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Ana Carolina Souza Rosa

AN EXPERIMENTAL STUDY ON SMOLDERING AND OTHER RISKS

Rio de Janeiro
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UFRJ

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Master's Thesis presented to the Environmental Engineering Program, Escola Politécnica & Escola de Química, from Universidade Federal do Rio de Janeiro, as part of the requirements for obtaining a Master's degree in Environmental Engineering.

Advisor: Assed Naked Haddad, Prof. D.Sc.

Co-Advisor: Laia Haurie Ibarra, Prof. PhD

Ana Maria Lacasta Palacio, Prof. PhD

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I dedicate this thesis to my mother and father who always supported me, my advisor, who always believed in my potential and God for giving me strength in this journey.

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All my thanks to those who encouraged and supported me during these years and whose names are not mentioned here, to all my friends.

“Man's real treasure is the treasure of his mistakes, piled up stone by stone through thousands of years” (José Ortega y Gasset)

RESUMO

Rosa, Ana Carolina Souza. An experimental study on smoldering and other risks. Rio de Janeiro, 2018. Dissertação (Mestrado) – Programa de Engenharia Ambiental, Escola Politécnica e Escola de Química, Universidade Federal do Rio de Janeiro, Rio de Janeiro, 2018.

"Smoldering combustion" é um risco muito comum e recorrente durante o armazenamento de material granulado e particulado. Dependendo das características do material e dos fatores externos, a combustão pode ser mais ou menos favorecida, influenciando diretamente a velocidade da combustão. Uma grande variedade de grãos agrícolas é suscetível a esse tipo de risco, como milho, soja e trigo. Este trabalho tem como objetivo avaliar como a variação de três fatores influenciadores pode modificar a velocidade de queima do milho. Como metodologia de estudo, os experimentos foram planejados e desenvolvidos em testes laboratoriais de pequena escala com a ajuda do software Minitab. No projeto de experimentos no Minitab, selecionou-se o tipo de experimento fatorial completo, definindo três fatores (diâmetro de partículas, umidade, condição de ventilação) com dois níveis e duas repetições. Dezesseis experimentos foram realizados com base na norma EN-50281-2-1 com o suporte de uma placa de aquecimento e termopares distribuídos no eixo central das amostras. Após a execução dos experimentos, calculou-se a taxa de combustão de cada experimento. Avaliou-se que o diâmetro das partículas teve maior influência na taxa de combustão, seguido pela umidade. A avaliação da influência de fatores influenciadores é de extrema importância para a compreensão deste fenômeno para o dimensionamento de estruturas adequadas de armazenamento com a integração de barreiras para reduzir o risco deste tipo de acidente.

Palavras-chave: 1. Smoldering. 2. Milho. 3. Planejamento Experimental. 4. Minitab.

ABSTRACT

Rosa, Ana Carolina Souza. An experimental study on smoldering and other risks. Rio de Janeiro, 2018. Dissertation (Master degree) - Environmental Engineering Program, Escola Politécnica e Escola de Química, Universidade Federal do Rio de Janeiro, Rio de Janeiro, 2018.

Smoldering combustion is a very common and recurring risk during the storage of particulate and granular material. Depending on the characteristics of the material and the external factors, the combustion can be more or less favored, influencing directly the smoldering speed. A wide variety of agricultural grains is susceptible to this type of risk, such as corn, soy, wheat. This work aims to evaluate how the variation of three influencing factors can modify the speed of corn burning. As a study methodology, the experiments were planned and developed in small-scale laboratory tests with the help of Minitab software. In the design of experiments in Minitab, the type of full factorial experiment was selected, defining three factors (particle diameter, humidity, ventilation condition) with two levels and two replicates. Sixteen experiments were performed based on the standard EN-50281-2-1 with the support of a heating plate and thermocouples distributed on the central axis of the samples. After the execution of the experiments, the smoldering rate was calculated for each one experiment. It was evaluated that the particle diameter had a greater influence on the combustion rate, followed by humidity. Evaluation of the importance of influencing factors is extremely important for the understanding of this phenomenon for the design of adequate storage structures with the integration of barriers to reduce the risk of this type of accident.

Kew-words: 1 Smoldering Combustion. 2. Corn. 3. Design of Experiments. 4. Minitab.

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LIST OF ACRONYMS

BLEVE	Boiling Liquid Expanding Vapour Explosion
CCPS	Center for Chemical Process Safety
CFD	Computer Fluid Dynamic
CSB	United de States of Chemical Safety Board and Hazard Investigation Board
DOE	Design of Experiments
EN	Normalización Europea
FTA	Fault Tree Analysis
GG	Godbert Greenwald
LEL	Lower Explosive Limit
LFL	Lower Flammability Limit
LOC	Limiting Oxygen Cconcentration
MEC	Minimum Explosible Concentration
MIE	Minimum Ignition Energy
MIT	Minimum Ignition Temperature
NFPA	National Fire Prevention Association
UEL	Upper Explosive Limit
UFL	Upper Flammability Limit
VCE	Vapor Cloud Explosion
UNE	Una Norma Española
RFM	Recursive Fitting Method

CONTENTS

1 INTRODUCTION

1.1 Background of the study

Accidents involving combustible dust are still a recurring topic, studies on the combustibility of dust and on understanding how the phenomenon occurs began with the explosions and fires in coal mines. At the same time, studies on metals, and then on plastic and agricultural products began. In most accident cases, these occur due to lack of knowledge of the combustibility potential of dust. With respect to agricultural products, grains throughout the handling, transport or storage process may, through friction between the particles or with some equipment produce particulate material, which under certain conditions may pose a variety of fire or explosion hazards.

Reports of explosions or fires with agricultural products date back to 1785, with the flour explosion in a warehouse when a spark of a lamp ignited a dust cloud. Among the agricultural products with combustibility, potential are corn, soybeans, wheat, barley, oats, rice, sugar, etc. Throughout its manufacturing process, clouds can be generated with the particulate material, and thus creating explosive atmospheres. After the production process, these materials are stored in structures such as bins, hoppers, and silos to be destined for the final consumer. The storage equipment provides an ideal level of containment to provide gas and dust explosions, which is why silos have been the scene of many accidents with grain dust.

Silos are structures to store raw materials or products with different materials sizes such as grains or dust. Before its construction should be considered the type of material to be stored and how they will be carried out the steps of loading and unloading. To assist in these stages are used elevators and conveyors system that are most responsible for promoting a certain level of friction and for leaving the dust in suspension. Therefore, for its construction, it is important to know the maximum level of pressure and temperature allowed so that it does not compromise the integrity of the structure of the silos and does not allow an accident.

Among the hazards related to fire are the combustion with flames - flash fire and combustion without flames - smoldering. The last hazard occurs due to the overheating of the particulate materials that can occur by any piece or part of a heated device or by a natural process initiated by the biological action according to Ogle (Ogle, 2016).

That could start an exothermic reaction and with the release of energy causing the heating of a certain mass of dust or grains. This process is a slow process, its detection is difficult in some cases and the product may continue to burn for weeks as described by Russo et Al (Russo, Rosa and Mazzaro, 2017). According to Vingerhoets, Snoeys, and Minten (Vingerhoets, Snoeys and Minten, 2016), smoldering may evolve to open fires and even to dust or gas explosions. With regard to explosion hazards, its confined structure for storage is the main factor responsible for the sudden rise of pressure, thus allowing the triggering of a gas or dust explosion. The loading and unloading steps in the silos are extremely crucial because they allow the particulate material to be suspended in the air and allow oxygen to enter the silos, as shown by Ogle et Al (Ogle, Dillon and Fecke, 2014).

Among the research carried out with this theme, several authors address the issue of dust explosion both experimentally and theoretically. However, the analysis of dust accidents in silos, as well as the phenomenon of smoldering combustion, is still a topic that needs a more profound and effective study, so that it is possible to evaluate the phenomenon, what factors influence and how this type of combustion behavior varies by influencing factors.

Due to this lack of specific studies, this dissertation presents two main objectives. To identify the principal risks related to silos and evaluate experimentally smoldering combustion by varying some specific conditions. Thus, observe the evolution of temperature over time in a column of dust and powders, which is one of the possible risks in silos.

1.2 Relevance

Smoldering is a process that requires little energy and little heat flow to initiate a combustion, as opposed to combustion with flames, a factor that may increase the likelihood of the occurrence of this risk. Another important fact is the high complexity and combination of transport and thermochemical processes inside a reactive porous area, which could hinder the activity in suppressing this type of combustion when compared to the flame combustion.

There are some theoretical and experimental studies related to the theme. However, the knowledge about this type of combustion is limited and there is still a need for further study on the subject to assure that the phenomenon could be better understood, and best preventive measures could be taken to avoid serious accidents.

1.3 Objectives

The present work has the objective to evaluate the principal risks in silos storage and analyze the theoretical differences between the possible scenarios of risks and the influence factors of each risk. Other objective is to promote a review of the literature in the Scopus database and analyze the research development. As an experimental objective to observe the phenomenon, understand how the smoldering process occurs in corn grains´-ççç, how the temperature varies inside the material samples and how this can be a source of ignition to another fires and explosions. As a final objective, model the phenomenon with the main factors influencing the rate of combustion of a material sample with different particle sizes.

1.4 Limitations of the study

The theoretical scope of this research is limited to the analysis of the risks related to fires and explosions that occur during grain storage in silos. The review of the literature performed was limited to two scenarios of accidents: dust explosions and smoldering combustion.

With respect to the scope of the practical part, the experiments were carried out with corn grains, as this type of grain is involved in many reports of explosions. Some experimental articles related to smoldering have used grains such as soybeans, wheat, corn, among others, which ensured that the corn grain was susceptible to smoldering phenomena in this work. The smoldering experiments were limited to corn grains with three different sizes. Other particle sizes and grains types were not tested for lack of time to test more samples.

During the preliminary tests some possible values for the temperature and the heating time of hot plate were examined. However, for the initial tests only a single value of each parameter was set to reproduce all the experiments. Which ensured a total process of smoldering on samples with corn flour.

Throughout the smoldering process, some toxic gases are released which also have a high level of flammability. At a certain concentration within flammability limits, they may become hazardous sources of fires or even sources of explosions. The quantity of gases and the production rate of these gases is an important way to characterize smoldering in structures such as silos. These gases may be the fuel to evolve from a scenario of smoldering combustion to a flame combustion. However, the analyzes of gases and the transition from smoldering to burning with flames were not present in the initial scope of the tests.

2 DUST EXPLOSION AND OTHER FIRES IN SILOS

2.1 Silos and their risks and accidents

Silos are structures designed for the temporary stocking of granular materials and have a large storage capacity. They are mainly used for grain storage, in order to protect them against weather, absorption of soil water and atmospheric humidity. Due to the level of combustibility of granular materials stored, the risks of combustion are always present during handling and storage. The material type stored in silos must be considered in order to completely recognize the potential risks and the substances likely to ignition.

In general, there are three possible substances liable to ignition: flammable or combustible gases, dust clouds and a settled layer of granular or particulate material (Krause, 2009). The first one is produced during some chemical reaction of the material stored and the second could be generated due to normal activities as filling and emptying the silo. The handling step can promote friction between the grains and partition into particles with smaller diameters as grain dust. This feature may favor the formation of dust clouds and may also facilitate the start of a combustion process due to the increased surface area of the particles.

Combustion is a self-sustained oxidation process of a determined fuel as a gas, liquid, or solid that generates heat, light or smoke. As the combustion development occurs in the gas phase, liquid and solid fuel must undergo vaporization or pyrolysis processes by absorbing the heat liberated during the combustion reaction. Pyrolysis is an endothermic process and a common feature in the combustion of most solid carbonaceous fuel with the development of a thermal degradation (Ogle, 2016).

The combustion process can be divided into two categories: flaming and smoldering combustion. The first one exhibit a cloud of gases between the lower flammability level (LFL) and upper flammability level (UFL) that can ignite and generate flames and sometimes smoke. Flammable gases or vapors and combustible solids can be the source of this combustion type. As opposed to combustion with flames, there are no flames in smoldering combustion but glowing char and smoke are presented in this process. Additionally, smoldering combustion may occurs with combustible solids as grains, powders, and dust.

According to CCPS (2005) dust presents four main inherent hazards in its characteristics: combustibility, instability, reactivity, and toxicity. The first three are directly related to accidents involving combustion process as fires and explosions. Much of the dust handled in the industries has combustible characteristics, which can lead to these accident scenarios. For the characterization of hazards, The standad 704 of National Fire Protection Association (NFPA) ('NFPA 704', 2007) presents classifications of three principal categories for particulate material: health, flammability, and instability.

For fire and explosion hazards, the most relevant characteristics are flammability and instability of the particulate material, which the first one address the degree of susceptibility of materials to burning and the second one address the degree of intrinsic susceptibility of materials to release energy. The categories include materials with larger particle diameter such as powders and pellets up to dust (Table 2-1). Materials in categories 1 through 4 are prone to the combustion process.

Table 2-1 - Hazard categories.
Source: (NFPA 704, 2007)

DEGREE OF HAZARD	DESCRIPTION	CRITERIA
4	Materials that are readily dispersed in air and burn readily	Materials that ignite spontaneously when exposed to air.
3	Materials in this degree produce hazardous atmospheres with air under almost all ambient temperatures or, though unaffected by ambient temperatures, are readily ignited under almost all conditions.	Finely divided solids, typically less than 75 micrometers (that present an elevated risk of forming an ignitible dust cloud, such as finely divided sulfur, aluminum, zirconium, and titanium); Materials that burn with extreme rapidity, usually by reason of selfcontained oxygen (e.g., dry nitrocellulose and many organic peroxides)
2	Materials that must be moderately heated or exposed to relatively high ambient temperatures could release vapor in sufficient quantities to produce hazardous atmospheres with air before ignition can occur.	Finely divided solids less than 420 µm (40 mesh) that present an ordinary risk of forming an ignitible dust cloud; Solid materials in a flake, fibrous, or shredded form that burn rapidly and create flash fire hazards, such as cotton, sisal, and hemp.
1	Materials that must be preheated before ignition and combustion can occur.	Combustible pellets, powders, or granules greater than 420 µm Finely divided solids less than 420 µm that are nonexplosible in ambient conditions, such as low volatile carbon black and polyvinylchloride (PVC).
0	Materials that will not burn under typical fire conditions.	Materials that will not burn in air when exposed to a temperature of 816°C (1500°F) for a period of 5 minutes.

Among the risks of fires and explosions, one can trigger the other, however, there are some differences between these two types of phenomenon. According to Crowl (Crowl, 2003), the difference depends on the time frame in which the events occur. Fires are events that occur much slower compared to explosions which are very short events with a sudden release of energy. Also to corroborate the idea of time frame, Rein (Rein, 2016) explain the basic difference is that fires are considered the flame spreading and explosions are considered the flame propagation, which its velocity is much faster than the first one.

Initially, to develop a fire is necessary the coexistence of three requisites: fuel, oxidizer (air) and a source of ignition, which is known as fire triangle (Figure 2-1). Actually, in a silo the fuel and the oxygen always be present, so any source of ignition with a minimal energy could be the possible third element of this fire triangle.

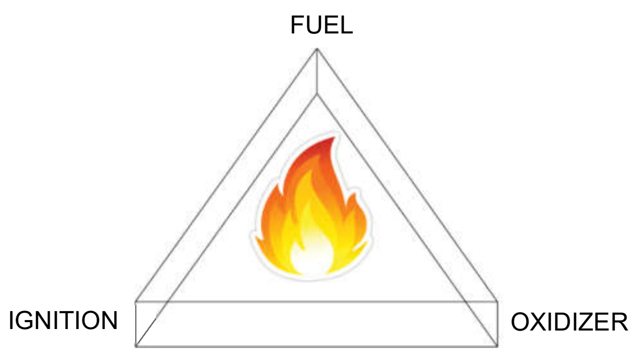

Figure 2-1 - Fire triangle.

To develop an explosion it is necessary the existence of five requisites of the explosion pentagon. Besides the three requisites of the fire triangle, it is necessary a perfect mixing between the fuel with an oxidizer (air) in the explosion range and a partial or complete confinement of the fuel-air mixture (Figure 2-2). If the fuel is a particulate matter as grain dust, the explosions are entitled as dust explosion. For this type of explosion, the dust must be suspended and form a cloud with a concentration between the explosive limits.

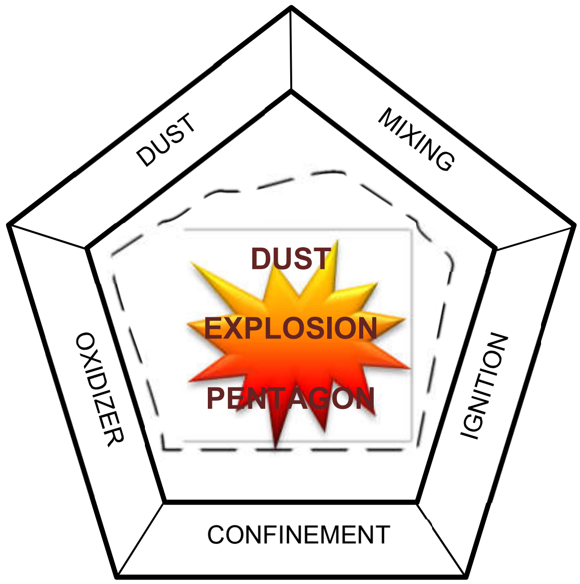

Figure 2-2 - Dust explosion pentagon.

In fires, the stage can begin with two fuel options: flammable gases or fuel vapor formed by pyrolysis step when particulate solid materials burn. The gaseous phases of the fuel and oxidant are not mixed prior to the combustion reaction. The oxygen and the fuel molecules are separated and move toward each other through a diffusion process until the flame surface to maintain the reaction, the flame present in this case is a diffusion flame. By contrast, gas explosions are different and are characterized by a pre-mixing of fuel and oxygen; it exhibits a premixed flame type that justifies the reason why they occur faster than fires. In dust explosions, a dust cloud shows a diffusion flame near the dust particles and a premixed flame in a greater scale (Rein, 2016).

Explosions exhibit a combustion reaction in the mixture flammable-air gas system in a region known as combustion wave, which can be of two types, deflagration or detonation wave. Figure2-3 has depicted a simple structure of a combustion wave. The basic difference between them is the propagation speed of the reaction front and the level of pressure achieved.

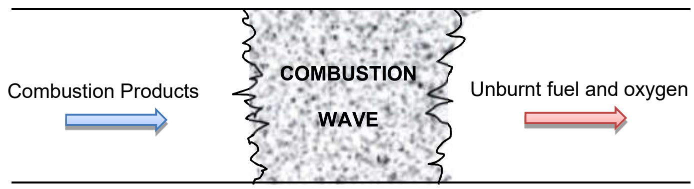

Figure 2-3 - Structure of a combustion wave.

In order to perform an analysis of an accident involving a combustion event, it is important the complete understand of the events related to fires and explosions. One must be taken into account the mechanisms of flame spreading, flame propagation and principally the characteristics of the material stored.

2.1.1 Types of silos

Several researchers have studied the complex structure of the silos in order to understand their behavior during storage, as well as the stages of filling and emptying. For the realization of a suitable design of a silo should be considered the type of material that will be stored, to minimize the risk of accidents. Despite storing some industrial materials such as aluminum dust, most of the material stored in silos are agricultural products.

Silos can be described as vessels that are used for the storage of particulate solids materials. Generally, silos are large structures used for long-term storage either at the beginning of a process to store raw materials or at the end of a process to store products. The material used in silos construction defines one of their classifications. The typical material used in its construction can be metal, concrete, masonry, and wood silos (used for seed storage). Additionally, there is a silo type called silo bag, which is an emergency mode of grain storage and it has been commonly used in Brazil areas with typical vegetation of the central regions of this country for corn and soybean storage.

Another silos classification is related to the silo and soil position that can be divided into three groups (Lacovic, 2014):

I - Elevated silos: characterized by being fixed above the ground.

II - Underground silos: are those located below ground level. They are simplified constructions, but of easy permeability and difficult discharge.

III - Semi-subterranean silos: they are silos of intermediate form to the elevated silos and underground. They are half below ground level and a half above ground level.

The silo capacity (size of each structure) depends on how much grain the producer wants to store and for how long, besides the number of crops per year. Based on inherent safety, it is safer to have more silos of medium size than silos of large size, which can reduce the risks of accidents and the severity of consequences.

Most silos are circular, but square or rectangular silos are often used due to the easily in fabrication and space economics. According to (CCPS, 2005), most silos used in chemical processing units are of modest size (3000 cubic feet or less). Silos of modest size are fixed on structures as legs, while very large silos are generally located on foundations.

For the design of a silo, it is essential the knowledge about the flow process and a mechanical perspective. Regarding process flow, there are two different flow patterns in the storage silo when it is being emptied: mass flow and funnel flow. In the first case, all of the material stored in a silo is constantly moving with very little difference in a velocity profile across the silo. However, in funnel flow, there is a central flow of material and a stagnant material near the silo walls. The material in the upper part around the flow continuously refills it (CCPS, 2005). These normal flow patterns are illustrated in Figure 2-3.

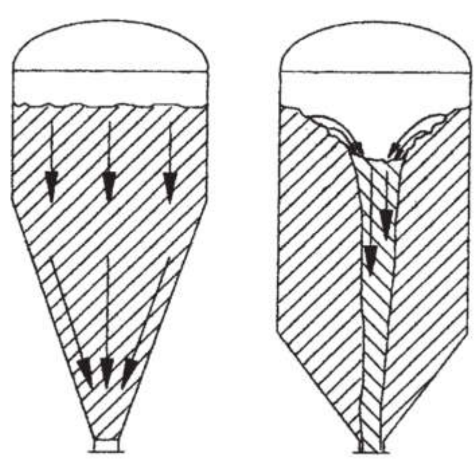

Figure 2-4 – Normal flow patterns: mass flow and funnel flow.

Source: (CCPS, 2005)

In relation to the other mechanical perspective, the stresses applied to the walls of the silo must be determined to design the silo to supports these loads. These stresses come from the static and dynamic conditions throughout the process of filling and discharging the silo. Experimental measurements on models and industrial size models have indicated that the distribution of stresses on the wall changes considerably when the flow begins after the initial filling, and these tensions remain after the exit is closed (CCPS, 2005).

The silos structure may have other auxiliaries in order to assist the usage and protect them from severe accidents such as fires and explosions. The usage is facilitated by auxiliaries like a bin vent filter, ladders, bulk elevators, platforms, and a railing around the roof.The industrial instrumentation can assist in the protection by measuring and monitoring using level, temperature and pressure instruments.

2.1.2 Risks and accidents

Several authors around the world documented cases related to accidents of dust explosion, such as Eckhoff (2003) that in his work elucidates some explosion historical cases with dust, such as grains, coal, aluminum, and silicone. The author begins with the Count Morozzo’s detailed a report of the first dust explosion accident cited in the literature that occurred in 1785 with flour dust in a warehouse at a bakery in Turin, Italy. Despite the low severity, Morozzo’s analysis of this accident allows the learning of important concepts such as the key elements for the development of explosions.

Additionally, it was illustrated a set of accidents evolving grain dust explosions in Norway and the United States with wheat, barley, and oats between 1970 and 1988. Besides, three accidents cases related to smoldering gas explosions in Norway, USSR and Sweden with grain, feedstuffs and pelletized wheat bran in silos between 1985 and 1989 were described. Some accidents with fires, coal dust and gas explosions in coal dust plants and silos were also reported.

The U.S. Chemical Safety and Hazard Investigation Board (CSB) has published several accidents reports with combustible dust explosions and fires. At least 281 combustible dust fires and explosions occurred in the industry between 1980 and 2005 that occurred in a variety of industries with different types of combustible dust and triggered 119 fatalities and 718 injuries in the United States (U.S. CSB, 2006)

Abbasi and Abbasi (Abbasi and Abbasi, 2007) exposed a list of illustrative examples of dust explosion incidents between 1911 and 2004 in different countries. According to the authors, some of the most damaging accidents in chemical and agro-processing industries have been caused by dust and not by flammable liquids or gases.

Among the risk of accidents, dust explosions pose the most serious and widespread of explosion hazards in the process industry alongside vapor cloud explosions (VCE) and boiling liquid expanding vapor explosions (BLEVE) (Abbasi and Abbasi, 2007). This is explained by the large amounts of heat and high pressures generated in milliseconds when a combustible dust cloud is accidentally ignited in a process enclosure. The resulting dust deflagration may, or may not compromise its structural integrity depending if the vessel is adequately protected or not (Taveau, 2017).

Dust explosions continue to be the exclusive cause of severe losses in diverse process industries. The complete understanding about an accident with a detailed report from accident investigation represents an important source of information to develop safety barriers.

2.2 Gas and Dust Explosions

2.2.1 Literature Review

Over the years several accidents have occurred with different combustible dusts. This stimulated the study of explosions with this type of solid fuel. Studies related to dust explosions have started from reports of incidents in coal mines, followed by studies with metal dust and agricultural grain dusts such as wheat, corn, rice, etc. In the literature review, some work reviewing the theoretical work on dust explosions were found. Work with explosions can be roughly divided into three main groups: air-dust mixture, ignitions, explosion parameters.

