layer on a hot surface and in baskets in an oven, J. Hazard Mater. 159 (2008) 528–535, https://doi.org/10.1016/j.jhazmat.2008.02.057.
[2] N. Santiago-De la Rosa, V. Mugica-álvarez, F. Cereceda-balic, F. Guerrero, K. Yáñez, M. Lapuerta, Emission factors from different burning stages of agriculture wastes in Mexico, Environ. Sci. Pollut. Res. 24 (2017) 24297–24310, https://doi.org/10.1007/s11356-017-0049-4.
[3] J.L. Torero, A.C. Fernandez Pello, Natural convection smolder of polyurethane foam , upward propagation, Fire Saf. J. 24 (1995) 35–52.
[4] F. He, F. Behrendt, Comparison of natural upward and downward smoldering using the volume reaction method, Energy Fuels 23 (2009) 5813–5820, https://doi.org/10.1021/ef900646p.
[5] F. He, W. Yi, Y. Li, J. Zha, B. Luo, Effects of fuel properties on the natural downward smoldering of piled biomass powder: experimental investigation, Biomass Bioenergy 67 (2014) 288–296, https://doi.org/10.1016/j.biombioe.2014.05.003.
[6] Á. Ramírez, J. García-Torrent, P.J. Aguado, Determination of parameters used to prevent ignition of stored materials and to protect against explosions in food industries, J. Hazard Mater. 168 (2009) 115–120, https://doi.org/10.1016/j.jhazmat.2009.02.013.
[7] M. Seco-Nicolás, M. Alarcón García, J.P. Luna-Abad, Experimental calculation of the mean temperature of flat plate thermal solar collectors, Results Eng 5 (2020) 1–7, https://doi.org/10.1016/j.rineng.2020.100095.
[8] R. Opoku, J.P. Kizito, Experimental investigation of heat transfer characteristics and performance of smooth and wicking surfaces in spray cooling for high heat flux applications, Results Eng 6 (2020) 100119, https://doi.org/10.1016/j.rineng.2020.100119.
[9] H.I. Akbar, E. Surojo, D. Ariawan, A.R. Prabowo, Experimental study of quenching agents on Al6061–Al2O3 composite: effects of quenching treatment to microstructure and hardness characteristics, Results Eng 6 (2020) 100105, https://doi.org/10.1016/j.rineng.2020.100105.
[10] NFPA 704 (2007) 11–14.
[11] CCPS, Guidelines for Safe Handling of Powders and Bulk Solids, 2005, https://doi.org/10.1002/9780470925072. New York.
[12] S. Umbrajkar, S. Rowe, Assessing thermal stability -the challenge of powders, in: 29th Cent. Chem. Process Saf. Int. Conf. 2014, CCPS 2014 - Top. Conf. 2014 AIChE Spring Meet. 10th Glob. Congr. Process Saf., 2014, pp. 62–80.
[13] R.A. Ogle, S.E. Dillon, M. Fecke, Explosion from a smoldering silo fire, Process Saf. Prog. 33 (2014), https://doi.org/10.1002/prs.11628.
[14] P. Russo, A. De Rosa, M. Mazzaro, Silo explosion from smoldering combustion: a case study, Can. J. Chem. Eng. 9999 (2017) 1–9, https://doi.org/10.1002/cjce.22815.
[15] U. Krause, M. Schmidt, Initiation of Smouldering Fires in Combustible Bulk Materials by Glowing Nests and Embedded Hot bodies.pdf, 1997.
[16] T.J. Ohlemiller, Smoldering Combustion, 1986, p. 38.
[17] J. Vingerhoets, J. Snoeys, S. Minten, Detection of smouldering fires by carbon monoxide gas concentration measurement, Chem. Eng. Trans. 48 (2016) 457–462, https://doi.org/10.3303/CET1648077.
[18] U. Krause, Fire in Silos: Hazard, Prevention and Fire Fighting, WILEY-VCH, 2009.
[19] G. Rein, SFPE Handbook of Fire Protection Engineering, fifth ed., 2016.
[20] M. Malow, U. Krause, Smouldering combustion of solid bulk materials at different volume fractions of oxygen in the surrounding gas, Fire Saf. Sci. (2008) 303–314, https://doi.org/10.3801/IAFSS.FSS.9-303.
[21] A. Ramírez, J. García-Torrent, A. Tascón, Experimental determination of self-heating and self-ignition risks associated with the dusts of agricultural materials commonly stored in silos, J. Hazard Mater. 175 (2010) 920–927, https://doi.org/10.1016/j.jhazmat.2009.10.096.
[22] Y. Chunmiao, H. Dezheng, L. Chang, L. Gang, Ignition behavior of magnesium powder layers on a plate heated at constant temperature, J. Hazard Mater. 246–247 (2013) 283–290, https://doi.org/10.1016/j.jhazmat.2012.12.038.
[23] U. Krause, M. Schmidt, The influence of initial conditions on the propagation of smouldering fires in dust accumulations, J. Loss Prev. Process. Ind. 14 (2001) 527–532.