Regarding the concentration of the air-dust mixture, there are studies concerning the evaluation of the quality of dispersion of particles in a sphere of 20 L (Wu, Liu and Zhang, 2017), numerical simulations to examine the dust particle distribution and its influencing factors during the dispersion (Chen *et al.*, 2015), comparisons between measurements of dust concentration and Computer Fluid Dynamic (CFD) modeling of silo filling processes in order to find configurations where the dust concentration exceeds the lower explosion limit (Klippel *et al.*, 2014). There are also work related to concentrations such as the limit oxygen concentration (LOC) in the work of Clouthier and Krause who investigated the LOC of fifteen fuel dusts and methane mixtures in the standard 20 L explosion chamber (Clouthier and Krause, 2017); as the minimum explosible concentration (MEC) in the work of Yuan et al with the extensive investigation on experimental test of dust MEC using the Siwek 20 L vessel (Yuan *et al.*, 2012) In addition, there are some studies evaluating the influence of particle size on the explosions. We et al (We *et al.*, 1991) studied the particle factors which affect the explosibility of dusts as particle size, dust concentration, pore size, and chemical composition. Di et al (Di *et al.*, 2013) used a previously validated CFD 3D model to simulate the dust dispersion inside the sphere at different dust diameters.

Ignition issues are related to the sensitivity of the stored materials, the lower the energy required to start the combustion, the greater the sensitivity. Khalili (Khalili *et al.*, 2012) demonstrated the peculiar behavior gas/dust explosions and proposed simple models to estimate th ignition sentivity. Hosseinzadeh et al (Hosseinzadeh *et al.*, 2015) measured the critical ignition of a variety of pure dusts with different particle sizes and the impact on the minimal ignition energy (MIE) on mixtures of them. Danzi, Marco, and Riccio (Danzi, Marmo and Riccio, 2015) evaluated the effects of a mixing of a combustible dust in the minimal ignition temperature (MIT) of the layer and of the cloud tested in a Godbert Greenwald (GG) furnace and on a hot plate.

Other works concern explosion parameters to understand their development and severity. Chen et al (Chen *et al.*, 2017) studied the flame propagation and dust particle movement of a large-scale dust explosion process using CFD. The work of Cuervo, Dufaud, and Perrin (Cuervo, Dufaud and Perrin, 2017) was developed a numerical tool to assess the flame propagation properties analyzing the flame velocity as a function of its stretching and hydrodynamic instabilities. Proust (Proust, 2017) performed large scale experiments which the flame velocity and turbulence characteristics were evaluated. Ben et al (Ben *et al.*, 2015) calculated radiative heat transfer exchanged between particles in suspension and proposed an original method of calculating heat transfer between dust and gas in the preheat zone of the flame.

The two essential parameters studied to assess the severity of explosions are the maximum explosion pressure reached ($P_{máx}$) and the maximum rate of pressure $(dP/dT)_{máx}$, alson known as the deflagration index (K_{st}). Fumagalli et al (Fumagalli *et al.*, 2017) proposed a mathematical model for the deflagration index as a function of the mean particle diameter for organic dusts. The work of Zhang, Ma, and Zhang (Zhang, Ma and Zhang, 2014) presented a method to calculate the rate of pressure rise for dust explosion known as Recursive Fitting Method (RFM), which had better repeatability and higher accuracy. Dyduch and Pekalski (Dyduch and Pekalski, 2013) presented two methods to determine the explosion severity parameters for more accurate determination of explosion severity parameters, a statistical method and a method that adjusts the results for variances of turbulence intensity.

Despite several studies, dust explosions still require further studies in order to understand the phenomena involved and the factors that influence and can aggravate an explosion, since accidents with dust explosions are still recurrent in the industries.

2.2.1.1 A cluster of Literature Review

The subject of a dust explosion in several industries and with different types of dust has been studied for years by several authors. For a better understanding of how this issue has developed and continues to develop over the years, it is important to conduct a search in literature. In this work, a bibliographic research was carried out in the Elsevier Scopus database.

For an initial research, we used the keywords with the following string: "dust explosion". The key words in this string were selected in order to obtain the largest number of documents related to this subject, which returned 1818 documents. Selecting only the documents in the English language and publications from 1967 untill 2018 reduced the number to 1057 documents. After analyzing the titles and abstracts, all that was not related to the keywords and those that did not present all the information in the database were eliminated and only 372 documents was selected. Another analysis of abstracts was perfomed and only 201 documents were listed from 1967 to 2018.

For the evaluation of the clusters formed in the topic of the dust explosion of this work, the software VOSviewer was used, in which the list of documents generated by the Scopus database was inserted. It presents three possibilities of types of cluster visualization: network, overlay, and density. There are some map options that allow the creation of different maps. It will be presented two clusters of dust explosions: a map based on text data and a map based on bibliographic data of authors.

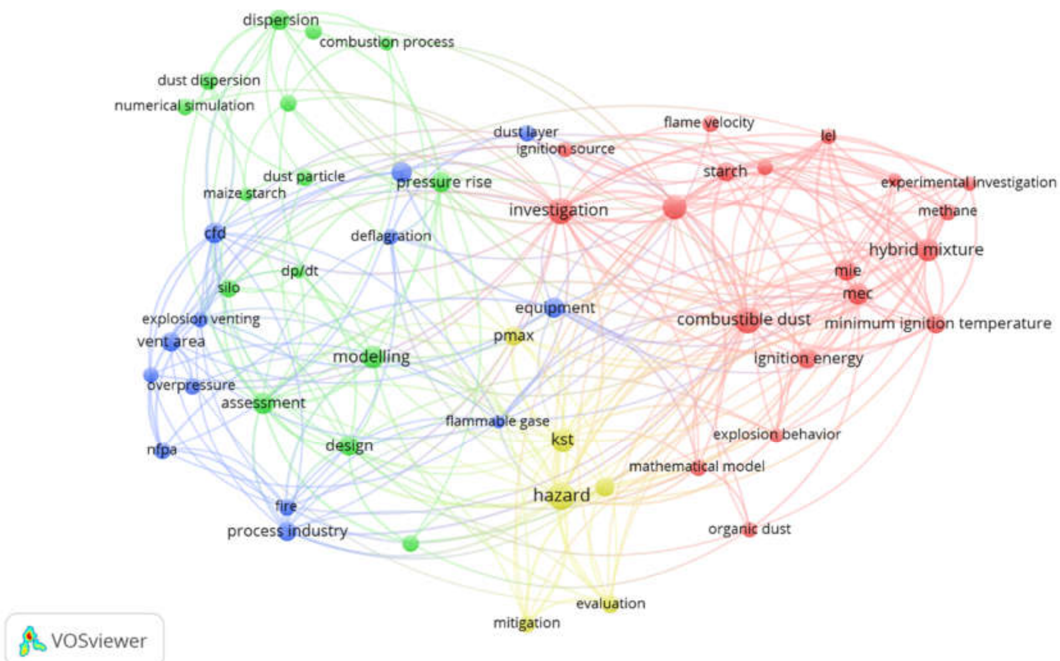

Figure 2-5 - Network visualization of dust explosion text data.

The fisrt map analyses words present in the titles and abstracts of the selected works and was developed selecting at least six as a minimum number of co-occurrence terms. Figure 2-5 shows the cluster with network visualization of text data, which indicates four clusters group connected with fifty three terms (Table 2-2). In this view, it is possible to check the links between the most used terms in the selected works according to the number of occurrences, that allows us to understand how the research is progressing in the topic of dust explosion.

Table 2-2 - Cluster groups of dust explosion text data.

Cluster 1	Cluster 2	Cluster 3	Cluster 4
Combustible dust	Assessment	CFD	Evaluation
Experimental investigation	Closed vessel	Deflagration	Hazard
Experimental result	Combustion process	Duct	Kst
Explosion behavior	Design	Dust layer	Mitigation
Flame velocity	Dispersion	Equipment	Pmax
Hybrid mixture	dP/dt	Explosion venting	Prevention
Ignition energy	Dust dispersion	Fire	
Ignition source	Dust explosion hazard	Flammable Gases	
Investigation	Dust particle	NFPA	
LEL	Maize Starch	Overpressure	
Lycopodium	Modelling	Pipe	
Mathematical model	Numerical Simulation	Process Industry	
MEC	Pressure rise	Vent area	
Methane	Silo		
MIE	Sperical vessel		
Minimum ignition temperature			
Organic dust			
Oxygen concentration			
Starch			

LEL = Lower explosible limit, MEC = Minimum Explosible Concentration, MIE = Minimum Ignition Energy

The overlay visualization depicts the topics covered and how the research developed over the years. Figure 2-6 shows this type of visualization and allows us to recognize the direction of the research regarding the dust explosion.

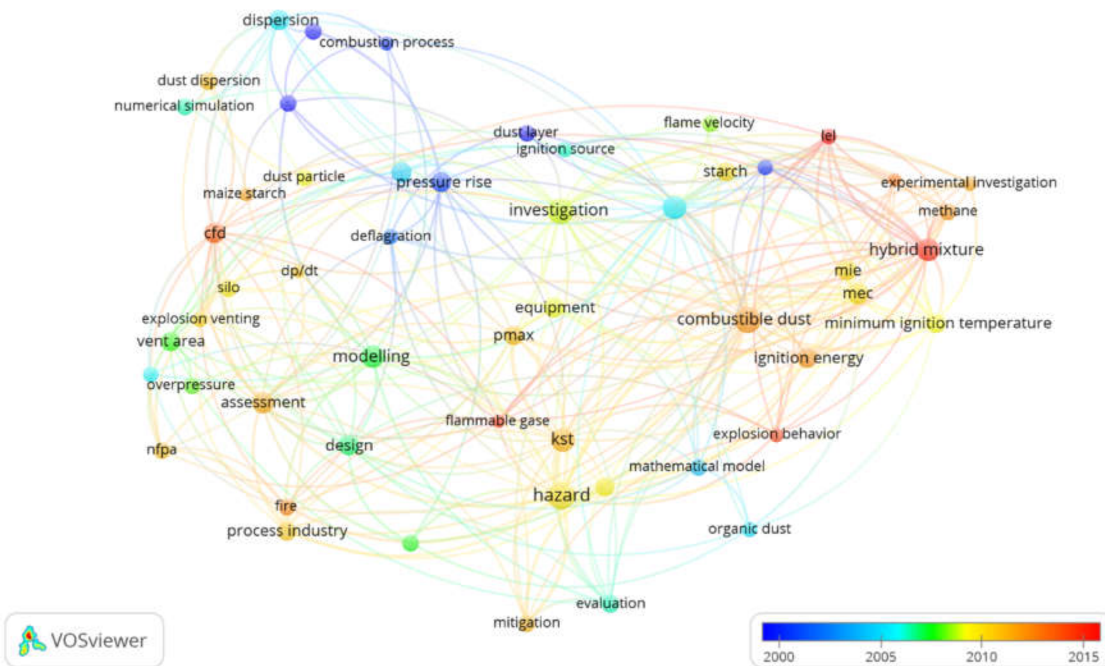

Figure 2-6 - Overlay visualization of dust explosion text data.

By the year 2000, studies were focused on combustion process and issues relates to pressure rise, deflagration with closed vessels. Near to 2005, the subjects were related to explosions inside ducts and pipes, studies of dispersion of the particles and its conection to the combustion process, and some works about mathematical modeling with organic dust. Around 2010, studies were focused on assessing the risk of combustible dust explosion. Several works were focused on the evaluation of the maximum pressures (P_{max}) obtained, the pressure variation with time (dP/dt) and other parameters related to temperature and minimum energy to ignite and trigger an explosion and fires. From 2015 until the present moment, there are some works evaluating CFD (Computer Fluid Dynamics) simulations, others studies analyzed the relationship between lower explosible limit (LEL) and the behavior of explosions and fires with hybrid mixtures.

In density visualization (Figure 2-7), there are several topics that have been well addressed over the years. On the map, there are several hot spots spread but there are two greater groups of hot spots. One is more directly connected with experimental investigations with the parameters like minimum ignition energy, minimum ignition temperature and minimum explosive concentration. In addition, there are also some works about experiments with hybrid mixtures. The other one is related to computational experiments such as CFD simulations in silos to evaluate some risks such as overpressure and explosion venting.

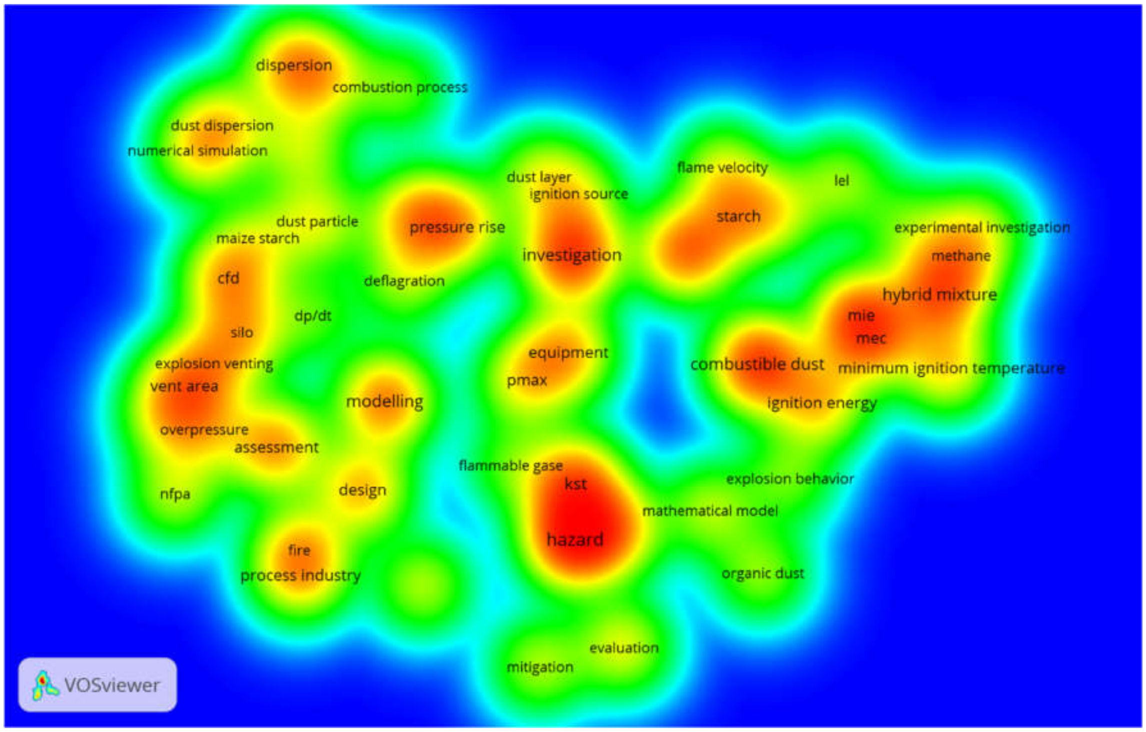

Figure 2-7 - Density visualization of dust explosion text data.

It was created a map with the analysis of authors of the selected works in the bibliographic search. It was developed selecting at least 4 as a minimum number of documents of an author. Figure 2-8 shows the cluster with network visualization of bibliographic and indicates thirteen clusters group connected with thirty-two authors (Table 2-3).

The network view illustrates all the listed authors and the links between them. It is possible to observe that the first five clusters are the greater clusters in terms of document numbers. But only cluster 3, cluster 4, and cluster 5 are more relevant than the others due to the link between the clusters and the number of published documents.

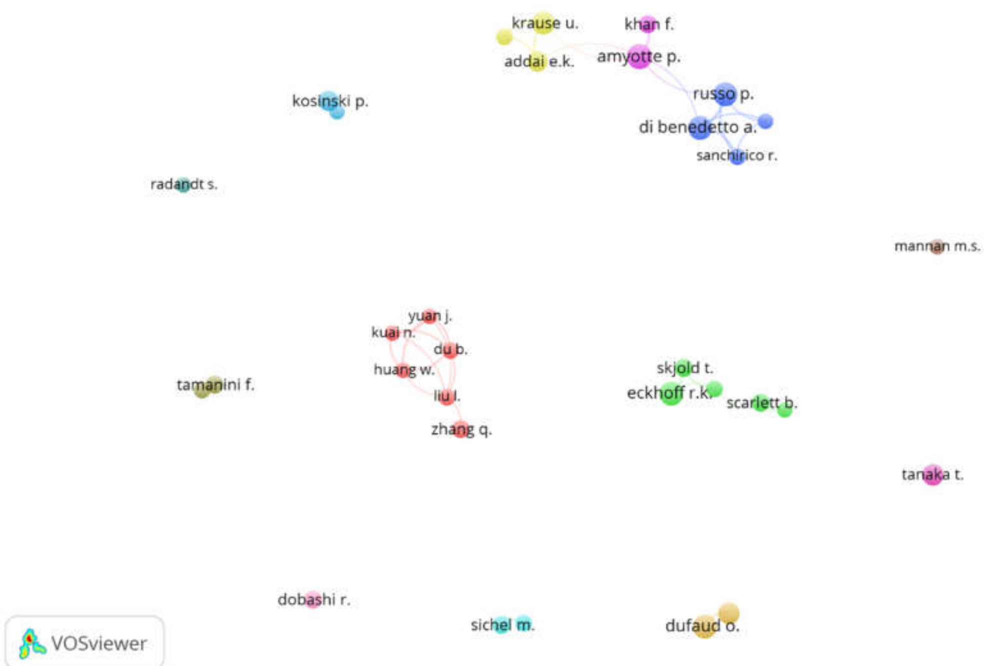

Figure 2-8 - Network visualization of dust explosion bibliographic data (authors).

Table 2-3 - Cluster groups of dust explosion bibliographic data (authors).

Cluster 1	Cluster 2	Cluster 3	Cluster 4
Du, B. Huang, W. Kuai, N. Liu, L Yuan, J. Zhang, Q.	Dahoe, A. E. Eckhoff, R. K. Scarlett, B. Skjold, T. Van Wingerden, K.	Di Benedetto, A. Di Sarli, V. Russo, P. Sanchirico, R.	Addai, E. K. Gabel, D. Krause, U.
Cluster 5	**Cluster 6**	**Cluster 7**	**Cluster 8**
Amyotte P. Khan, F.	Kauffman, C. W. Sichel, M.	Klemens, R. Kosinski, P.	Dufaud, O. Perrin, L.
Cluster 9	**Cluster 10**	**Cluster 11**	**Cluster 12**
Tamanini, F. Ural, E. A.	Dobashi, R.	Mannan, M. S.	Radandt, S.
Cluster 13			
Tanaka, T.			

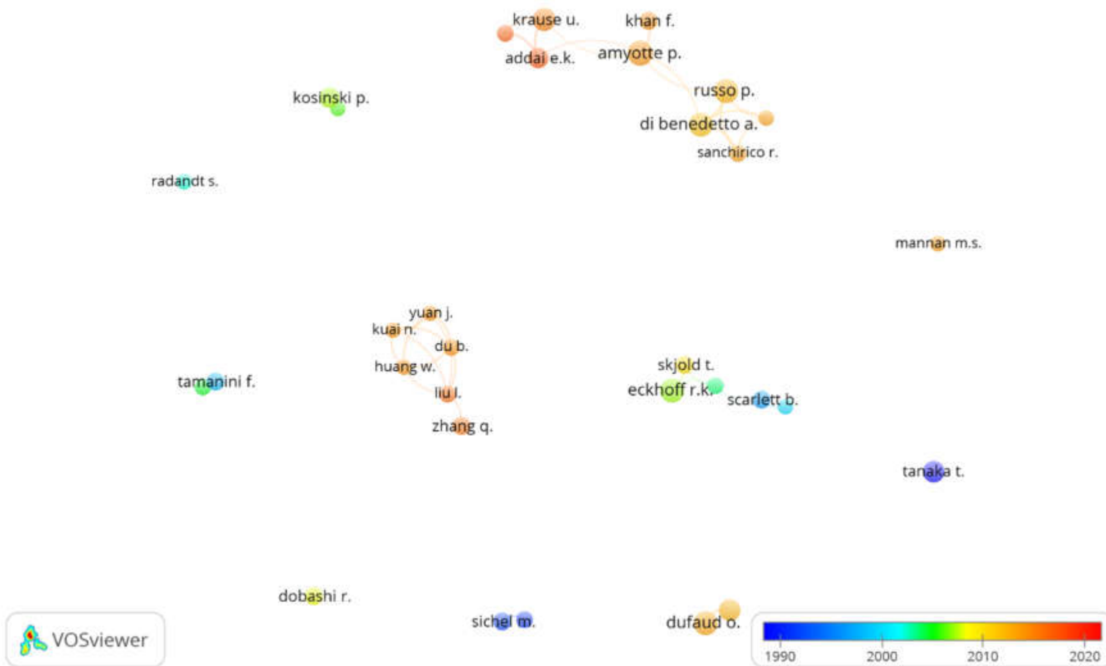

Figure 2-10 - Overlay visualization of dust explosion bibliographic data (authors).

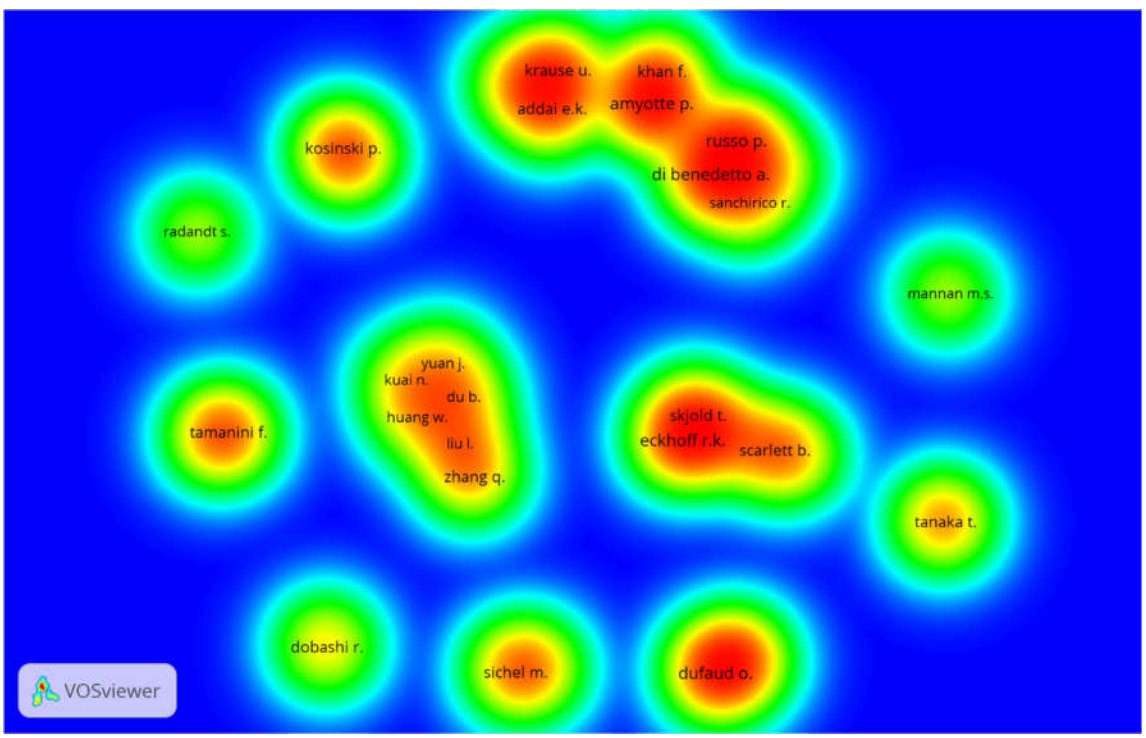

Figure 2-9 - Density visualization of dust explosion bibliographic data (authors).

In the overlay visualization (Figure 2-9), we can verify that the most recent cluster are: cluster 4 that presents publications between the years 2014 and 2016, and the clusters 1 with publications around 2013 and 2015.

Another visualization to check the distribution of the authors according to the publication of documents is the density view (Figure 2-10). The authors with the greatest amount are placed in some hot spots and authors with fewer publications appear with lighter colors like green and yellow distributed throughout the map. As already seen in the network visualization, the first five clusters showed greater relevance that had more intense hot spots, followed by the clusters 6, 7 8, and 9.

2.2.2 Theoretical Basis

As previously mentioned, the structures designed for material storage as the silos have a containment level that favors the development of scenarios for explosions. According to Crowl (Crowl, 2003), an explosion can be understood as the set of three essential characteristics: a sudden release of energy, a rapidly moving blast or shock wave and a blast magnitude large enough to be potentially hazardous. Inside a silo, two possible explosions can occur gas explosion and dust explosion.

During storage of granular or particulate material, flammable gases may be generated from certain chemical reactions within the material pile, which permeate all material until they are placed in the upper part of the silo. With the presence of an ignition source with sufficient energy to ignite these gases, explosions with gases can be triggered inside the structure. For the development of gas explosions, it is necessary that the flammable gases and the oxygen present inside the silo form a mixture within the limits of explosiveness. If this mixture is below the lower explosion limit (LEL) or above the upper explosion limit (UEL), explosions will not occur.

A possible source of inflammable gases are the gases from the pyrolysis reaction, the first step of the smoldering process. During this stage some gases such as CO and CH_4 can be released during the pyrolysis reaction, forming a flammable mixture with the oxygen in the upper part of the silo.

Another possible explosion inside a silo is a dust explosion. It is not a new problem in industrial installations, for over than 300 dust explosions are reported worldwide.

Several accidents involving dust can be found in the literature, and its consequences may be as catastrophic as caused by gas explosions, resulting in deaths, injuries, and destruction of the installations.

For initial combustion, the three requirements are a fuel (grain, powder or dust), an oxidizer and a sufficient source of heat or ignition. However, for a dust explosion, particulate material as dust must be dispersed and developing a dust cloud at the same time that the ignition source is present (Cashdollar, 2000). The combustible material must be able to form a dust cloud that initiates and sustains an explosion. Several factors can influence this first stage influencing the rate of combustion and consequently the spread of the flames. There are some studies regarding the minimum fuel concentration, the limiting amount of oxygen, the influence of particle size, shape and humidity.

According to (Abbasi and Abbasi, 2007) dust explosion is initiated by the rapid combustion of flammable particulates suspended in air. The fuel can be any finely divided solid material, capable of reacting rapidly and exothermically with a gaseous oxidizer. In this case, the fuel is a combustible dust and the particle size is typically in the range 1-100 micrometers (Skjold and Eckhoff, 2016). Smaller particles present a greater risk due to their weight they can be suspended in the air longer.

As previously mentioned, the combustion occurs inside a combustion wave and can be of two types: deflagration or detonation. The primer is almost always present in dust explosions scenario (Lemkowitz and Pasman, 2014). However, a deflagration can develop to a detonation wave due to the effect of flame acceleration when burnt gas expansion increases the turbulent characteristics of the flame resulting in an increase in the mas burning rate (Ogle, 2016).

The propagation of flames in a deflagration wave is carried out by heat conduction. The heat from the hot zone is transferred to the cooler unburned material ahead of the flame and this heating will promote the achievement of ignition temperature, thus igniting and burning the material.

Due to the level of confinement, the rapid oxidation of the dust leads to a rapid increase in temperature and consequently increase in pressure. Both types of combustion wave in a closed structure are characterized by a maximum explosion pressure (P max) and a maximum rate of pressure increase ((dP/dt)max). These two parameters are important for evaluating particulate explosion severity.

The mechanism for dust explosions can be seen in Figure 2-11, which represents the sequence of events that can trigger the primary and secondary explosions. For the occurrence of the first explosion with dust, it is necessary that the dust must be suspended and reaches the minimum concentration in the air that it will become explosive, thus forming a dust cloud that when finding a source of ignition with sufficient minimal energy can be ignited. After the ignition of the dust, the flame propagation and pressure wave process occur and if the place where the dust cloud ignition occurred presents some level of containment, the pressure increase may become sufficiently high to break the structure of the confinement, and thereby trigger the first explosion.

After the first explosion, the following will occur as a cycle with the fuel feeding through the dust layers of any uncontrolled deposits, which will be suspended due to turbulence generated by the pressure wave, being later ignited by the propagation of the flame and allowing the sequence of secondary explosions.

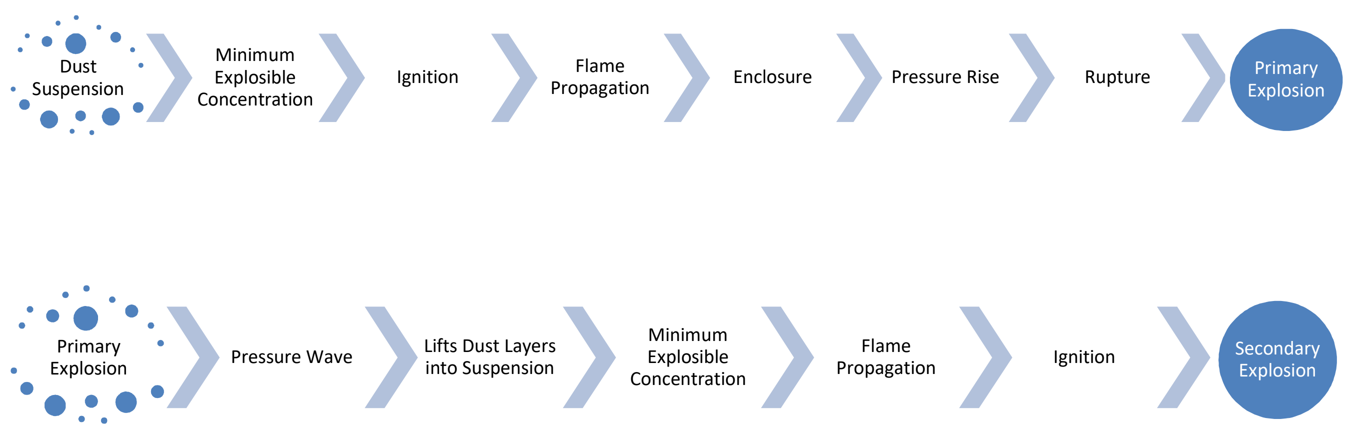

Figure 2-11 - Mechanism of Dust Explosions.

2.2.3 Analysis of explosions scenarios

Regarded to the history of accidents and the severity of the consequences, the dust explosion phenomenon is studied with the objective of improving the comprehension of susceptibility to this latent risk. As more than 70% of the dust processed in the industries are combustible, most of the installations with dust processing equipment are susceptible to explosions with dust (Abbasi and Abbasi, 2007).

Accidents are regarded as a powerful tool in the acquisition of knowledge on the development of explosion, the lessons learned in each incident or accident can assist in managing the associated risks in handling and storing of dust or powder.

Evaluating some accidents and works with risk assessment of explosions in confinement structures as silos, bin, vessels, etc. It was elaborated a fault tree analysis (FTA) as an attempt to comprehend the events related to explosions. Figure 2-12 depicts the possible evolving basic and intermediate events for the development of an explosion.

Analyzing the FTA, we find that there are four intermediate events to trigger the explosions, which are based on the five factors necessary for a dust or gas explosion.

Fuels that feed an explosion in a silo can be flammable gases generated from the steps of smoldering process into the stored material (pyrolysis and oxidation reactions) or dust clouds. However, they need to have particle size within an explosive range and must be in suspension. Dust clouds can be formed during the loading or unloading procedure or resuspended during some inappropriate operation or even after primary explosions where pressure waves can resuspend dust in some connection with the silo.

The oxygen is an element that will always be present inside the silo and to avoid any accident of explosions it is necessary that it be kept below the limit oxygen concentration (LOC). The possible faults related to this element is the failure of the inertization, which may allow the oxygen concentration to be above or near LOC. Another possible scenario of failure is the inadvertent entry of oxygen into the silo which may be either a rupture in the silo or some connection of it, or even a failure in the blocking of the air intake inside the silo.

For an ignition source to become effective it is necessary that it reach the minimum energy required to ignite the material stored inside the silo, this parameter is usually evaluated in experiments by the minimum ignition energy or by the minimum ignition temperature. Among ignition sources, there may be faults in external or internal sources. The latter originates from the self-heating triggered by microbiological action or by hot embers that can fall into the stored material and stimulate self-heating of the material. Among the sources of external ignition, the possible faults are related to flames, eletric current, static electricity, hot solids, and etc.

However, according to Krause (Krause, 2009), some experiments have determined that very high temperatures are necessary to ignite a gas and that either very large areas or very high temperatures are required to ignite dust clouds by means of a hot solid.

The last necessary factor is the partial or total confinement, which is an elementary feature of the structure of a silo. It allows an increase in temperature and consequently an increase in pressure. There are some devices such as vent that assist in the relief of the maximum explosion pressure of the silo, a failure in this device can allow a higher elevation in the internal pressure and a failure in the strength of the structure can cause the silo rupture, and trigger an explosion releasing the accumulated energy

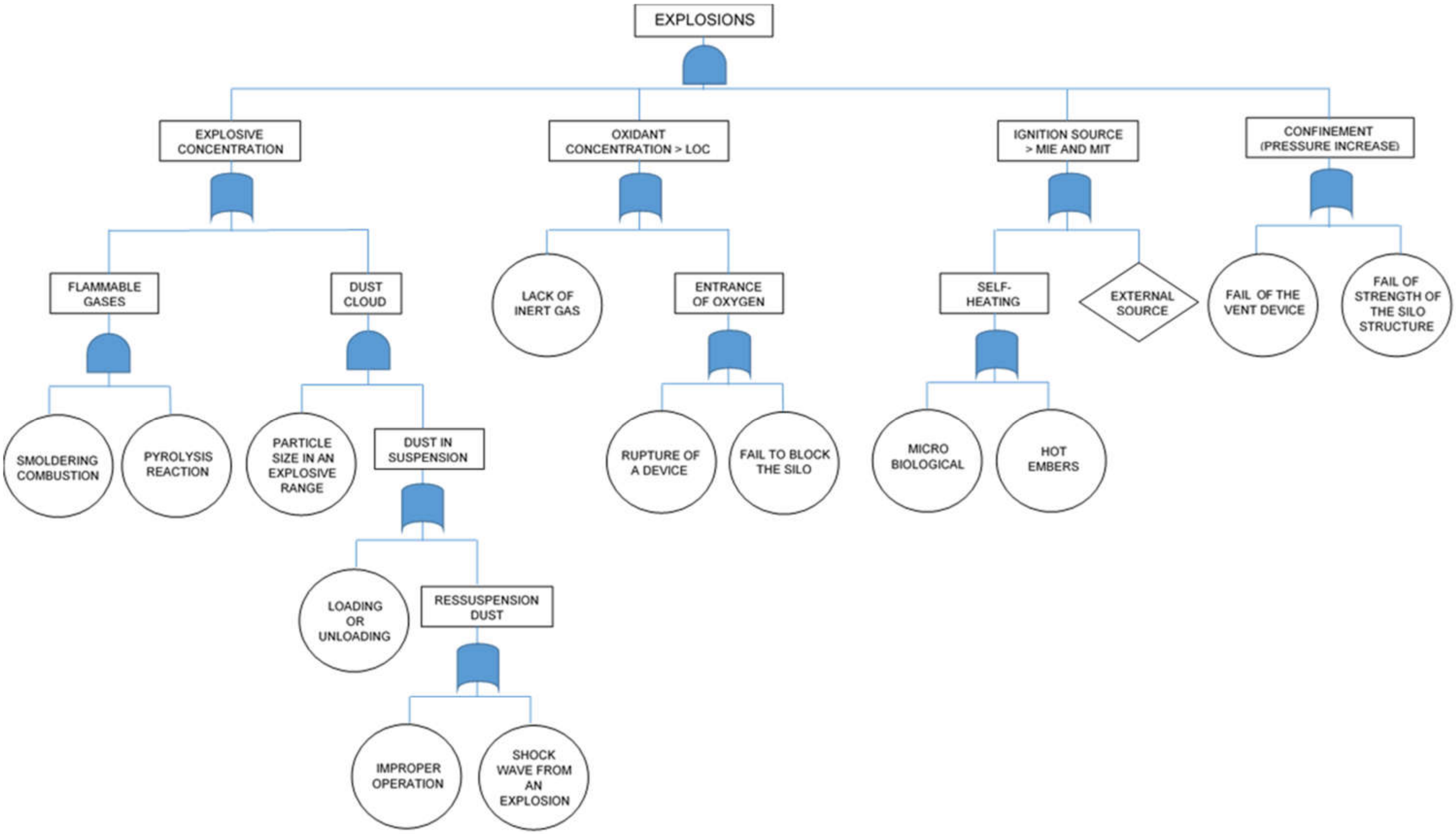

Figure 2--12 - FTA of explosions.

2.3 Smoldering and other fires

2.3.1 Literature Review

It has been studied the issue of smoldering combustion a few years ago. However, studies related to smoldering in particulate materials was made by Palmer in 1957, who measured smoldering velocities and temperatures in small, thin layers of the cork, wood and grass dust.

Leisch, Kauffman, and Sichel (Leisch, Kauffman and Sichel, 1984) examined the combustion, propagation, and ignition properties of horizontal layers of grain, grain dust, and wood dust. In this work, unknown properties such as combustion wave velocity, maximum temperature, charring temperature, combustion zone length, minimum ignition energy, and minimum power per unit area to establish ignition have been measured for grain and wood dust.

Krause and Schmidt (Krause and Schmidt, 1997) in their work they reported experimental investigations on thermal conditions, which may cause or promote an ongoing smoldering process. Glowing nests or heated spherical bodies were used as a source of ignition into the powder deposit of three particulate material: cork dust, beech wood dust, and cocoa powder. The results obtained characterized the smoldering process in terms of smoldering propagation velocities depending on some parameters as the size of the glowing nest or the hot body, size of the deposit and properties of the bulk material.

In addition, Krause and Schmidt (Krause and Schmidt, 2000) did some extensions of their experiments analyzing more sample sizes and measured the oxygen content within the dust sample. An important observation was that the smoldering combustion may be sustained at a very low oxygen content of about 2%. Moreover, some organic materials like wood dust, grain dust, cellulose, etc. contain bound oxygen that can be released during the pyrolysis reaction of the material, thus sustaining the smoldering process.

El-Sayed and Abdel-Latif (El-sayed and Abdel-latif, 2000) performed a series of experiments with corn flour dust and a mixture with corn flour and wheat flour to determine the ignition characteristics for the cases of constant hot surface temperature and constant heat flux. They had concluded that the time to ignition was determined as a function of layer depth, the ignition time increase as layer depth increases. Another important conclusion was related to the amount of energy and the sample size. Dust with high depth and small particle size can be ignited by the same quantity of energy given to dust with low depth and large particle size.

Krause and Schmidt (Krause and Schmidt, 2001) presented a mathematical model to predict propagation temperatures of smoldering fires within dust accumulations. In order to validate it, some experiments were performed to obtain the necessary information on the propagation temperatures and velocities of the reaction front. The model was applied to predict self-ignition temperatures of five different specks of dust: black coal, cork dust, methionine, cocoa powder, and cornstarch. The numerical computations of self-ignition and the experimental data presented a model accuracy of ±5 % or less.

Chong, Chen, and Mackereth (Chong, Chen and Mackereth, 2006) determined some features of milk powder smoldering by observing the generation of CO and O_2 concentrations per unit mass of milk powders and the changes in temperature as milk powder samples underwent external heating with hot air at 300 ºC. Whole milk powder and skim milk powder were analyzed. The first produced significantly higher amounts of CO and attained much higher temperatures than the second one. With the increase of the sample size, the amount of CO generated and the maximum sample temperature increased.

He and Behrendt (He and Behrendt, 2009) evaluated two categories of the smoldering process: forward and reverse smoldering. The objective of the work was to predict and compare theoretically intrinsic characteristics of natural forward and reverse smoldering using the volume reaction method. Results indicated that both categories occurred at very low oxygen fractions (less than 4%). Reverse smoldering was dominated by the kinetics of char reaction oxidation, while forward smoldering was dependent on oxygen diffusion. Regard to propagation velocity reverse smoldering was faster than forward smoldering and the temperature inside the fuel bed was significantly lower than forward smoldering.

The study of (Chunmiao *et al.*, 2013) observed the ignition behavior of magnesium powder layers with four different particle size (6, 47, 104 and 173 μm). A model was developed describing temperature distribution and its change over time while considering the melting and boiling of magnesium powder. This proposed model was in close agreement with experimental data over a wide range of particle size.

El-Sayed and Khass (El-sayed and Khass, 2013) performed some experimental tests to determine the minimum hot surface temperature for rice husk dust ignition, the ignition temperature, and the ignition times. Additionally, the authors evaluated the effects of the dust particle size and the depth of the dust layer on ignition parameters. The rice husk material was sieved into three particle diameters bands: 75-106, 106-120, and 120-150 µm. The results showed that as the initial temperature of the hot plate increases, the maximum temperature of rice dust increases and the ignition delay decreases. In addition, as the dust layer height increases the minimum hot plate temperature for ignition decreases.

He et al. (He *et al.*, 2014) in order to validate the modeling of one-dimensional biomass smoldering and combustion, the authors investigated experimentally some effects as fuel type, moisture content and particle size on natural downward smoldering. To perform the experiments with natural downward smoldering, it was used four materials (pine trunk, corn stalk, activated char, and pyrolysis char), different moisture contents varying between 0 - 21% and the powder was separated using five sieves with meshes of 20, 40, 60, 80, and 100. Results indicated that the fuel type influenced the propagation velocity of the char oxidation front and the moisture content influenced the propagation velocity of the drying front. Additionally, the maximum temperature inside the fuel sample was not sensitive to the variation of fuel type or moisture content. As the particle size decreases, it increased slightly for corn stalk and it was almost unaffected for pine trunk.

Vingerhoets, Snoeys, and Minten (Vingerhoets, Snoeys and Minten, 2016) elaborated some experiments with several organic products, where the size and temperature of the smoldering nest and the available oxygen were correlated with an emission level of carbon monoxide. In order to detect CO emission amount, three set of experiments were performed. The first one, samples of skim milk powder and methylcellulose were stored for several hours in an oven at 200ºC. The second one, an almost equal sample of skim milk powder was installed at the ceiling of an operational spray drying, close to the hot air inlet. The third one different sizes of small skim milk powder samples were inserted in approximately 300°C hot air stream. Results revealed a significant increase in CO emission when the sample was aerated and similar emission levels in industrial process equipment were found in lab test conditions.

Wu et al. (Wu *et al.*, 2017) investigated the heterogeneous reactions of a bituminous coal dust using a novel hot-basket apparatus with an emphasis on the roles of O_2 and diluent gas in smoldering combustion. Experiments showed that O_2 and CO_2 have opposite effects. An increase of O_2 mole fraction accelerated self-ignition and the smoldering combustion. While CO_2 retarded heterogeneous oxidation by decreasing O_2 mass diffusivity, consequently increasing the temperature of ignition and reducing the maximum smoldering temperature. Nonetheless, any small increase in O_2 concentration can compensate the retarding effect of CO_2, since the promoting effect of CO_2 is relatively stronger than CO_2.

Urban et al. (Urban *et al.*, 2017) conducted an experimental and analytical study of how the flaming and smoldering susceptibilities of a powder grass blend in contact with hot metal particles (stainless steel and aluminum) are affected by differences in the particle characteristics, as size and temperature. Both metal particles required the analogous temperature to develop a smolder in the fuel bed. However, results indicated that the ignition could not be determined simply by the initial particle temperature and energy. Additionally, the ignition time for flaming and smoldering after the metal particle impact presented some differences. The timescale of the primer was greater than the other one. The authors also concluded that the effects of parameters related to the metal particle, the fuel (chemical composition, moisture content), ambient conditions (air flow, humidity, and temperature) affect the ignition boundaries reported in the work.

Wu, Schmidt, and Berghmans (Wu, Schmidt and Berghmans, 2018) evaluated the self-ignition parameters of three coal samples in industrial scales. The authors used two steady-state models for extrapolation based on the standardized hot-basket test data, and a comprehensive 2-D model with 2nd-order heterogeneous kinetics considering coal density and oxygen density to predict the self-ignition characteristics of coal dust accumulations. Comparisons between computational results, experiments in lab scales and the extrapolated results based on steady-state methods in industrial scales indicated that numerical model was more reliable than the others to evaluate self-ignition temperature. They concluded that only numerical simulation could be the best way to estimate the induction time of coal samples in industrial scales as a function of the influencing factors as storage temperature, the shape of the deposit, the availability of oxygen and moisture content of the material.

2.3.1.1 A cluster of Literature Review

The subject of smoldering combustion or self-heating of granular or particulate materials has been studied for years by several authors. For a better understanding of how this issue has evolved and continues to evolve over the years, it is important to conduct a literature search. In this work, a bibliographic search was carried out in the Elsevier Scopus database.

For an initial research, we used a set of keywords 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". The key words in this string were selected in order to obtain the largest number of documents related to this subject, which returned 234 documents. Selecting only the documents in the English language reduced the number to 209 documents. After analyzing the titles and abstracts, all that was not related to the keywords and those that did not present all the information in the database were eliminated. Thus, only 97 documents were listed from 1970 to 2018 that is indicated in Table in Appendix A.

For the evaluation of the clusters formed in the topic of the smoldering process of this work, the software VOSviewer was used, in which the list of documents generated by the Scopus database was inserted. This enabled three different types of cluster visualization: network, overlay, and density. In this topic will be presented two clusters of smoldering: a map based on text data and a map based on bibliographic data of authors.

For the analysis of words present in the titles and abstracts of the selected works in the bibliographic search, a term co-occurrence map based on text data was created. This map was developed selecting at least five as a minimum number of co-occurrence terms. Figure 2-13 shows the cluster with network visualization of text data, which indicates four clusters group connected with thirty nine terms (Table 2-4). In this view, it is possible to check the links between the most used terms in the selected works according to the number of occurrences. This allows us to infer how the research is evolving in this topic of smoldering combustion experiments. Another type of visualization is the overlay that allows us to evaluate the variation of the research over the years.

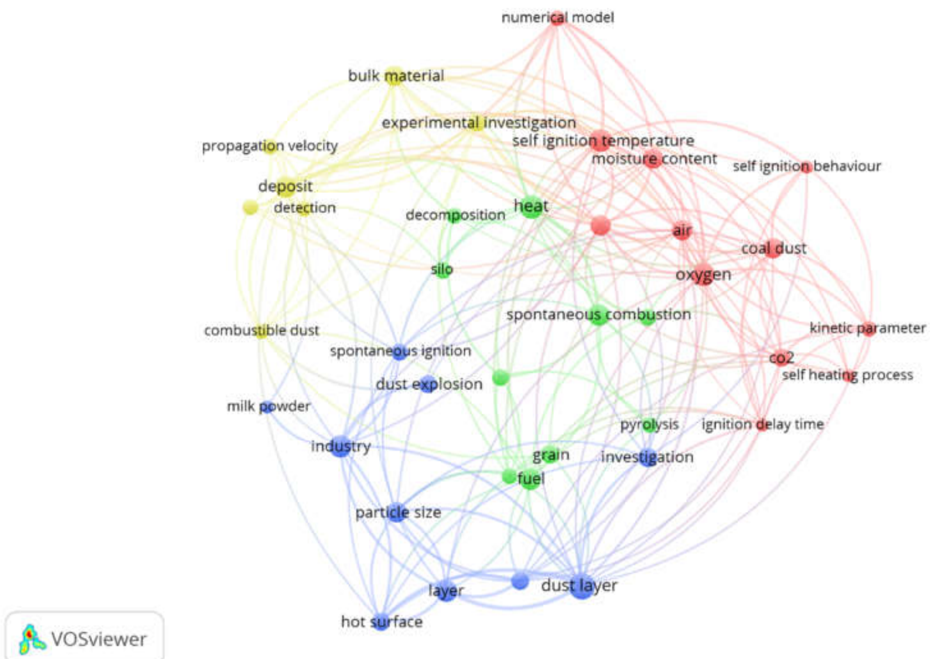

Figure 2-13 - Network visualization of text data.

Table 2-4 - Clusters group of smoldering text data map.

Cluster 1	Cluster 2	Cluster 3	Cluster 4
Air	Biomass	Dust explosion	Bulk material
Ambient temperature	Decomposition	Dust layer	Combustible dust
CO2	Emission	Hot surface	Deposit
Coal dust	Fuel	Industry	Detection
Ignition delay time	Grain	Investigation	Dust deposit
Kinetic parameter	Heat	Layer	Experimental investigation
Moisture content	Pyrolysis	Milk powder	Propagation velocity
Numerical model	Silo	Minimum ignition Temperature	

Oxygen Self-heating process Self-ignition Behavior Self-ignition temperature	Spontaneous Combustion Thermal analysis	Particle size Spontaneous ignition	

In the overlay view (Figure 2-14), we checked which topics were covered over the timescale. It is possible to understand the direction of the research regarding the smoldering process.

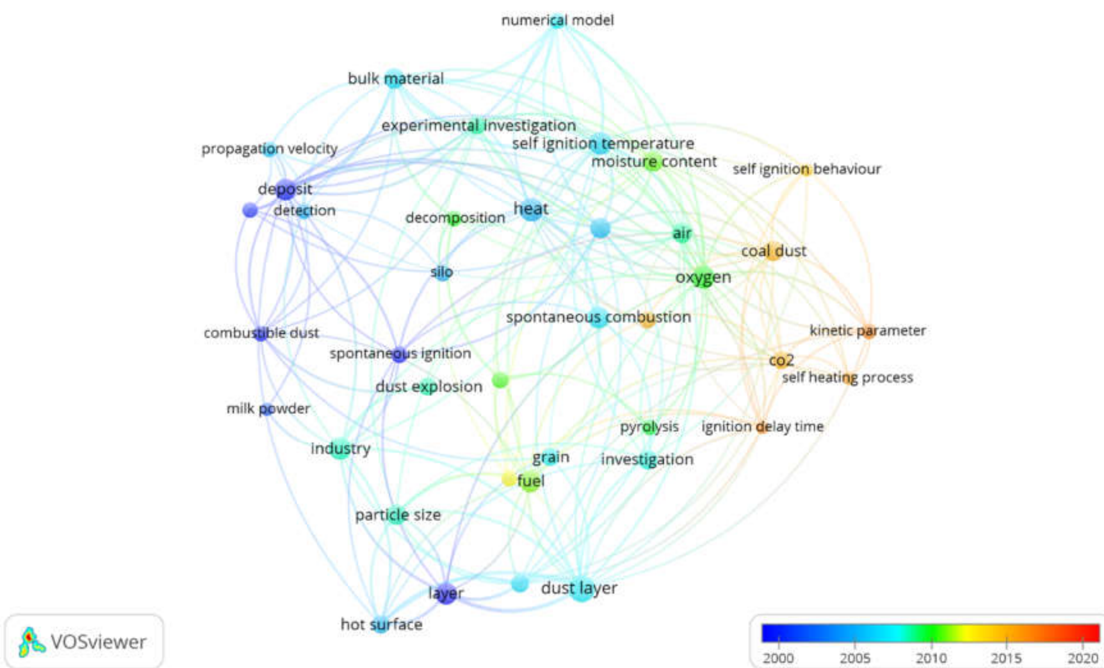

Figure 2-14 - Overlay visualization of text data.