[24] J.L. Urban, C.D. Zak, J. Song, C. Fernandez-Pello, Smoldering spot ignition of natural fuels by a hot metal particle, Proc. Combust. Inst. 36 (2017) 3211–3218, https://doi.org/10.1016/j.proci.2016.09.014.

[25] D. Wu, M. Schmidt, X. Huang, F. Verplaetsen, Self-ignition and smoldering characteristics of coal dust accumulations in O2/N2 and O2/CO2 atmospheres, Proc. Combust. Inst. 36 (2017) 3195–3202, https://doi.org/10.1016/j.proci.2016.08.024.

[26] Á. Ramírez, J. García-Torrent, A. Tascón, Experimental determination of self-heating and self-ignition risks associated with the dusts of agricultural materials commonly stored in silos, J. Hazard Mater. 175 (2010) 920–927, https://doi.org/10.1016/j.jhazmat.2009.10.096.

[27] S.A. El-Sayed, A.M. Abdel-Latif, Smoldering combustion of dust layer on hot surface, J. Loss Prev. Process. Ind. 13 (2000) 509–517, https://doi.org/10.1016/S0950-4230(00)00004-8.

[28] K.N. Palmer, Smouldering combustion in dusts and fibrous materials, Combust. Flame 1 (2) (1957) 129–154, https://doi.org/10.1016/0010-2180(57)90041-X.

[29] B.C. Hagen, V. Frette, G. Kleppe, B.J. Arntzen, Onset of smoldering in cotton : effects of density, Fire Saf. J. 46 (2011) 73–80, https://doi.org/10.1016/j.firesaf.2010.09.001.

[30] X. Huang, G. Rein, Upward-and-downward spread of smoldering peat fire, Proc. Combust. Inst. 37 (2019) 4025–4033, https://doi.org/10.1016/j.proci.2018.05.125.

[31] J.M. Jones, A. Saddawi, B. Dooley, E.J.S. Mitchell, J. Werner, D.J. Waldron, S. Weatherstone, A. Williams, Low temperature ignition of biomass, Fuel Process, Technol. 134 (2015) 372–377, https://doi.org/10.1016/j.fuproc.2015.02.019.

[32] O. Dufaud, D. Bideau, F. Le Guyadec, J.-P. Corriou, L. Perrin, A. Caleyron, Self ignition of layers of metal powder mixtures, Powder Technol. 254 (2014) 160–169, https://doi.org/10.1016/j.powtec.2013.11.053.

[33] D. Wu, F. Norman, F. Verplaetsen, J. Berghmans, E. Van Den Bulck, Experimental analysis of minimum ignition temperature of coal dust layers in oxy-fuel combustion atmospheres, in: Procedia Eng., 2014, pp. 330–339, https://doi.org/10.1016/j.proeng.2014.10.441.

[34] D. Wu, M. Schmidt, J. Berghmans, Spontaneous ignition behaviour of coal dust accumulations: a comparison of extrapolation methods from lab-scale to industrial-scale, Proc. Combust. Inst. (2018), https://doi.org/10.1016/j.proci.2018.05.140.

[35] D. Wu, F. Norman, M. Schmidt, M. Vanierschot, F. Verplaetsen, J. Berghmans, E. Van den Bulck, Numerical investigation on the self-ignition behaviour of coal dust accumulations: the roles of oxygen, diluent gas and dust volume, Fuel 188 (2017) 500–510, https://doi.org/10.1016/j.fuel.2016.10.063.

[36] N. Fernandez Anez, J. Garcia Torrent, L. Medic Pejic, C. Grima Olmedo, Detection of incipient self-ignition process in solid fuels through gas emissions methodology, J. Loss Prev. Process. Ind. 36 (2015) 343–351, https://doi.org/10.1016/j.jlp.2015.02.010.

[37] F. He, W. Yi, J. Zha, Measurement of the heat of smoldering combustion in straws and stalks by means of simultaneous thermal analysis, Biomass Bioenergy 33 (2009) 130–136, https://doi.org/10.1016/j.biombioe.2008.05.006.

[38] L. Engel, S. Kimpel, J. Franke, Determination of the smouldering temperature on the basis of adiabatic tests, Chem. Eng. Trans. 48 (2016) 127–132, https://doi.org/10.3303/CET1648022.

[39] H. Park, Hot Surface Ignition Temperature of Dust Layers with and without Combustible Additives, Worcester Polytechnic Institute, 2006. http://onlinelibrary.wiley.com/doi/10.1002/fam.810120306/abstract.

[40] F. He, F. Behrendt, Experimental investigation of natural smoldering of char granules in a packed bed, Fire Saf. J. 46 (2011) 406–413, https://doi.org/10.1016/j.firesaf.2011.06.007.