By the year 2000, studies were focused on combustible dust and spontaneous ignition. Near to 2005, the subjects were related to the propagation velocity of bulk material and dust, dust explosions in industries, experimental evaluation of the size of the particles and presented some studies of grain dust, numeric modeling of self-ignition temperature and experiments with hot surface related to dust layers and minimum ignition temperature. Around 2010, studies were focused on thermal analysis related to pyrolysis and decomposition in silos and bulk material. In addition, some experimental investigations with combustible dust in deposits and influence of moisture content in biomass have also been carried out. From 2015 until the present moment, work is presented with numerical models referring to self-ignition, the relationship between the self-heating process and moisture content. In addition, some articles relate the kinetic parameters to topics such as coal dust, CO_2 emission, ignition delay time, self-heating and self-ignition.

In density visualization (Figure 2-15), we note that there are several topics that have been well addressed over the years. On the map, there are several hot spots spread like a self-ignition temperature related to moisture content, the speed of propagation with dust deposits and grain near pyrolysis.

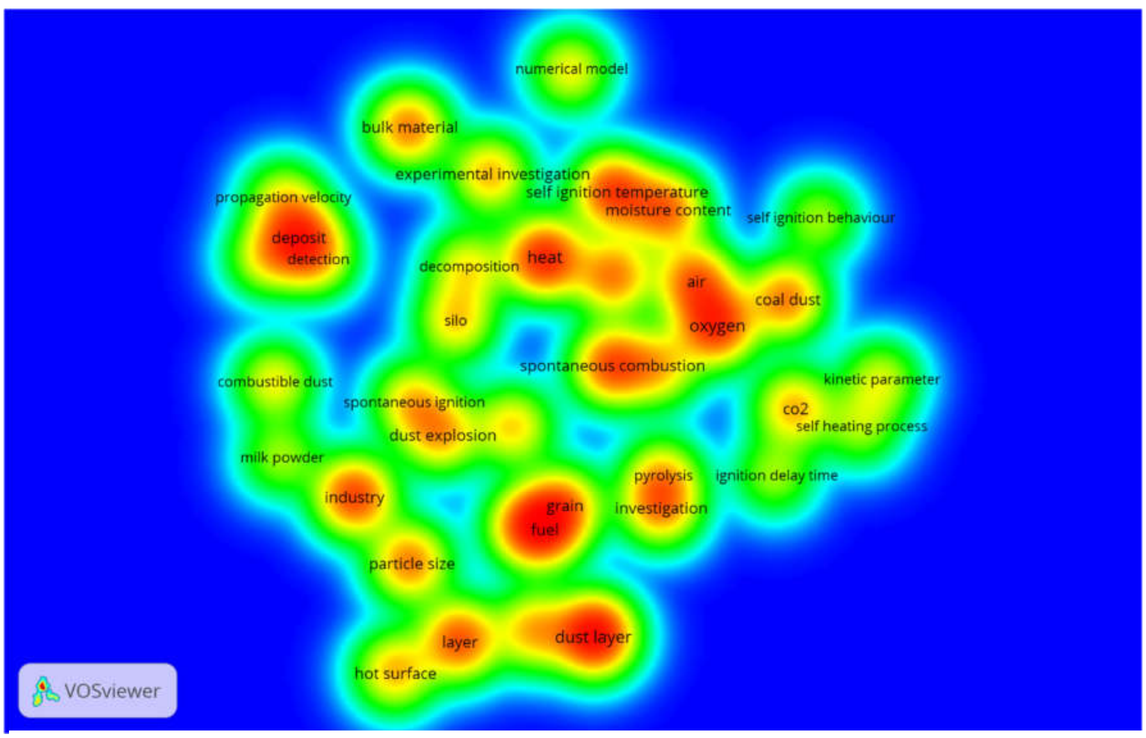

Figure 2-15 - Density visualization of text data.

For the analysis of authors' co-authorship of the selected works in the bibliographic search, a map based on bibliographic data was created. This map was developed selecting at least 2 as a minimum number of documents of an author. Figure 2-16 shows the cluster with network visualization of bibliographic, which indicates sixteen clusters group connected with forty-five authors (Table 2-5).

In network view, we find that there are no links between all listed authors. There are some small groups and two larger groups interconnected (cluster 1 and cluster 3). However, the first six clusters are the most relevant in terms of the number of documents.

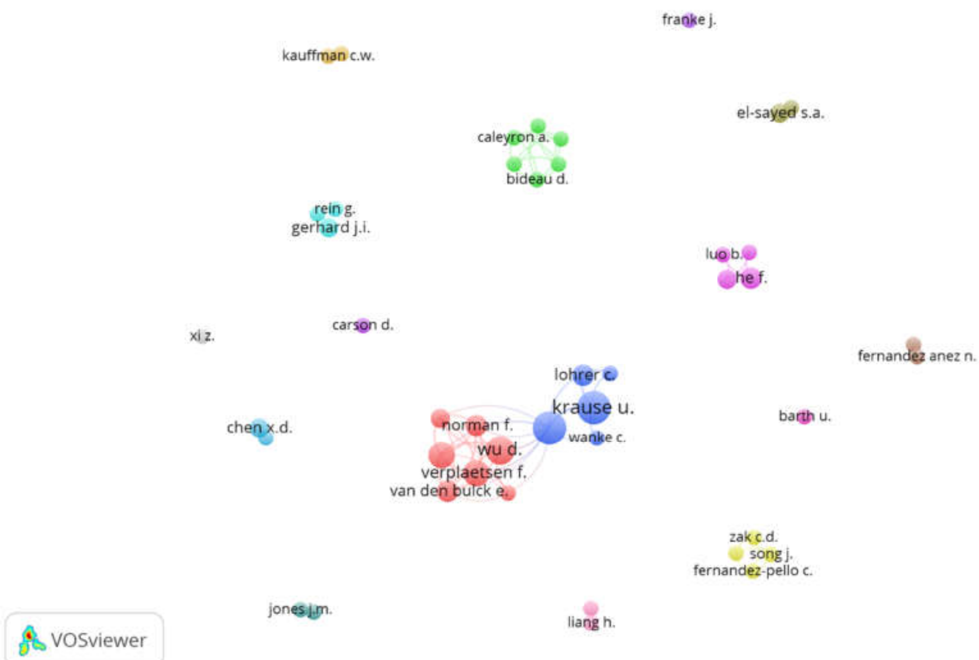

Figure 2-16 - Network visualization of bibliographic data (authors).

Table 2-5 - Cluster groups of smoldering bibliographic data (authors).

Cluster 1	**Cluster 2**	**Cluster 3**	**Cluster 4**
Berghmans, J. Huang, X. Norman, F. Van Den Bulck, E. Vanierschot, M. Verplaetsen, F. Wu, D.	Bideau, D. Caleyron, A. Corriou, J. P. Dufaud, O. Le Guyadec, F. Perrin, L.	Krause, U. Lohrer, C. Schmidt, M. Steinbach, J. Wanke, C.	Fernandez-Pello, C. Song, J. Urban, J. L. Zak, C. D.
Cluster 5	**Cluster 6**	**Cluster 7**	**Cluster 8**

He, F Luo, B. Yi, W. Zha, J.	Gerhard, J. I. Rein, G. Torero, J. L.	Chen, X. D. Chong, L. V.	Kauffman, J. I. Sichel, M.
Cluster 9	**Cluster 10**	**Cluster 11**	**Cluster 12**
El-Sayed, S. A. Khass, T. M.	Liang, H. Tanaka, T.	Fernandez, Anez N. Medic, Pejic L.	Jones, J. M. Williams, A.
Cluster 13	**Cluster 14**	**Cluster 15**	**Cluster 16**
Barth, U.	Carson, D.	Franke, J.	Xi, Z.

In the overlay visualization (Figure 2-17), we can verify that the most recent cluster are: cluster 1 that presents publications between the years 2015 and 2017, clusters 4, 15 and 16 with publications around 2016, and cluster 13 with publications around 2017.

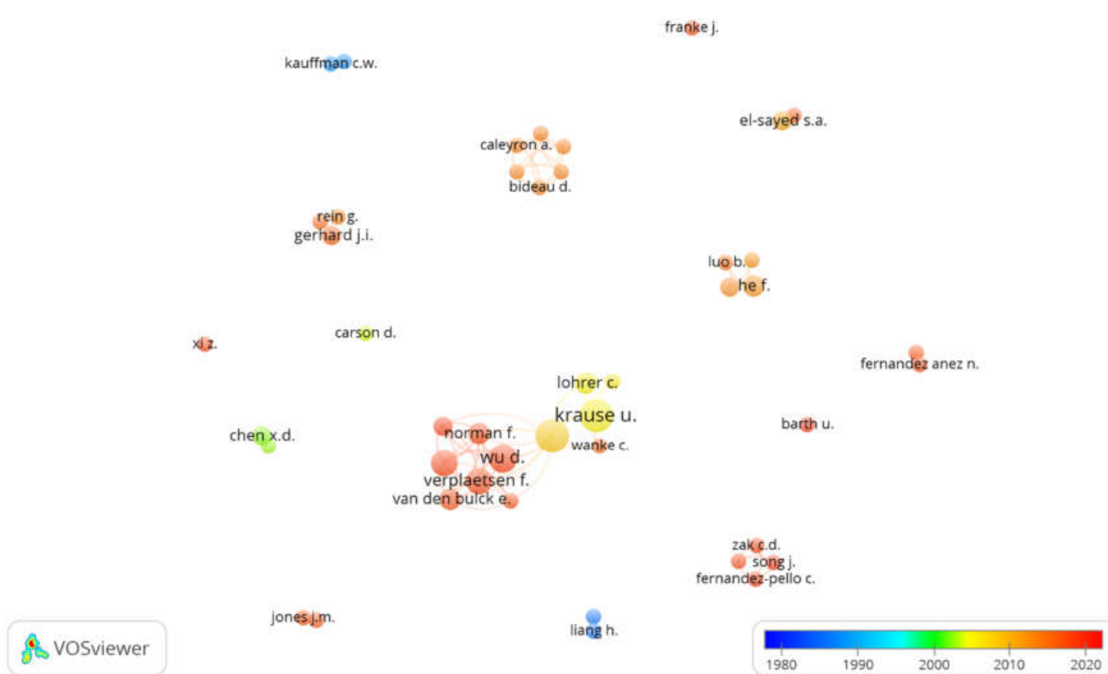

Figure 2-17 - Overlay visualization of bibliographic data (authors).

In the density view (Figure 2-18), we check the distribution of the authors according to the publication of documents. The authors with the greatest amount present themselves in some hot spots distributed throughout the map. As already seen in the network visualization, cluster 1 and cluster 3 showed greater relevance, followed by cluster 2 and cluster 4.

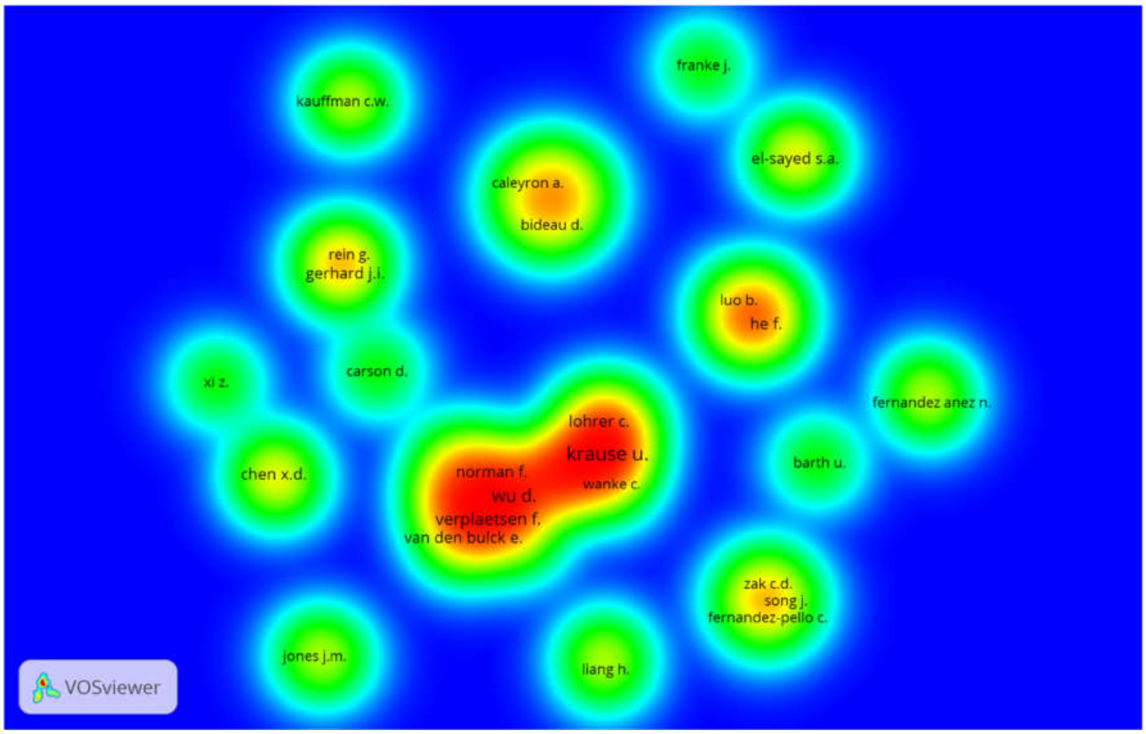

Figure 2-18 - Density visualization of bibliographic data (authors).

2.3.2 Theoretical Basis

Smoldering combustion is a constant hazard in grain silos due to the fact that the stagnated material and physical conditions inside the silo structure are favorable to self-heating and spontaneous ignition (Ogle, Dillon and Fecke, 2014; Russo, Rosa and Mazzaro, 2017). Smoldering could be considered as a combustion wave that travels through a fixed bed of solid particulate material. According to Ohlemiller (Ohlemiller, 1986), smoldering is a slow, low temperature, flameless form of combustion, feed by the heat evolved in the reaction when the oxygen attacks the surface of a condensed phase fuel. Ogle (Ogle, 2016) also defines smoldering as a flameless combustion and points that agricultural grains are susceptible to this scenario of ignition.

As Rein (Rein, 2016) points out, this hazard it is a fundamental combustion problem involving heterogeneous chemical reactions and the transport of heat, mass, and momentum in the gas and solid phases. The smoldering process begins with a pyrolysis reaction producing char, ash, and pyrolysis gases (Equation 1). Then followed by the heterogeneous oxidation reaction with the char product of the pyrolysis reaction, where oxygen reacts with the char (Equation 2). Besides, the hazard of smoldering combustion could exist another oxidation reaction with pyrolyzate gases (Equation 3) which will develop another hazard during the storage, the flaming combustion.

$$Dust\ (s)\ +\ Heat\ \rightarrow Pyrolyzate\ (g)\ + Char\ (s)\ + ash\ (s) \quad (1)$$

$$Char\ (s) + O2\ (g) \rightarrow Heat + Ash\ (s) + CO2\ (g) + H2O\ (g) + Other\ gases \quad (2)$$

$$Pyrolyzate\ (g) + O2\ \rightarrow Heat + CO2\ (g) + H2O\ (g) + Other\ gases \quad (3)$$

The smoldering process presents some basic events and Rein described as four distinct chemical and thermal sub fronts responsible for the structure of smoldering fire, formed by preheating, drying, pyrolysis and oxidation. The pyrolysis follows the preheating and drying sub fronts just when the fuel temperature increases above a certain threshold. Then the oxidation sub front consumes char and oxygen, releasing heat. Besides the sub fronts of oxidation and pyrolysis may overlap in space, and the extent of it will depend on the conditions of propagation and the air flow supply, what emphasize the great influence of the air in the process. Figure 2-19 depict the one-dimensional upward smoldering process.

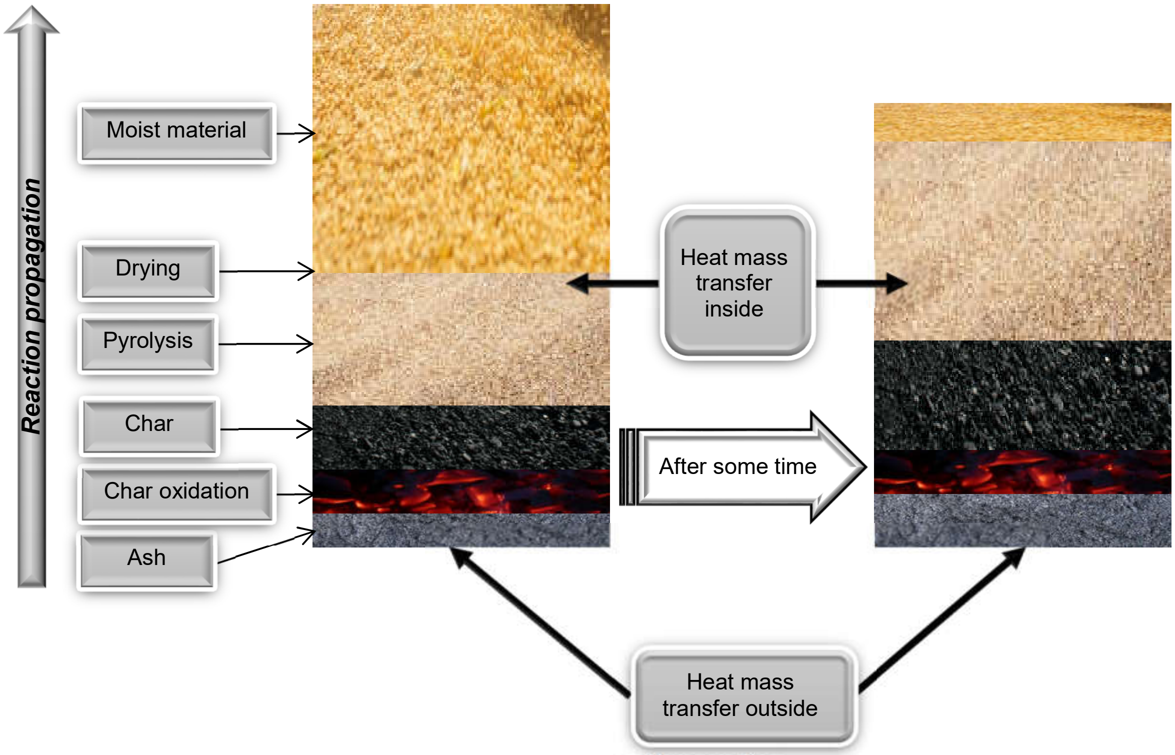

Figure 2-19 - Reaction Propagation.

The occurrence of smoldering combustion depends on four fundamentals factors: fuel with a porous material, oxidizer, the minimum thickness of layer deposit and an ignition source, which can be identified as smoldering combustion square, depicted in Figure 2-20. The material needs to present a porous structure, which will permit the occurrence of the reaction inside of it. In addition, the quantity of oxygen inside and between the particles should be sufficient to sustain the oxidation reaction. The size of the pile is an important influence factor. The pile needs to be sufficiently large to guarantee that the heat released from the exothermic oxidation reaction could be superior to the heat lost to the pyrolysis reaction and could lead to a sustainable combustion. The energy of the ignition source should be sufficient to ignite the pile and initiate the smoldering process, could be an external source or even an internal source as a self-ignition of the stored particulate material.

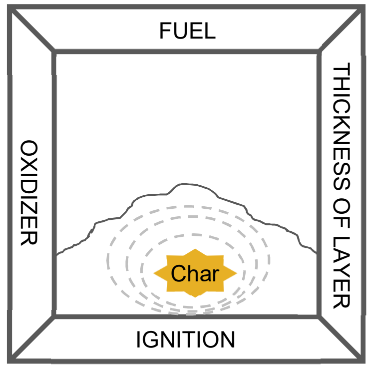

Figure 2-20 - Square of smoldering combustion.

Another important feature of smoldering is that a small mass of smoldering granular material called as glowing nest can be an additional ignition source. There is a number of studies on this topic, but no universally applicable guidance exists. Ogle (Ogle, 2016) elucidated some conclusions:

I - Nests with weights of 15g and temperature of 700 ºC falling through dust clouds initiated some ignitions when achieved the bottom.

II - Tests performed at the BAM oven with nests with a surface area of 75 cm² and 900 ºC indicated that they are able to ignite dust clouds with auto-ignition temperature (AIT) of 600ºC.

III - The risk of nest disintegration in a free fall and create a fireball that ignites the dust cloud is lower because it would fragment before reaching any significant distance.

2.3.2.1 Influencing Factors

Smoldering combustion is a complex process due to the factors that influence directly the process, decreasing or increasing the combustion velocity. They are all related to the smoldering combustion square, which could generate a great variety of influencing factors. In a smoldering combustion in grain, some factors related to the characteristics of the grain, the deep of the layer, type of ignition and variations on the level of oxygen could influence the combustion propagation rate.

The type of the grain, particle diameter, porosity, and humidity are examples of the influencing factors related to the fuel. For a better combustion rate, it is necessary a larger surface area to promote the attack of oxygen at the char surface. Both the particle diameter and the porosity can increase the surface area developing a higher or lower oxygen attack.

Additionally, the particle diameter has an important impact on the level of heat transfer. Between the particles, the heat is mainly transferred by conduction while within the voids the heat occurs by convection. According to Rein (Rein, 2016), in very small particles, the layers of the material exhibit poor internal convection limiting the airflow, which requires a long ignition time. After a specific size, the rate of heat conduction between the particles decreases, also requiring a longer time to ignite.

Humidity also plays an important role in the ignitability of the grain. The level of humidity above a certain level may have an inhibiting influence on the propagation of smoldering. The water present in the voids of the grains will absorb the thermal energy released during the preheating to complete the drying step of the grain. Thus decreasing the amount of energy available for the pyrolysis and oxidation steps of the smoldering process. At levels below this, it can present either an inhibiting or promoting influence. According to Krause (Krause, 2009), some experiments show that organic material with a mass fraction of water above 16%, the fermentation process could be developed inside the stored material within the silo raising the temperature to a level as high as 70 ºC. This reaction with the oxygen present inside the voids of the material represents a favorable condition for self-ignition.

The height of the stored material also has a great influence on the heat transfer between the particles. As previously mentioned, for the development of the smoldering process it is necessary that the energy generated by the process be greater than the energy lost to the environment. The stored material 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 temperature. These heat accumulation results in a sustained increase of the temperature inside the barrier, which accelerate the oxidation rate. Although the occurrence of this acceleration, the smoldering process can take a long time until it becomes self-sustainable. Large piles sizes are favorable to insulate the heat generated and enable this heat buildup.

Additionally, the smoldering combustion also can migrate to a flaming combustion, when the reaction spreads from the interior to the outside of the grain and reaches the free surface. The flammable gases released during the process can reach the lower flammability limit and ignite with smoldering.

With regard to ignition, all source of ignition requires the supply of heat to develop an ignition. The process of smoldering ignition is primarily governed by heat transfer and reaction kinetics, with the oxygen supply representing a secondary role. Initially, the heat supply is commenced with the self-heating of the grains through exothermic chemical reactions releasing heat or with a hot ember inside the grains. After a certain level of heat, the endothermic step of pyrolysis is initiated and then is followed by the char oxidation. When the heat released by oxidation is higher than the heat required for the endothermic processes and heat losses, propagation will follow and the reaction might become self-sustaining. Thereafter the oxygen supply rate will develop an important part, supplying the oxidation reaction.

Although the importance of the oxygen supply rate to be greater only after the smoldering reaction be self-sustained, the oxygen level between the particles and inside the voids is critical at the beginning of the reaction to start the oxidation. There is an ideal oxygen limit to start the oxidation reaction and then develop the smoldering. Air ventilation can supply the required oxygen to the reaction but can also dissipate the heat generated delaying or extinguishing the smoldering process.

2.3.3 Others Fires

In addition to smoldering, there are other fire hazards in silos. The flammable gas generated through pyrolysis, the first stage of the smoldering process can become an explosion or flash fire hazard. Another fuel source for this hazard is related to the formation of small dust clouds during normal procedural operations in silos.

A mass of flammable gases or a mass of a combustible dust cloud dispersed inside a silo creating an explosive concentration can be ignited and results in a flash fire. This hazard is a deflagration that does not lead to a rise in pressure, thus does not rupture the storage structure and does not develop a gas or dust explosion. However, according to Ogle (Ogle, 2016), this hazard requires that the confinement volume must be much larger than the volume of the unburnt gas or dust cloud.

Some factors will influence the severity of the flash fire hazard in silos as the size of the dust deposit, the propensity to generate flammable gases and the potential for dust dispersal.

2.3.4 Analysis of fire scenarios

Evaluating the gas phase and the solid phase inside a silo, we observed that in the two phases there is the possibility of combustion. In the solid phase, there is a cycle between the reactions of pyrolysis (thermal degradation) and smoldering (oxidation of char). As mentioned earlier, the pyrolysis reaction generates char and inflammable gases as products. The first will feed the oxidation reaction that will generate more inflammable gases and the heat needed to sustain the pyrolysis reaction. Another cycle is related to the combustion of inflammable gases (volatile products) originated from the reactions in the solid phase, which in the presence of oxygen of the air and a source of ignition generate gases of combustion as CO, CO_2 and H_2O. The heat generated in the gas phase also supports the reaction in the solid phase. Figure 2-21 exemplifies the reactions in the gas and solid phases and their interactions for the development of the fire scenarios.

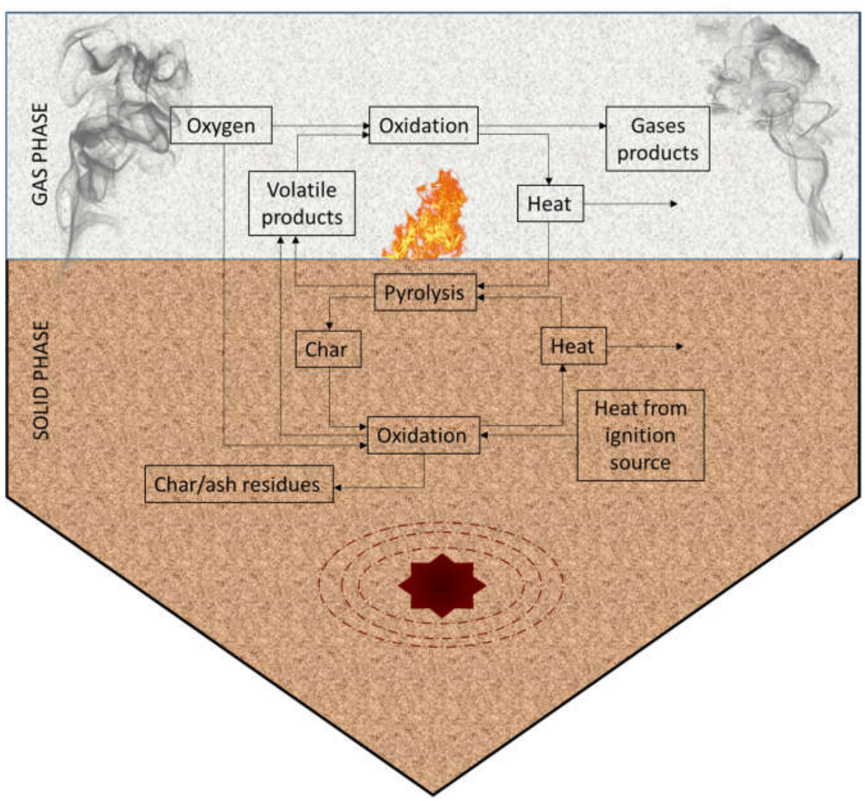

Figure 2-21 - Process diagram of reactions in silos.

A simplified FTA was developed with the objective of identifying the sequence of events related to fires in silos as shown in Figure 2-22. As mentioned before, fire scenarios require three factors to initiate a combustion reaction: oxygen, fuel, and a source of ignition.

The oxygen concentration should be above the minimum level to feed this reaction. Possible failures are a lack of inert gas inside the silo during operations or the entry of oxygen into the silo due to a rupture in the estructure or a failure in the seal.

In relation to the fuel, it can be from gas phase or solid phase. The gas phase must be between lower and higher flammability limits, and may be originated from both char oxidation reaction and pyrolysis reaction. The basic event of the origin of the gases is basically from the smoldering process or from the pyrolysis step as in the case of dust clouds and the settled layer. Another fuel possibility is the pyrolysis char in the solida phase achieved during the oxidation reaction in the smoldering process.

The energy of the ignition source should be greater than the minimum ignition energy (MIE) and can be an external source or a self-heating due to microbiological action or hot embers embedded into a granular or particulate material.

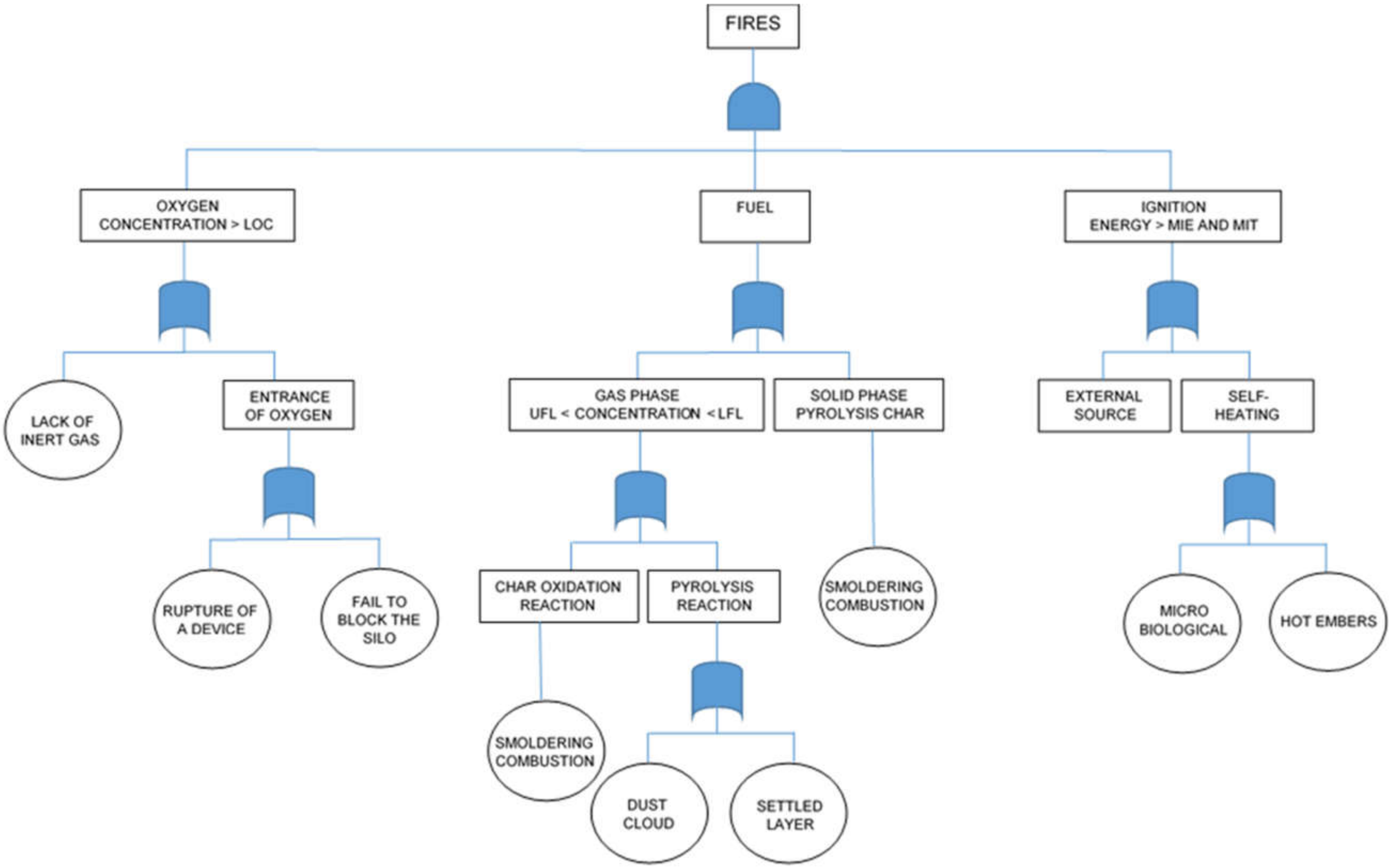

Figure 2-22 - FTA of fires in silos.

3 METHODOLOGY

In this work, it was experimentally analyzed one of the risks in silos storage, the smoldering process. The methodology was distributed in four steps composed of the phases planning, preliminary, performance, and evaluation, exemplified in Figure 3-1.

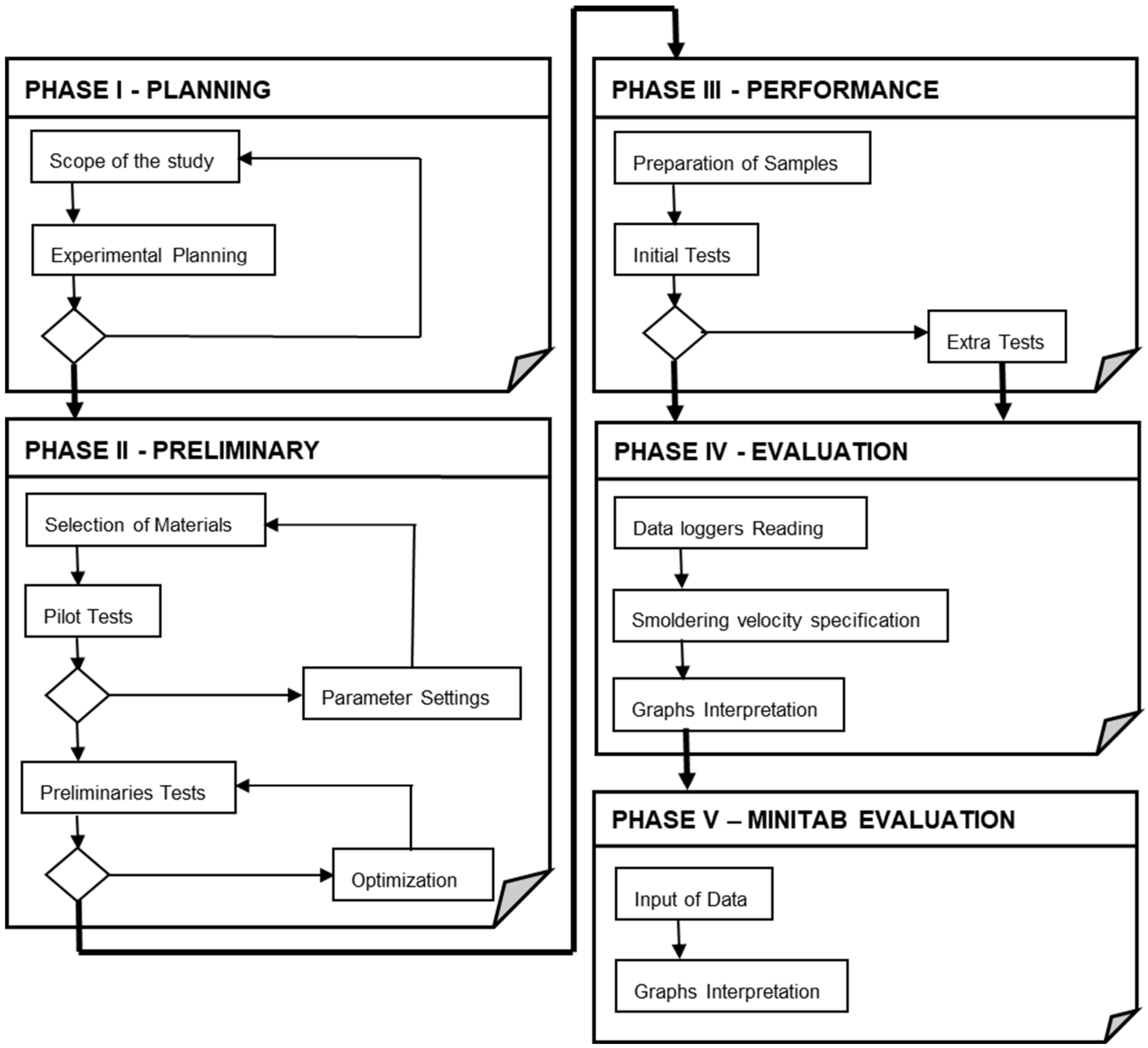

Figure 3-1 - Methodology of the experimental tests.

At the first phase, it was planned the scope of the smoldering study and then designed a proposal of experimental planning with grain. Before begin the experiments, it was defined the minimal quantity of experiments using the Design of Experiments (DOE) via Minitab software. DOE helps to investigate the effects of input variables (influencing factors) on output variables (response) at the same time. These experiments consist of a series of tests, in which deliberate changes are made to input variables. At each test, data are collected. DOE helps to identify process conditions that affect the response variable, and then determine factor configurations that optimize results. After conducting the experiment and entering the results, Minitab offers several graphical and analytical tools to help the comprehension of the results.

Among the potential experiments presented in Minitab software, in this work we selected the full factorial experiment in which three input variables were selected with two possible values to evaluate the behavior of smoldering combustion in grains. It was established two replicates, then it was defined sixteen as the minimal quantity of experiments. It was proposed to use two different values of particle diameter, humidity, and ventilation condition.

As defined in the proposal of experiments, the experimental apparatus was based on the European Standard EN-50281-2-1: 1998. This norm specifies the experimental methods to determine the minimal inflammation temperature of thin dust layers on a heated surface, usually the sample thickness are 5 mm, 12,5 mm or 15 mm. The temperature evolution is measured by a thermocouple inserted in the center of the dust sample. In this study, a slightly modified EN-50281-2-1 test setup was used to analyze the problem of smoldering combustion inside a mass of particulate material or dust. The experimental apparatus was composed by a hot plate, a cilindrical structure to accommodate the dust instead of a ring and some thermocouples were located along the central axis instead of just one at the center to monitor the temperature variations within the dust mass.

At the preliminary phase, the goal and the scope were clarified, the experimental material defined was a grain commonly stored in silos and with a level of combustibility to cause a smoldering or an explosion hazard, as corn grain. Some pilot tests were performed with some corn dust to verify the process and to guarantee the smoldering combustion. Then, some adjustments were required in the hot plate and thermocouple configurations. Afterwards all the preliminary tests and implementations for optimization, the design of the experimental apparatus was validated for the actual test.

In the third phase, all the samples were separated into two different diameters and placed a fraction in a desiccator and another fraction in a climatic chamber. Defined by the software Minitab, the sequence of the experiments was followed and some extras experiments were done to evaluate some variation in ventilation condition. Besides, a complementary experiment were performed to evaluate the ignitability features of the particulate material used.

In the fourth, the data obtained with data logger readings were evaluated. At this level, it was possible to elaborate some graphs to evaluate the maximum temperatures reached in each thermocouple and the smoldering speed in each experiment.

In the fifth phase of this methodology, the data obtained during the experiments with the hot plate were inserted into the Minitab software to experimentally evaluate the relations between the influencing factors and the smoldering speed.

The main objectives of the experiments were to evaluate the smoldering velocity in different conditions and analyse the ignitability features of the particulate material used.

3.1 Preparation of the samples

It was necessary the search for a large number of samples with different particle sizes for performing the tests. To accomplish the planned experiments, two large samples of maize from a grain producer in Spain were obtained, one of the samples was corn flour, and the other sample was broken corn grains. To obtain particle diameter smaller than the broken corn grains, the grain was crushed in a grinder and later sieved with sieves with 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.

Figure 3-2 – The three particle sizes of the experiments – corn flour, grain 0.5, and grain 1.0.

After selecting the three particle sizes (Figure 3-2), a small sample of each size was analyzed by Beckman Coulter LS Particle Size Analyzer. It is a sophisticated laser diffraction analyzers used to determine the particle distribution and to estimate the particle diameter of each sample. Figure 3-3, Figure 3-4, and Figure 3-5 show the particle diameter distribution of the samples selected of the corn flour, the grain separated with the sieve of 0.5 mm and the grain separated with the sieve of 1.0 mm. The complete document of the analyzes is found in Appendix B.

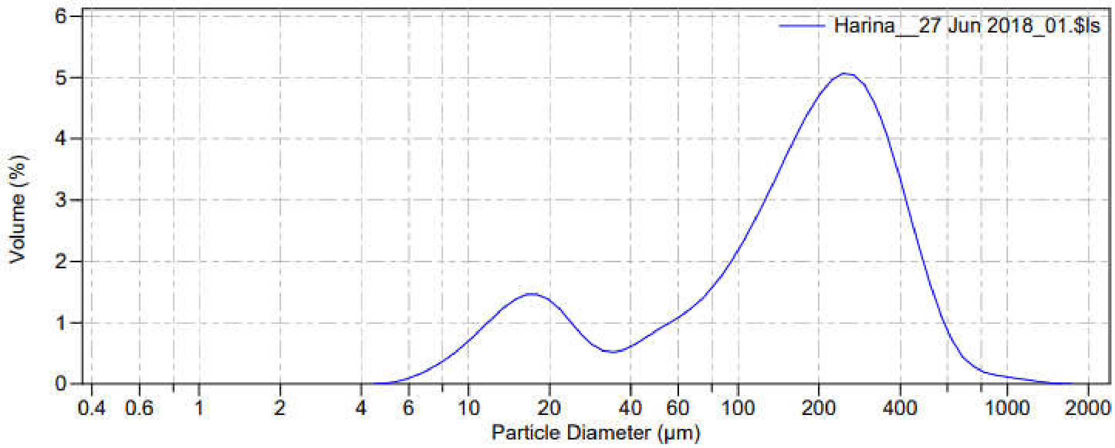

Figure 3-3 - Particle diameter distribution of corn flour.

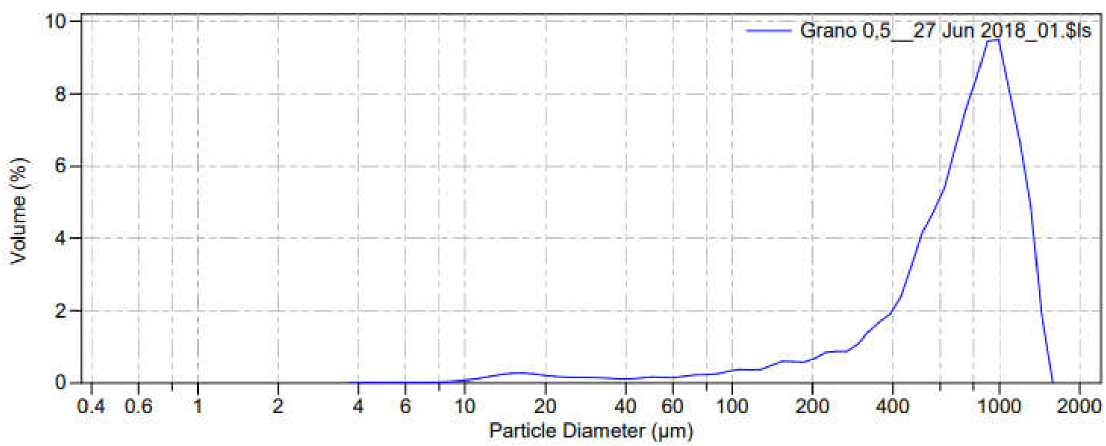

Figure 3-4 - Particle diameter distribution of grain sieved with 0.5 mm.

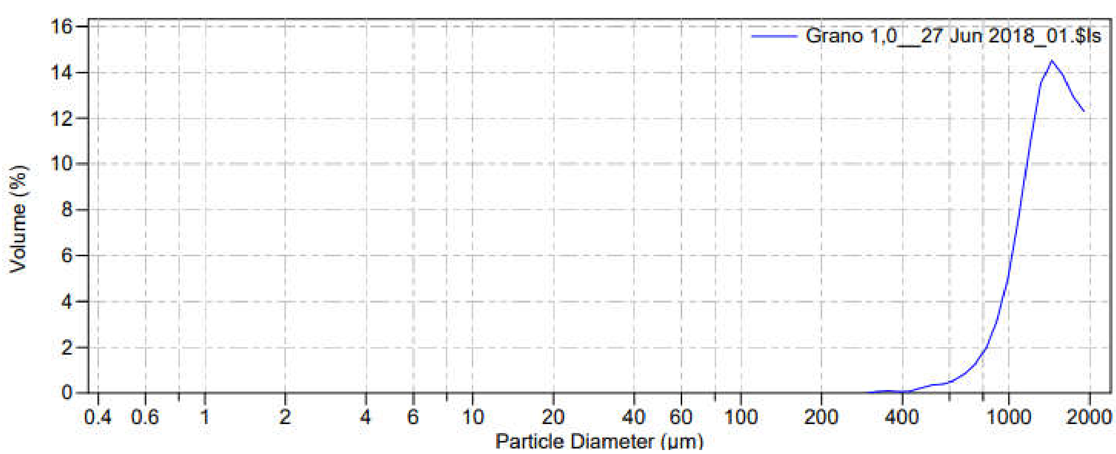

Figure 3-5 - Particle diameter distribution of grain sieved with 1.0 mm.

Table 3-1 shows the diameter of the particles d10, d50, and d90 for the samples analyzed.

Table 3-1 - Particle diameters of the samples.

SAMPLE	D10 (µM)	D50 (µM)	D90 (µM)
CORN FLOUR (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

The samples selected for the main experiments were those of corn flour (F) and grain 0.5 (GS). However, experiments with grain 1.0 (GL) were also performed. The samples were separated into two environments. The first one was a climatic chamber with humidity of 60% and a temperature of 16ºC. The other was a desiccator with a temperature of 100ºC. All samples were left for at least 3 days in these environments to ensure the same moisture and in the case of the desiccator to ensure that the samples were totally dry.

3.2 Experimental apparatus and the methodology of the tests

3.2.1 Experiment 1 – Hot plate

The validated experimental apparatus was composed by one hot plate connected to an electric panel, a cylindrical structure to place the samples, a system of ventilation, thermocouples and data loggers.

At the stage of the preliminaries tests, the experimental apparatus was tested with both diameters of corn grain and the samples were totally dry. As an ignition source was used a hot plate of 1500 W that could achieve temperatures higher than 400ºC. It was connected and controlled by an electric panel which was settled to achieve 550 W of power with the objective of reducing the variation of temperature during the experiments. This value of power can reach approximately 350ºC and guarantee that this temperature will continue the same during the experiment.

For the tests, it was designed a cylindrical structure with small holes and with topside and bottom side open to permit a better contact of the dust with the oxygen in the air and with the hot plate. The structure with 8 cm of diameter and 12 cm of height was composed by three layers, two of steel and one of aluminum. In each experiment was used the same height of the dust samples.

The temperatures were measured by eight type K thermocouples; one above the hot plate and seven distributed in different positions in the central axis of the structure. The values in each thermocouple were recorded every minute until the end of the experiments with the support of two data loggers. The first thermocouple was placed 0,3 cm away from the hot plate, and the others were placed 1,5; 3,0; 4,5; 6,0; 7,5 cm away from the hot plate.

The experimental apparatus was allocated in a specific area equipped with an exhaust gas system. The hot plate was left at full power and then connected to the control panel, which allowed the final power to be controlled only by the panel. The cylindrical structure was placed in the center of the plate, and the thermocouples were adjusted in the respective positions, seven allocated in the central axis of the cylindrical structure and one allocated on the plate. For tests with air ventilation, the system with a compressor and a conical nozzle was positioned 14 cm away from the cylindrical structure. The air velocity during the tests was measured with the support of an anemometer, which indicated a velocity of 0.1 m/s. Figure 3-6 shows a representation of the experimental apparatus.

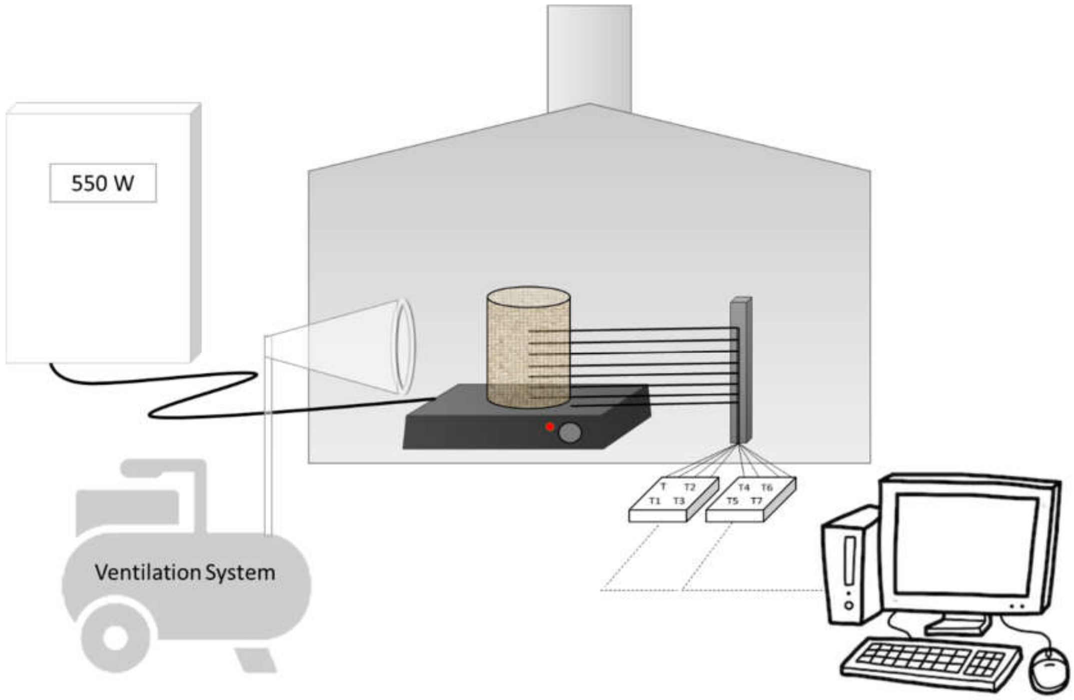

Figure 3-6 - Experimental Apparatus.

The samples were placed carefully inside the cylindrical structure with the support of a funnel, trying to avoid that the compaction of the samples. Thereafter, the controller panel was turned on and maintained at 550 W for 4 hours. For the ventilation tests, the system was switched on at the same time as the heating and it was also maintained for 4 hours. The recording of the experiments continued until the temperatures on all thermocouples dropped to room temperature.

Also to corroborate this experiment, a similar test set up and methodology to evaluate the velocity of the smoldering front of corn stalks was defined by Palumbo et al (Palumbo *et al.*, 2017). A specimen of 40x40x160 mm was placed on a hot plate with five type K thermocouples placed every 3 cm along the sample central axis .The heating was conserved until a predetermined temperature between 280 ºC and 330 ºC and then it was allowed to cool to room temperature. Afterwards, it was defined the smoldering velocities for the samples that the temperature was sufficient to initiate the smoldering process.

According to EN-50281-2-1, tree prospects of the temperature variation in time to the ignition in a layer of dust on a hot surface at a given temperature are possible. To characterize a smoldering combustion is necessary a behavior of the temperature evolution similar to the evolution indicated in Figure 3-7. The ignition should be considered to have occurred in these three possibilities shown. If it is observed a visible glowing or flaming, or a measure of a temperature inside the dust layer of 450º C, or a temperature rise of 250K above the hot plate temperature.

This norm also points out that with organic dust combustion will usually take the form of charring followed by the smoldering process with glowing that will progress through the layer and leave an ash residue. After performing the experiments, the graphs plotted with the evolution of the temperature over time in each thermocouple should exhibit a similar behavior in cases where the smoldering process occurs.

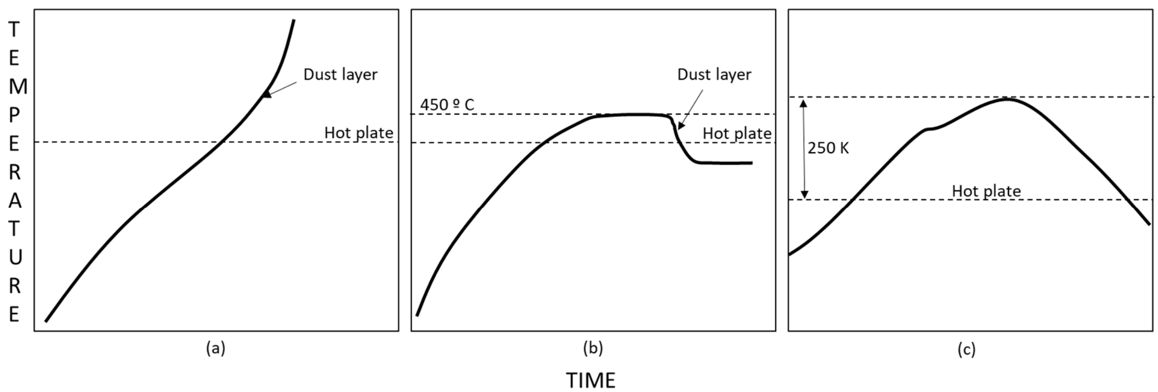

Figure 3-7 - Typical temperature-time curves for ignition of dust layer on the heated surface.

3.2.2 Experiment 2 – Radiator device

As complementary tests of phase III, some experiments were carried out to verify the ignitability of the materials used in the experiments with the hot plate. The experimental apparatus for these experiments was composed by a radiator device connected to an electric panel, a metallic box, one type-K thermocouple, and a data logger.

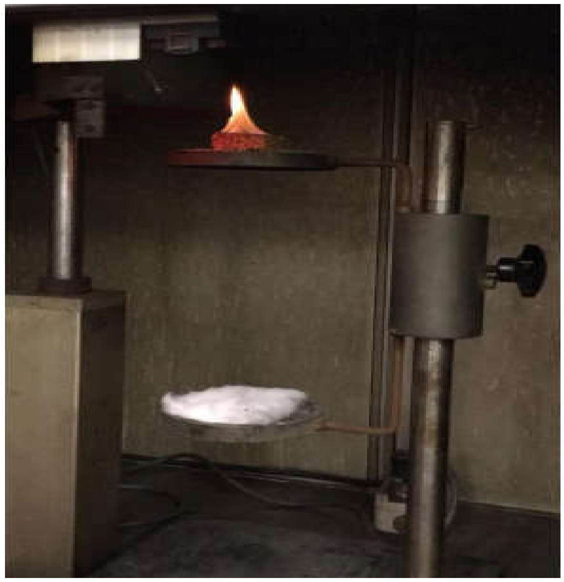

Figure 3-8 - Experimental apparatus of Ignition time and extinguishability

The samples of the selected materials were placed on a square metallic box of 7cm and height of 1cm. According to the Spanish Standard UNE 23725:1990, the samples should be placed 3 cm below a heat source of 400W, which was removed 3 seconds after each ignition and replaced after each extinction, during the 5 minutes of the test. Additionally, the thermocouple was positioned right above the sample to evaluate the variatios of temperature every 5 seconds through the experiment. The most important informations determined were the time of the first ignition, the number of ignitions and the average value of combustion extent during the 5 minutes of the test.

4 RESULTS AND DISCUSSION

In this work, two experiments were carried out concomitantly. The first one evaluated the time evolution of the smoldering in the different types of samples. For this was used as the basis for the realization of the experiments the standard EN-50281-2-1: 1998 referring to tests for evaluation of the minimum ignition temperature of layers of dust on a heating plate. The second experiment evaluated the time the sample continued to burn and the amount of ignition during the 5-minute interval in the different sample types. This experiment was based on the UNE 23725:1990 titled as a reaction to fire tests of building materials. dripping test with electrical radiator used for melting materials.

4.1 Experiments 1 - Smoldering evolution with a hot plate

As indicated in the methodology of this work, the number of experiments was determined according to the DOE in Minitab. Eight experiments were defined as varying three influencing factors and to ensure a better reproducibility the tests were performed in duplicate. Table 4-1 presents eighteen experiments suggested by the software.

Table 4-1 - Experiments tests.

StdOrder	RunOrder	CenterPt	Blocks	Diameter (µm)	Humidity (%)	Ventilation (m/s)	Code
8	1	1	1	776.4	15	0.1	GS-Wet-V_1
3	2	1	1	181.6	15	0	F-Wet-NV_1
12	3	1	1	776.4	15	0	GS-Wet-NV_1
14	4	1	1	776.4	0	0.1	GS-Dried-V_1
7	5	1	1	181.6	15	0.1	F-Wet-V_1
15	6	1	1	181.6	15	0.1	F-Wet-V_2
11	7	1	1	181.6	15	0	F-Wet-NV_2
6	8	1	1	776.4	0	0.1	GS-Dried-V_2
13	9	1	1	181.6	0	0.1	F-Dried-V_1
9	10	1	1	181.6	0	0	F-Dried-NV_1
2	11	1	1	776.4	0	0	GS-Dried-NV_1
4	12	1	1	776.4	15	0	GS-Wet-NV_2
10	13	1	1	776.4	0	0	GS-Dried-NV_2
16	14	1	1	776.4	15	0.1	GS-Wet-V_2
1	15	1	1	181.6	0	0	F-Dried-NV_2
5	16	1	1	181.6	0	0.1	F-Dried-V_2

During the tests, it was necessary to perform extra tests varying the conditions of these three factors due to failures in the experimental apparatus and data recording. However, only the result of sixteen tests was used in the Minitab evaluation. Besides the tests executed with ventilation of 4 hours, other tests were performed varying the ventilation time with 1, 2 and 8 hours.

All experiments followed the experimental methodology as indicated in the previous chapter. At the end of each experiment, the data recorded by the data loggers were plotted on graphs showing the temperatue evolution in each thermocouple over time. Figure 4-1 shows a sequence of photos taken during a test with dried corn flour without the ventilation system. After a few hours, the samples with corn flour, at the top of the sample appeared some cracks (B). This behavior was not observed in the larger grain sizes. During the tests, the samples with corn flour produced a lot of smoke (D) and it was also visibly detected the appearance of a glowing nest in some cases (C). At the end of the tests, in the case with corn flour there were many ashes (E) and in the case of the two grain types there were ash and char residues.

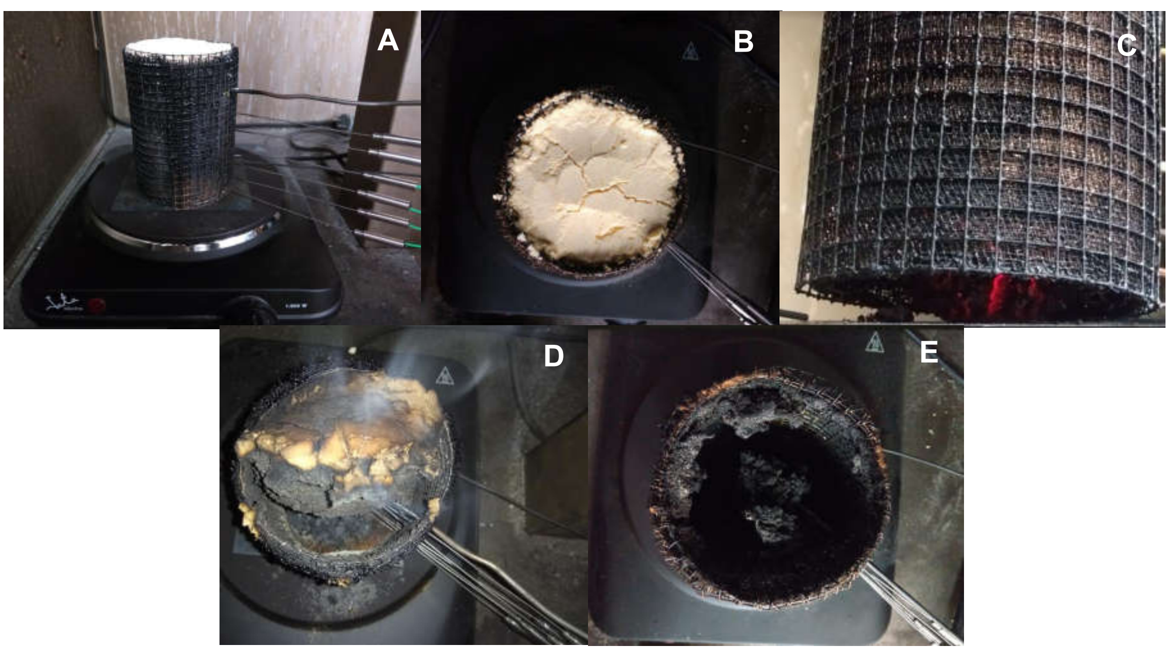

Figure 4-1 - Experiment sequence of dried corn flour without the ventilation system (F-Dried-NV).

Figure 4-2 - Residues of the three sizes of material used after the smoldering process: (F), (GS), (GL)

Figure 4-2 exemplifies the residues of each particle size used in the tests. The corn flour (F) residues were essentially ashes and in some cases, part of the top of the sample was not completely burnt. On the other hand, samples with two grain sizes (GS and GL) had few ashes, a large amount of unburned material in the upper half of the sample and a large mass of compacted char.

Part of the temperature evolution graphs will be indicated below in this section and the replicates and extras tests are annexed at Appendix B. With the reading of the data, loggers were possible to plot graphs indicating the temporal variation of the temperatures along the central axis of the sample. In all graphs are indicated the temperatures of the eight thermocouples, the black line shows the temperature of the hot plate and the rest of the curves show the temperatures of the different thermocouples. The curves located to the right of the graph correspond to the points farthest from the plate.

Figure 4-3 shows the temporal evolutions obtained in an experiment with dried corn flour and without a ventilation system. It is possible to observe how, after a certain time, in each of the positions the temperature increases to values well above the temperature of the hot plate. The process is slow, which could be noticed at the point located 6 cm from the plate (T5) needs approximately two hours and twenty minutes to increase significantly its temperature. However, once the process starts, its temperature continues to increase until it reaches a maximum of 786 ºC at approximately five hours. The last thermocouple that maintained the self-healing (T7) has reached its peak of 517 ºC nearly six hours and the smoldering process ended after seven hours of the experiment.

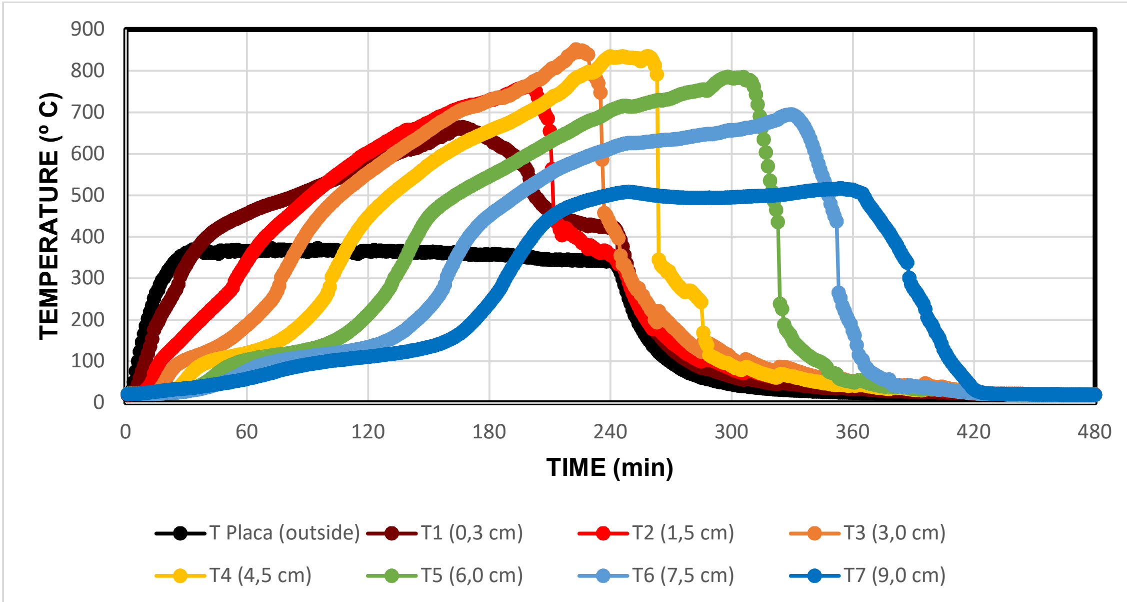

Figure 4-3 - Dried corn flour without a ventilation system (F-Dried-NV_1).

Figure 4-4 presents the experiment with dried corn flour with the ventilation system. In this graph, it is possible to observe some variations in the maximum temperatures. They exhibit relatively lower values than in the case without ventilation. The thermocouple T5 also, at approximately five hours. achieved its maximum temperature of 745 ºC. Comparing the smoldering process between this case and the previous one is possible to notice an increase of almost one hour in the process. The temperature decay of thermocouple T7 ends near eight hours of the experiment.

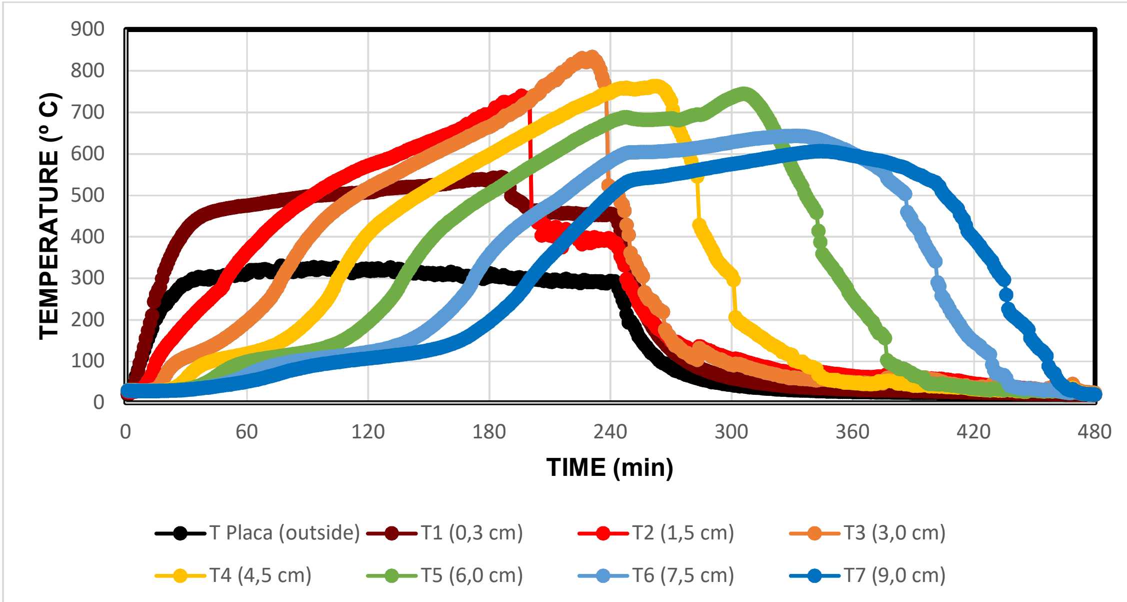

Figure 4-4 - Dried corn flour with ventilation system (F-Dried-V_1).

Figure 4-5 shows the result for the case of wet corn flour without ventilation (F-Wet-NV). It is possible to observe that the thermocouple T5 required more than two hours and forty minutes to reach the hot plate temperature, and the thermocouple T7 only reached the temperature after the four hours of heating.

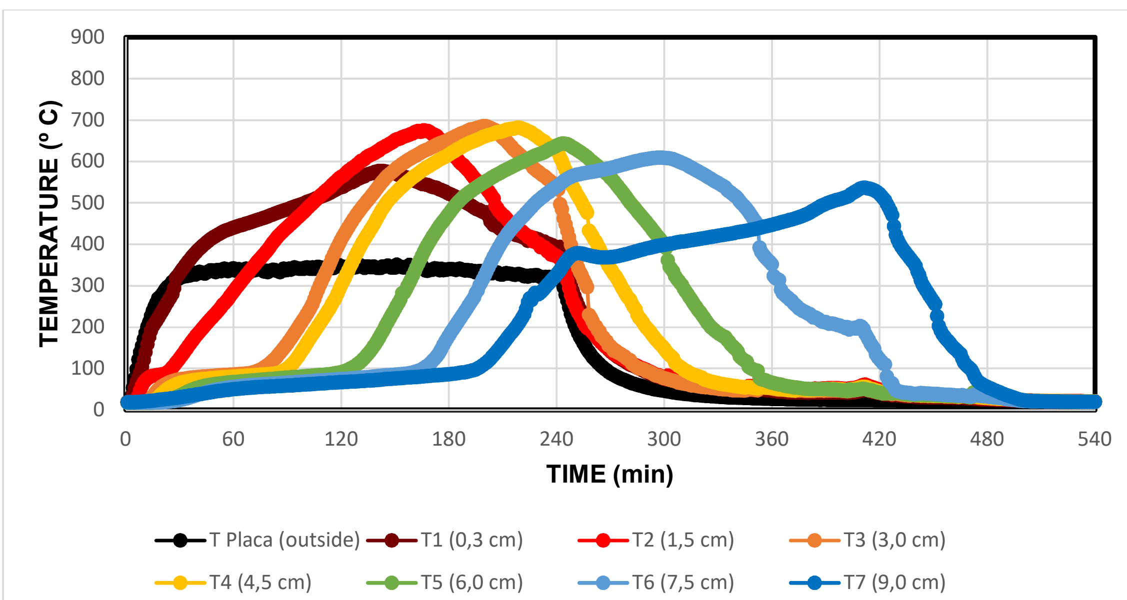

Figure 4-5 - Wet corn flour without a ventilation system (F-Wet-NV_1).

Figure 4-6 shows the temperature changes for the case of wet cornflour with the ventilation system (F-Wet-V). It is possible to observe that all the thermocouples took more time to reach the plate temperature when compared to the case with the same corn flour conditions but without air ventilation (F-Wet-NV). The thermocouple T5 located in the half of the sample reached the hot plate temperature after approximately three hours and thirty minutes. Another point is the temperature of the last thermocouple that only showed high temperatures after the heating plate was switched off. Its temperature peak of 597 º C was achieved nearly nine hours of experiment and after thirty minutes all the process was over.

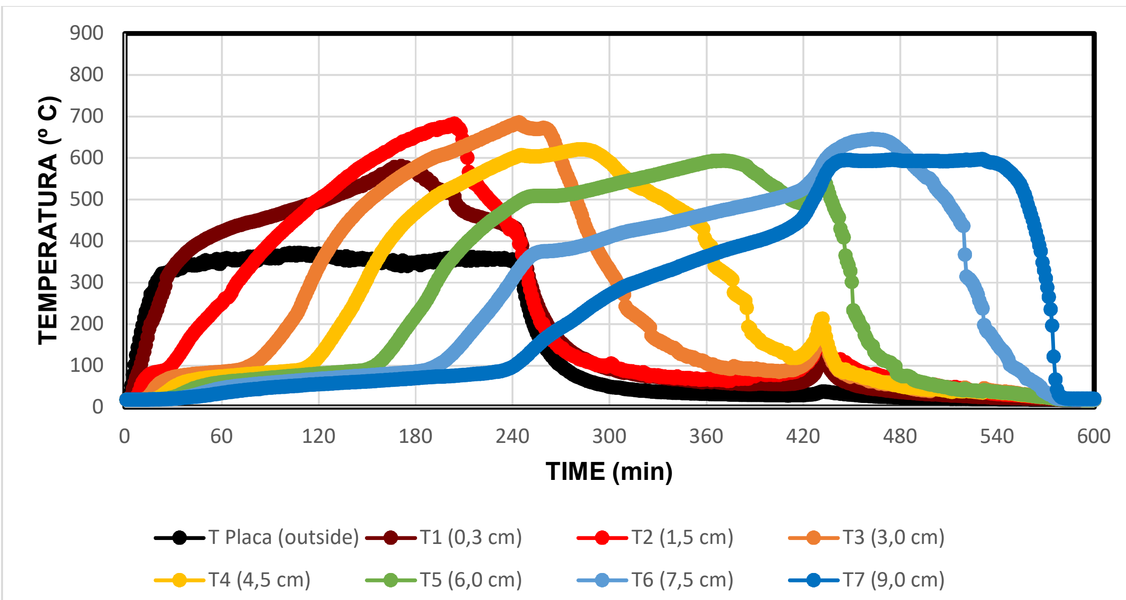

Figure 4-6 - Wet corn flour with ventilation system (F-Wet-V_2).

Figure 4-7 shows the temperature variation for the case of dried grain (776.4 μm) without ventilation (GS-Dried-NV). In this case, the temperatures of the thermocouples required a relatively long time to reach the temperature of the heating plate. The thermocouples T5 and T6 exceeded the hot plate temperature nearly three and four hours, respectively. Moreover, the time of permanence in the process of smoldering was superior to the other cases made with corn flour. A longer smoldering time was observed predominantly in T5 and T6 that completed the process after eleven hours. The last thermocouple that sustained the smoldering process (T6) achieved its temperature peak of 423º C after ten hours. The sample around the thermocouple T7 was unable to sustain the heat to maintain the smoldering.

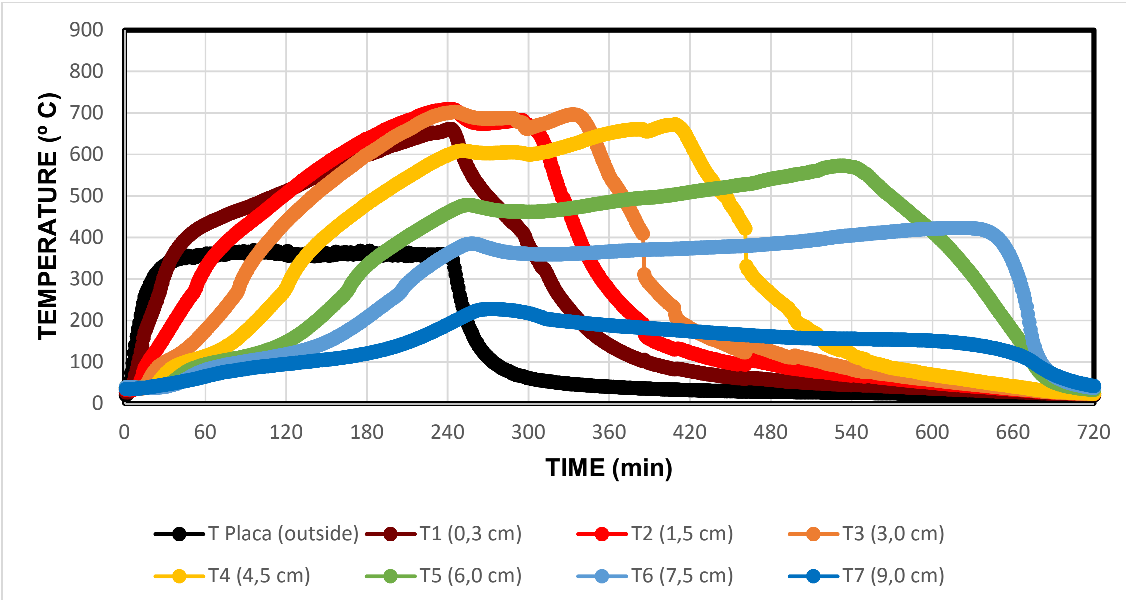

Figure 4-7 - Dried grain 776.4 μm without a ventilation system (GS-Dried-NV_3).

Figure 4-8 indicates the behavior of dried grain (776.4 μm) with the ventilation system (GS-Dried-V). In this test, the maximum temperatures were relatively lower than the temperatures of the dried grain test with no ventilation. In addition, the temperatures remained elevated for a longer time compared to the other tests performed. The mass of grain near the thermocouple T5 was the last one to exceed the hot plate temperature and to keep the burning process. Its maximum temperature of 394 ºC was only reached a few minutes after the shutdown of the hot plate. And the self-heating was sustained until almost eleven hours.

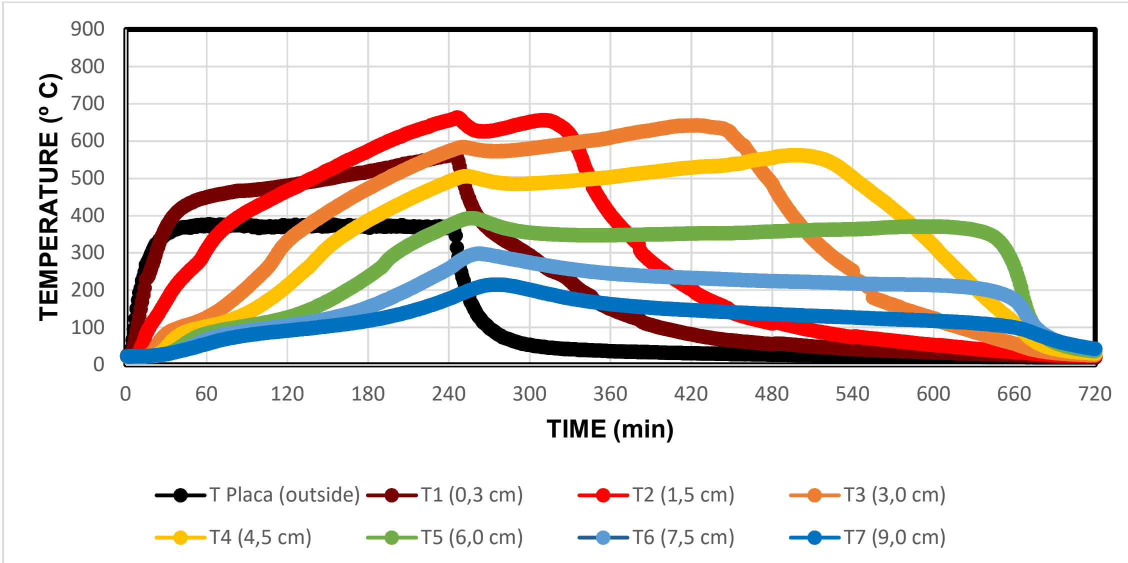

Figure 4-8 - Dried grain 776.4 µm with ventilation system (GS-Dried-V_3).

Figure 4-9 shows the case of wet grain (776.4 µm) without a ventilation system (GS-Wet-NV). Temperatures were lower than the other tests. The smoldering process was only kept in the sample closest to the plate until the height of the thermocouple T4 located 4.5 cm from the heating plate. After switching off the heating source, the temperature of the thermocouples T3 and T4 remained elevated for more than 3 hours. The maximum temperature of thermocouple T4 with the value of 382 ºC was attained after the turned off the hot plate.

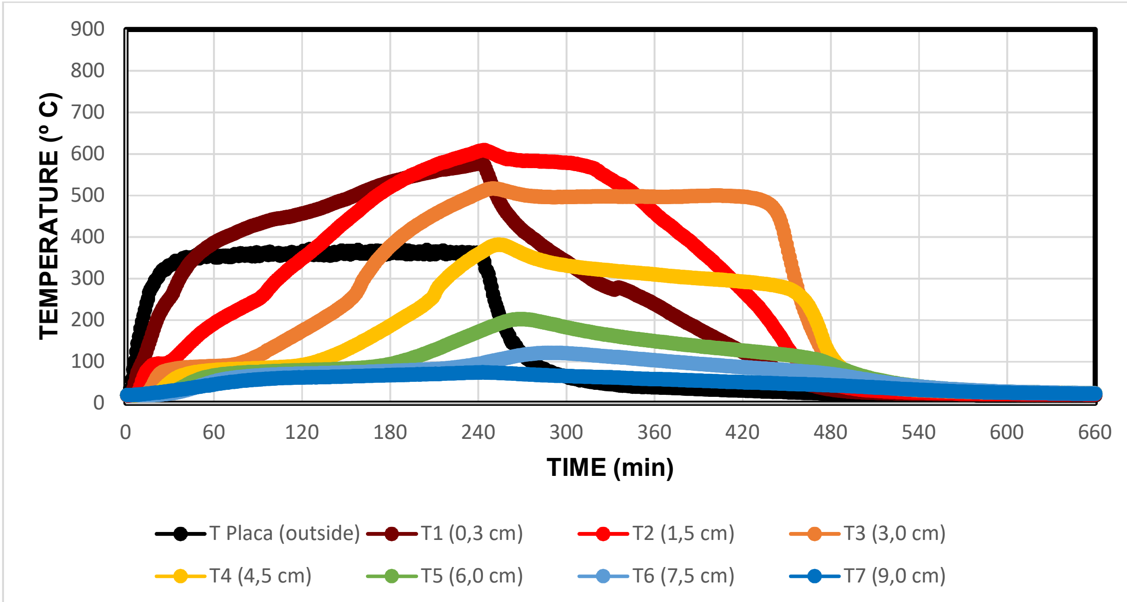

Figure 4-9 - Wet grain 776.4 µm without a ventilation system (GS-Wet-NV_2).

Figure 4-10 shows the case of wet grain (776.4 µm) with the system of ventilation (GS-Wet-V). The temperature evolutions were very similar to the case with the same grain and with no ventilation (GS-Wet-NV). However, the period in which the sample remained burning was slightly lower, which can be observed in the curves decay of the first four thermocouples. If we compare the values of the thermocouples T3 and T4 in seven hours of experiment with the same type of samples without and with ventilation, it is possible to verify that in the second case the temperatures of these thermocouples are smaller. In the first case, T3 and T4 presented the values of 497 ºC and 293 ºC, and in the second case 343 ºC and 286 ºC, respectively.

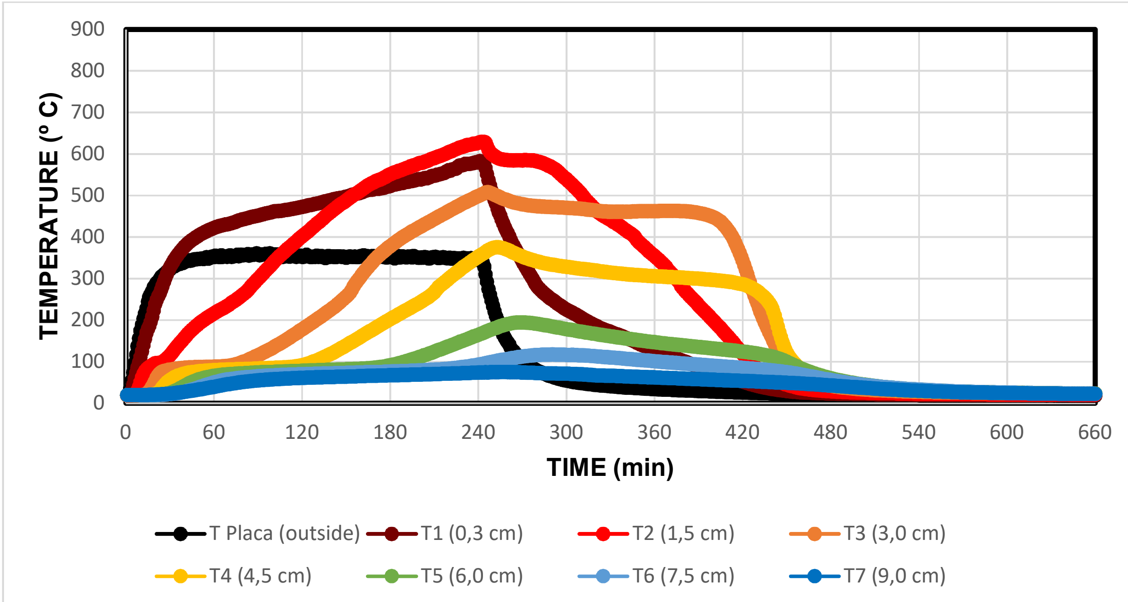

Figure 4-10 - Wet grain 776.4 µm with ventilation system (GS-Wet-V_2).

In addition to the tests performed with the two particle sizes. It was decided to carry out two tests with a particle size of 1411 µm dried and wet. Both tests were performed without a ventilation system. In the Figure 4-11 and Figure 4-12, it is possible to observe the evolution of the smoldering in the case with the larger grain size. Due to the greater particle diameter, both tests indicated a partial smoldering process. But the case with dried grain maintained elevated temperatures in T3 and T4 for at least four hours after shutting off the hot plate.

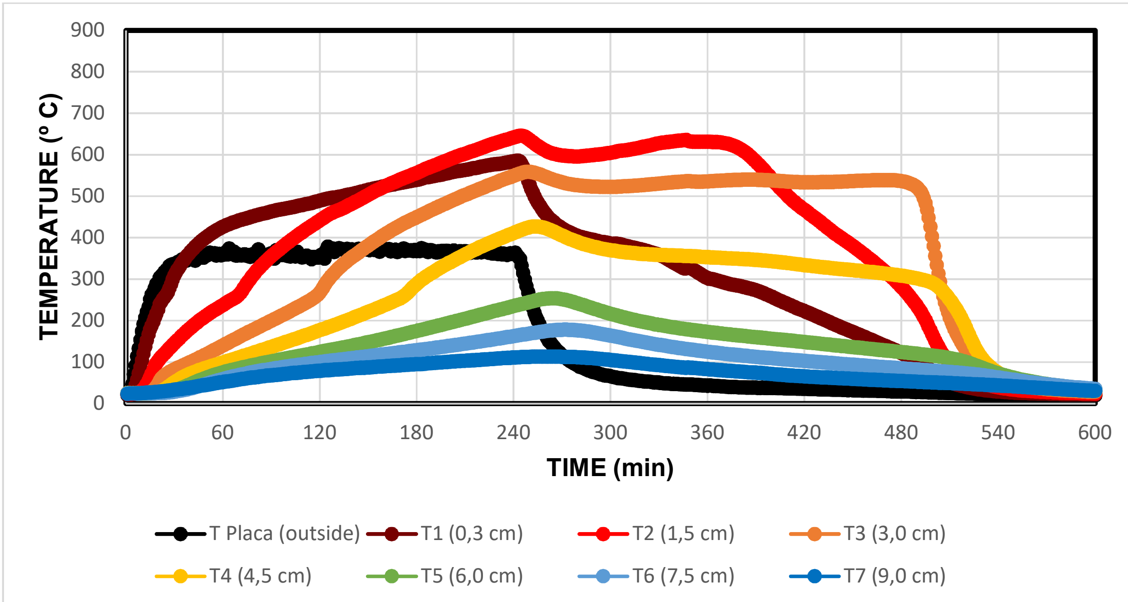

Figure 4-11 - Dried grain 1411 µm without a ventilation system (GL-Dried-NV_1).

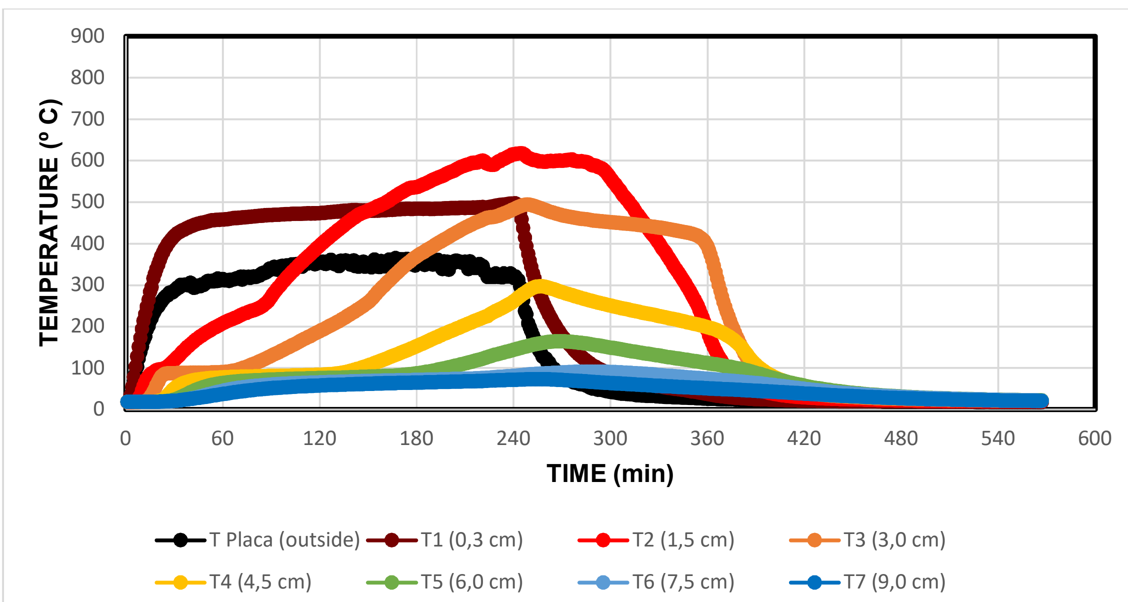

Figure 4-12 - Wet grain 1.0 mm without a ventilation system (GL-Wet-NV_1).

From the temperature evolutions graphs, the speed at which smoldering occurs can be determined. The time has been measured in each thermocouple, located at a certain distance from the hot plate that reaches a temperature of 350ºC. Then, a graph was plotted with the time values of each thermocouple for all experiments tests and the smoldering velocity was achieved by the slope of the regression line. Figure 4-13 shows the linear regressions and their equations for the tests with the three dried particle sizes and non-ventilated cases, which indicates that the line slope is larger for smaller particles. Appendix C shows the results of experiments performed.

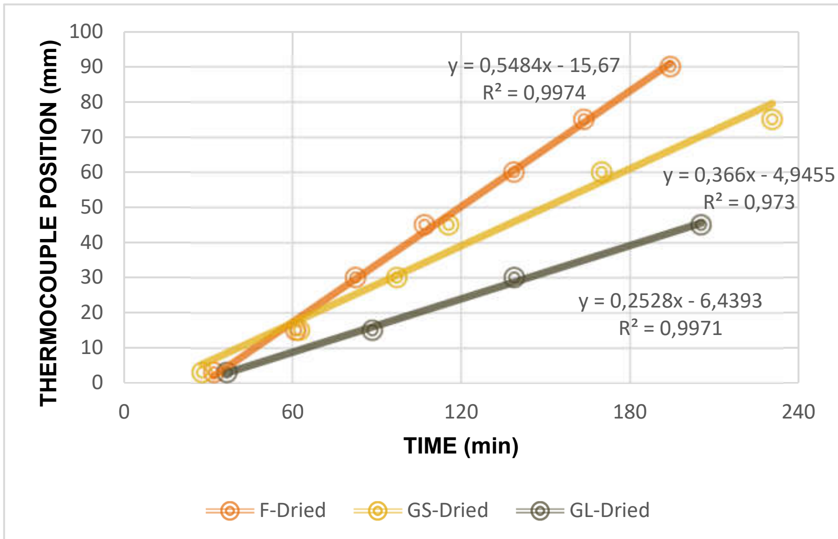

Figure 4-13 - Comparison of linear regressions of dried and non-ventilated cases.

As the smoldering process is slow if compared to flaming combustion, the temperatures along the sample took a long time to achieve the hot plate temperature and then sustain the self-heating process, which can be noticed in all graphs. For F-Dried, the temperature recorded by the T6 (located in the middle of the sample) took more than two hours to surpass the hot plate temperature. In the case with GS-Dried, the temperature of T5 took more than three hours to raise its temperature and for the wet sample it didn't achieve the hot plate temperature hindering the maintenance of the smoldering process. For GL-Dried, the temperatures were higher only near the heat source.

Analyzing the temperature development of each thermocouple, different smoldering rates were verified in each test. The smaller the particle size of the grain, the greater the speed of smoldering propagation. Regarding the ventilation system, the cases with ventilation presented a slower speed compared to the cases without ventilation.

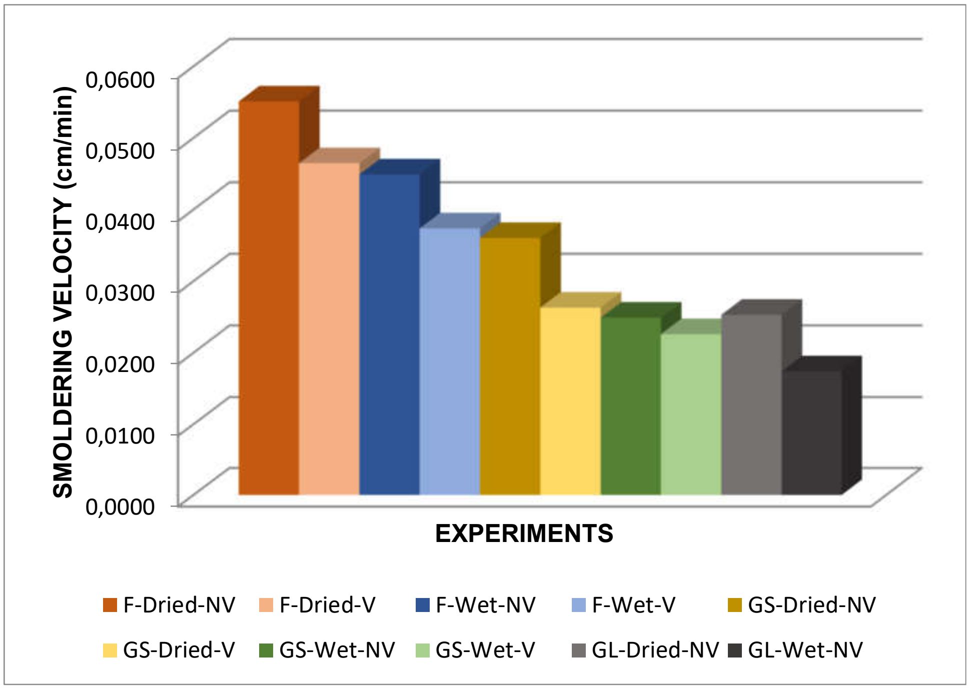

Figure 4-14 - Smoldering velocity of different particle sizes.

Figure 4-14 compares the speeds obtained for different corn material sizes, two levels of humidity, and with or without a ventilation system. It is clearly observed that the smaller the particle size, the greater the speed. Concerning the dried material, for both grain sizes without ventilation (GS-Dried-NV and GL-Dried-NV) the speed reached were 0.025 and 0.036 cm/min, respectively, while for the tests with F-Dried-NV the value was 0.055 cm/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 greater reduction was achieved between F-Dried and GL-Dried was 54.1%.

Analyzing only the influence of the ventilation system during the tests with and without ventilation, it is possible to observe that the tests with ventilation show slightly lower speeds. It was verified a reduction of 15.6% and 16.7% in the cases F-Dried and F-Wet. While in the cases GS-Dried and GS-Wet there were a reduction of 26.9% and 9.2%. Which indicates that ventilation had an influence more expressive with dried grain.The humidity also has an influence on the speed of propagation of the smoldering. It can also be verified that, as expected, the product with higher humidity produces slower smoldering rate. Comparing the samples F-Dried and F-Wet there was a reduction of 18,5% when increasing the level of humidity of the sample. While in the experiments with GS and GL there was a reduction of 30.8% and 31.2%. Moreover, one can conclude that the humidity has more influence in larger particles than smaller particles.

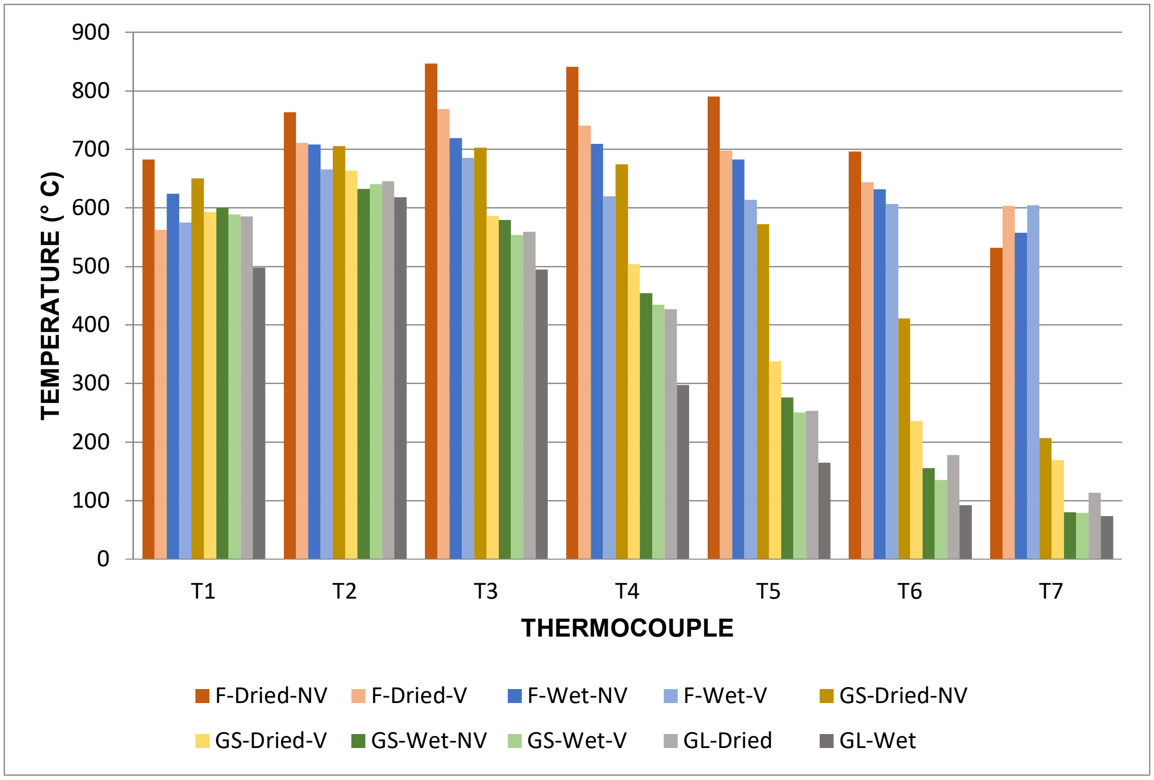

Figure 4-15 - The average maximum temperature of the thermocouples.

Figure 4-15 shows the average maximum temperature achieved in each thermocouple for the ten tests varying the influence factors. Usually, the highest temperatures achieved in samples with corn flour were the thermocouples T3 and T4. On the other hand, in the samples with the two grain sizes, the highest temperatures were obtained in thermocouples T2 and T3. An important point observed in this graph is that the values of thermocouple T1 for GS and GL samples are relatively higher than the temperatures of F-Wet samples. As observed in the individual charts of the temperature evolution, in this graph it is possible to note that in samples with corn flour, the maximum temperatures in all thermocouples remained high enough to sustain the smoldering. While in samples with GS and GL, temperatures begin to decrease from the T3 thermocouple, indicating a partial smoldering process and the incapacity to sustain the reaction.

An attempt to better understand the influence of ventilation, six extra tests were performed by varying the duration of ventilation and using F-Dried and GS-Dried. The experimental procedure of the hot plate remained the same for these extra experiments. Related to the one-hour ventilation experiment, the system was turned on only one hour after the hot plate operation and it was kept ventilating for one hour. Experiments with two and eight hours of ventilation were connected at the same time to the hot plate and the ventilation system was maintained for two and eight hours, respectivately.

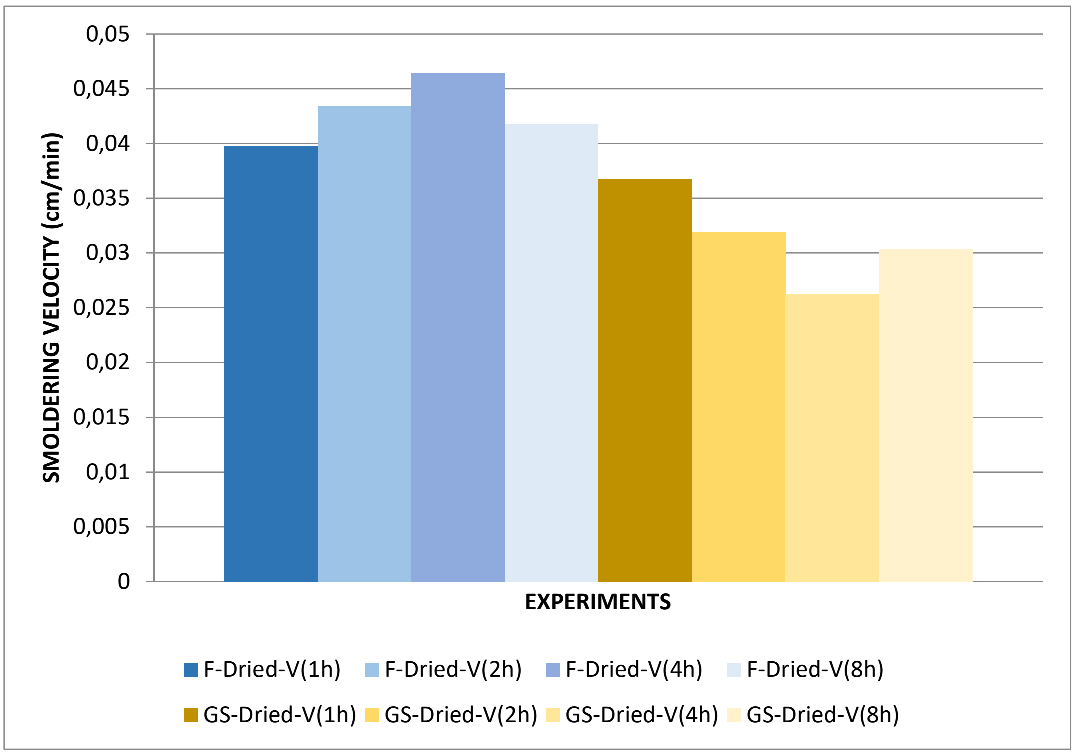

Figure 4-16 - Smoldering velocities for different ventilation times.

Figure 4-16 shows the smoldering velocity for the six new cases and the two cases of F-Dried and GS-Dried with four hours ventilation. The influence of the ventilation time on the smaller particles and on the larger particles presents a different behavior. The samples F-Dried presented an increase in the speed with the increase of the ventilation time, suffering a reduction in the experiment with eight hours of ventilation. However, the opposite effect was observed in samples GS. The velocity of smoldering decreased with the increasing in ventilation time, and with eight-hour ventilation experiment the speed had increased.

Analyzing the experiments with F-Dried samples, varying only one hour in the ventilation a 9% increase in the smoldering velocity was observed. And a total of 16.8% between the ventilation system from one hour to the four hours. However, if we compare the values between the systems with four and eight hours of ventilation, there was a reduction of 10% in speed. Regarding the experiments with GS-Dried samples, the addition of one hour of ventilation showed a reduction of 13.3%. And comparing the one-hour and four-hour ventilation systems, the speed dropped by 28.5%. However, the ventilation system for 8 hours implied an increase of 15.6% compared to 4 hours.

4.2 Minitab Evaluation - Smoldering evolution with a hot plate

For a better analysis, the experiments were developed according to the Design of Experiments (DOE) in Minitab. As mentioned before three influence factors were selected, two levels of them were varied, and the experiments were performed with two replicates, which led to the execution of sixteen experiments (Table 4-2). After inserting the values of the three factors and the smoldering speed in Minitab, it was possible to analyze the influence of each factor, as each contributed to the evolution of smoldering.

Not all experiments presented the same level of smoldering. Although all samples had a specific smoldering level, only the corn flour samples presented a level of total smoldering. As it could be verified visually at the end of the tests, some samples were completely burned with only ash residues and others were partially burnt with char residue and unburnt material.

Table 4-2 - Results of experiments.

EXP.	TYPE	PARTICLE DIAMETER (μm)	HUMIDITY	(%)	VENT. CONDITION	VENTILATION (m/s)	SMOLDERING VELOCITY	LEVEL OF SMOLDERING
1	GS	776.4	Wet	15	Y	0.1	0.0207	Partial
2	F	181.6	Wet	15	N	0	0.0465	Total
3	GS	776.4	Wet	15	N	0	0.0226	Partial
4	GS	776.4	Dried	0	Y	0.1	0.0292	Partial
5	F	181.6	Wet	15	Y	0.1	0.0387	Total
6	F	181.6	Wet	15	Y	0.1	0.0389	Total

7	F	181.6	Wet	15	N	0	0.0451	Total
8	GS	776.4	Dried	0	Y	0.1	0.0291	Partial
9	F	181.6	Dried	0	Y	0.1	0.0484	Total
10	F	181.6	Dried	0	N	0	0.0548	Total
11	GS	776.4	Dried	0	N	0	0.0355	Partial
12	GS	776.4	Wet	15	N	0	0.0231	Partial
13	GS	776.4	Dried	0	N	0	0.036	Partial
14	GS	776.4	Wet	15	Y	0.1	0.021	Partial
15	F	181.6	Dried	0	N	0	0.0554	Total
16	F	181.6	Dried	0	Y	0.1	0.0473	Total

The residual plots (Figure 4-17) shows the quality of the data set inserted for the smoldering velocities. In the normal probability plot, since the residuals lie approximately along a straight line, no severe abnormality in the data set was suspected. There are also no indications of outliers. So the data could be considered satisfactory for the analysis. The histogram presented a distribution similar to a gaussian indicating that the number of experiments was sufficient to guarantee that this phenomenon could be represented by a normal distribution.

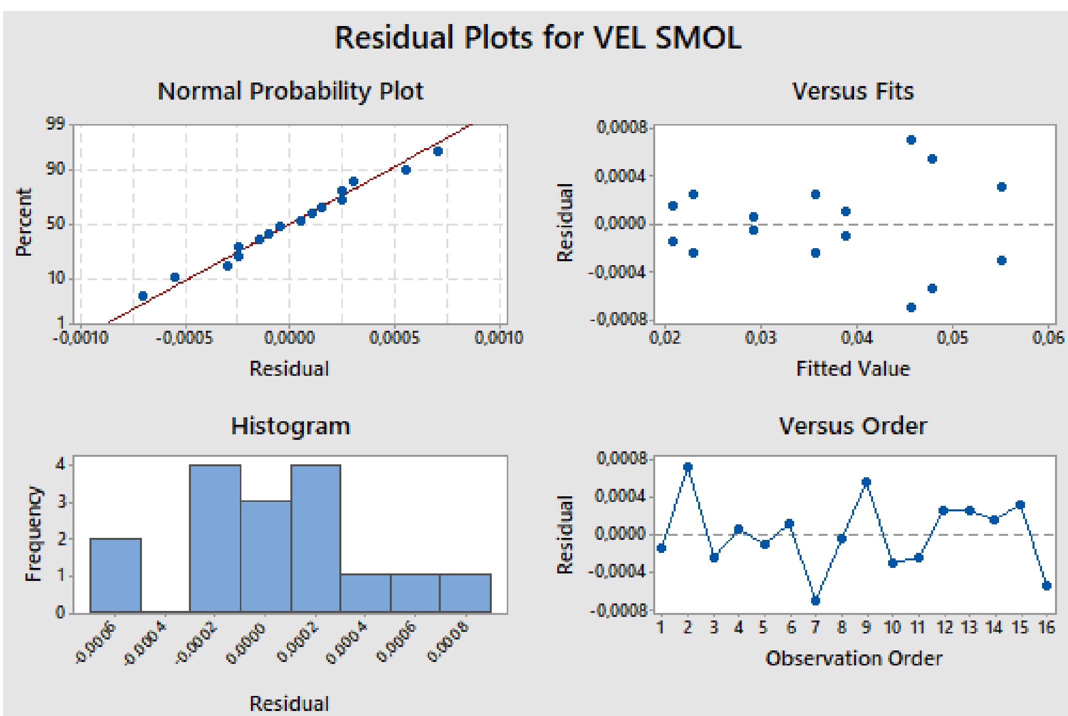

Figure 4-17 - Residual plots for smoldering velocity.

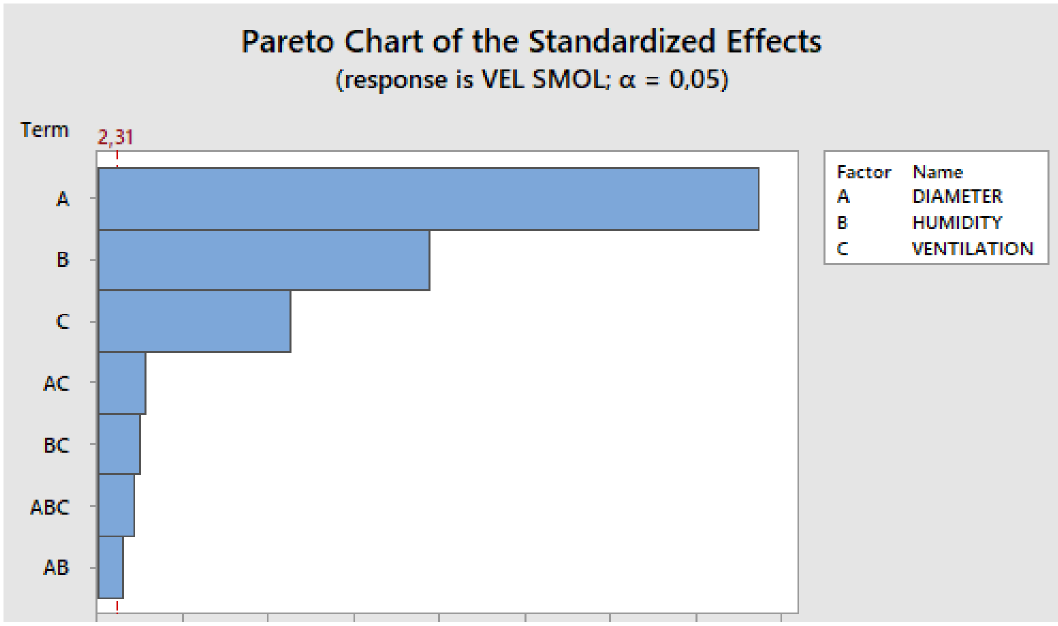

Figure 4-18 - Pareto chart of the standardized effects.

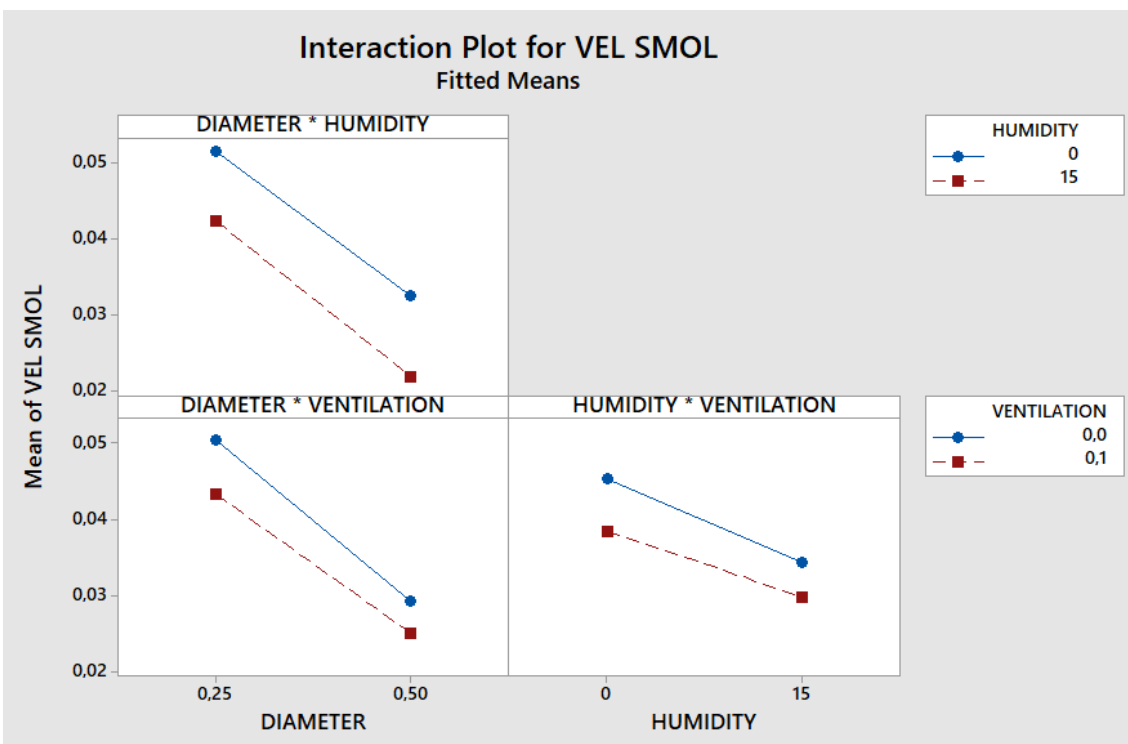

Figure 4-19 - Interaction plot for smoldering velocity.

One of the first graphs provided by Minitab was the Pareto Chart that evaluates the effect of the factors (Figure 4-18). It is possible to verify in the experiments that the factor with greater influence was the material diameter followed by humidity. It is important to emphasize that the influence of the material size is almost twice as great as the influence of humidity. Also according to Pareto Chart, the influence of two or more factors together was not so relevant. This fact could be verified in the set of interaction plots (Figure 4-19) for the smoldering velocity, no one of the factors presented interaction between them.

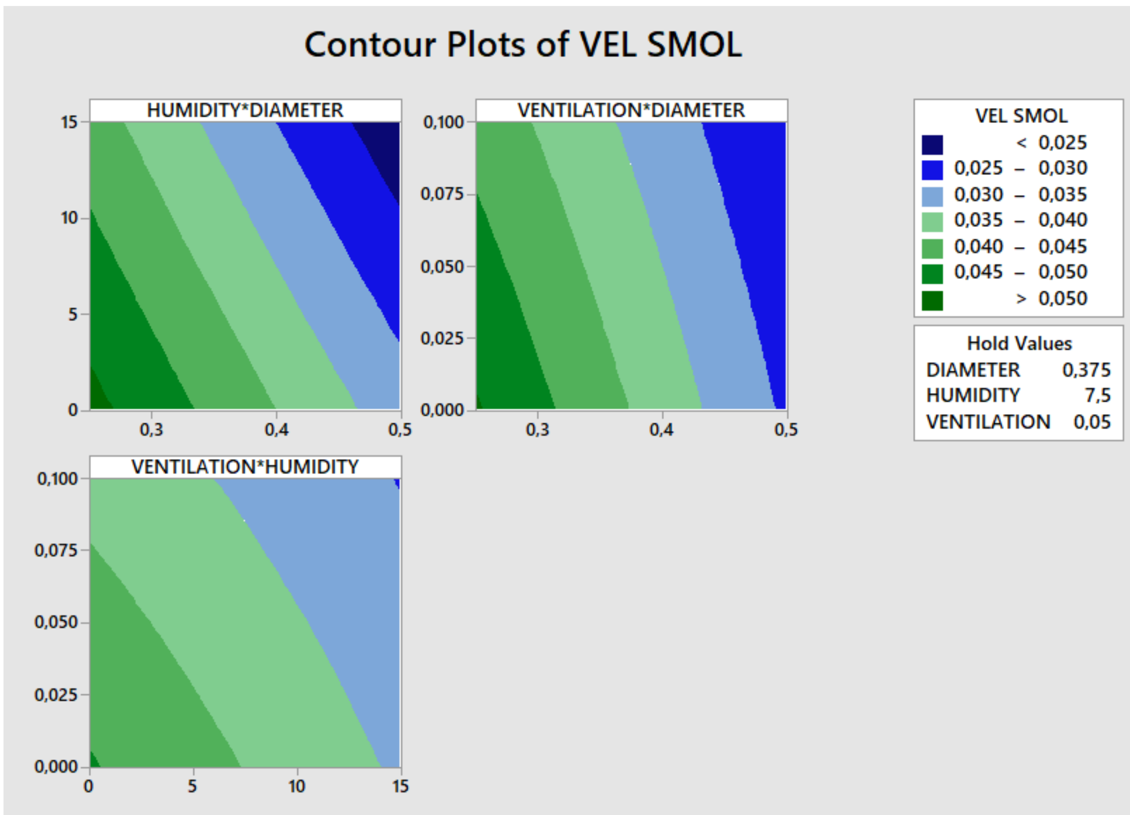

Figure 4-20 - Contour plots of smoldering velocity.

Another important graph was the contour plots (Figure 4-20), which evaluates the smoldering velocity with the variation of two factors. These graphs showed a variation of colors indicating the different values of velocity that made possible the evaluation of other cases between the limits of factor levels. The contour plot of humidity and diameter presents more bands than the others contours plot since these factors were the most influential in velocity during the experiments. In these contour plots were possible to analyze how variations between the levels influence the smoldering and predict the best cases where the smoldering speed presents smaller values. The best case was in the dark blue band with greater diameter particles, higher humidity and a higher level of ventilation condition that exhibited bands of areas with lower speeds. After the analysis of the bands with lower speed, it is also possible to find out other values of the factors that fit in the same bands. As it is possible to observe in the first contour plot, we can obtain the same speed inferior to 0.025 cm/min, if we have a level of humidity superior to 10% and a diameter of grain a little smaller than 0.500 mm. The next band with velocities between 0.025 and 0.030 cm/min was wider including humidities among 5% and 15% and diameter greater than 0.400 mm. In the second and third contour plot appeared fewer bands and the blue bands were more verticalized. This has shown that for a given range of diameter, ventilation may vary within the specified limits between 0 and 0.1 m/s. Other visualization of the variation of the velocity could be observed in the surface plots (Figure 4-21) that indicated the same result presented in the contour plots.

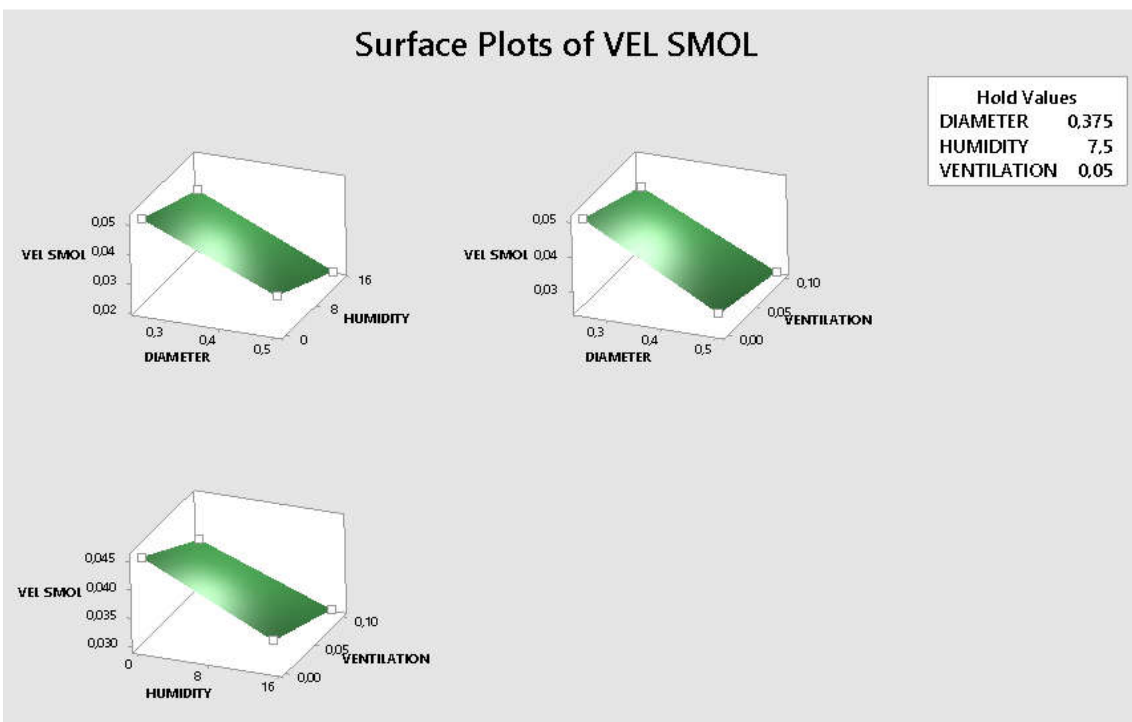

Figure 4-21 - Surface plots of smoldering velocity.

With the data inserted in Minitab it was also possible to obtain a mathematical equation representing the smoldering phenomenon with the three influencing factors (Dp –

Diameter of particles, H – Humidity, V – Ventilation) as shown in Figure 4-22. Therefore, it is possible from any set of values of these parameters to obtain the value of the smoldering rate for corn grains. This equation can be very useful for future work investigating the smoldering process with this type of material.

VEL SMOL = 0,074450 - 0,07740 **Dp** - 0,000380 **H** - 0,0790 **V** - 0,000960 **Dp*H** + 0,0260 **Dp*V** - 0,00273 **H*V** + 0,01160 **Dp*H*V**

Figure 4-22 - Mathematical equation of the smoldering process.

4.3 Experiments 2 – Ignition time and extinguishability

As previously mentioned, these experiments were performed concomitantly and in a way to complement the smoldering experiments. The aim is to analyse the fire behavior of a material in a testing scale, which can indicate how the material will behave in case of fire.

The three particle sizes with the two humidity conditions used in the smoldering experiment were evaluated with the procedure indicated in Section 3.2.2. Each sample was placed in a square rectangular box and then placed under the radiator device, which was removed and replaced after each ignition and extinction Through five minutes, the ignition time and the time to extinguish the flame were observed. After each test, the material was weighed and compared to the weight before starting the experiment. In addition, the thermocouple placed just above the sample surface measured the temperature variation during the test.

The sequence of photos shown in Figure 4-23 (items A and B) shows the start of ignition in the dried corn flour samples. And item C shows the near extinction of the flames in the dried grain sample.

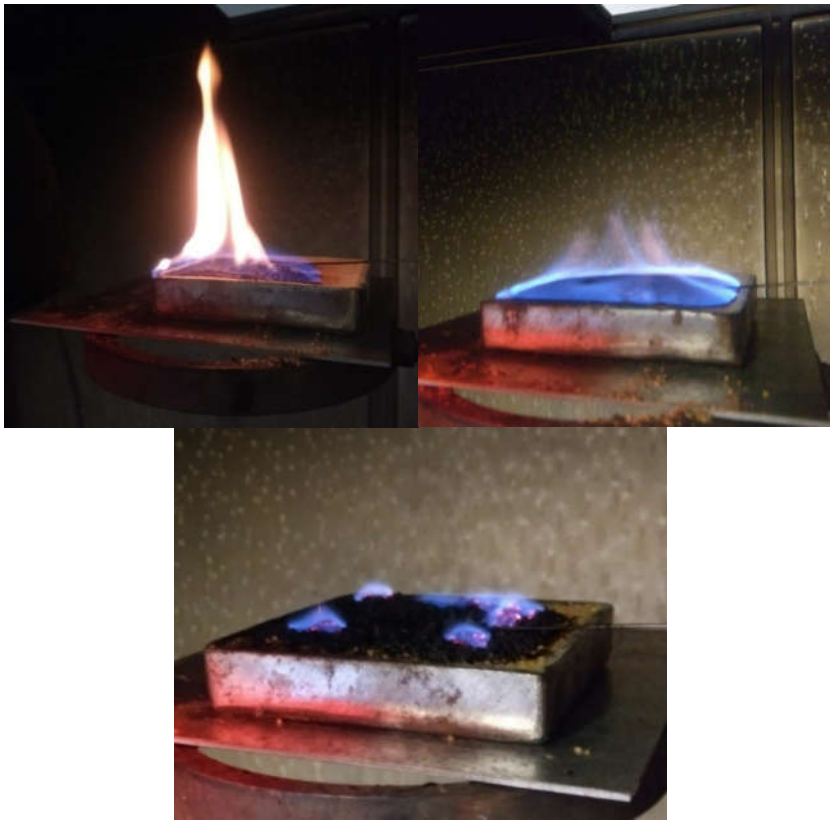

Figure 4-23 - Moments of ignition and flame extinction in samples.

Figure 4-24 - Wet grain 1.0 after the experiment.

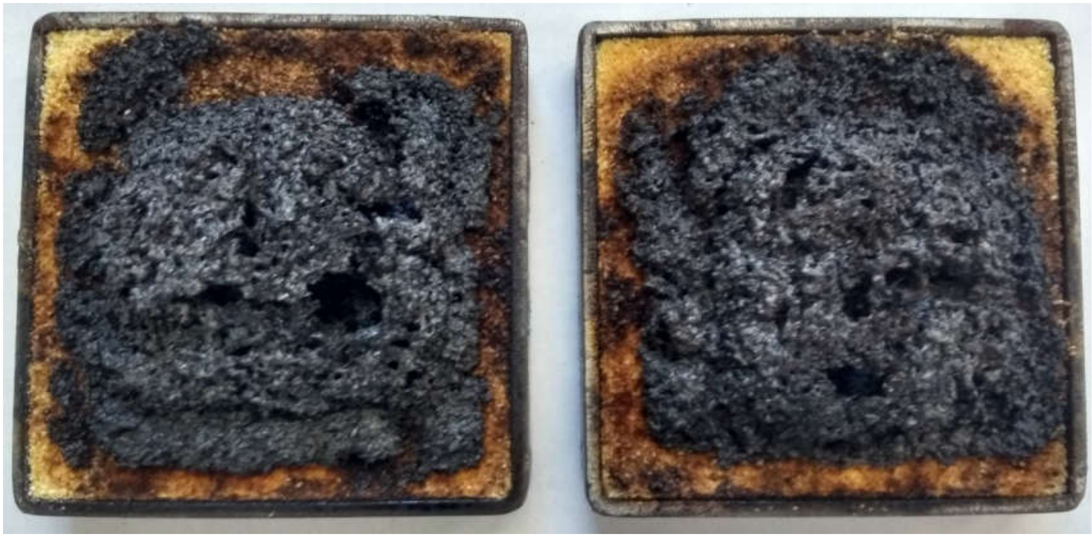

Figure 4-25 - Wet grain 0.5 after the experiment.

Figure 4-26 - Wet corn flour after the experiment.

Figures 4-24, Figure 4-25, and Figure 4-26 represent the samples with the three types of particle sizes for the wet condition. Only the top layer of the samples that have undergone the combustion process.

Table 4-3 shows the experiment results, it depicts the values of the first ignition time (t_1), the quantity of ignitions (N_{ig}), the average of combustion time (Δt_{IG}), and the percentage of mass loss for each sample (M_{LOSS}). Since the experiments were performed in duplicate, all the values found in the table are the average of them.

Table 4-3 - Ignition and extinguishability test results.

SAMPLE	t_1 (s)	N_{IG}	Δt_{IG} (s)	M_{LOSS} (%)
WET GRAIN 1.0	19.0	14.5	15.6	9.8
WET GRAIN 0.5	13.0	15.0	16.0	11.7
WET CORN FLOUR	10.5	22.0	9.5	11.8
DRIED GRAIN 1.0	16.0	5.0	61.0	5.5
DRIED GRAIN 0.5	10.5	5.5	64.4	7.9
DRIED CORN FLOUR	9.0	5.0	61.2	8.0

The ignition time and extinguishability test results indicate the ease of igniting the material sample and its potential to sustain the combustion and contribute to the fire propagation. Very long time between ignition and extinction of sample indicates a high potential to fire propagation.

The wet samples showed a slightly greater time of the first ignition, a superior number of ignitions and a greater loss of mass than the dried samples. However, the combustion time for the dried samples was much higher.

The results for Δt_{IG} show that the dried materials have ignitions of longer duration than those with wet materials, and therefore a worse behaviour might be expected in case of fire.

5 CONCLUSION

The principal risks in silos related to combustion are fires and explosions. With the bibliographical review, it was possible to analyze the evolution of the research related to the topic of dust explosions and smoldering combustion in particulate materials, which are the main risks in silos. The behavior of dust explosion and smoldering combustion are still very studied, since many factors influence the various phenomena related to the complexity of process, such as dispersion, heat transfer, mass transfer and reaction kinetics. Besides the risks of dust explosion and smoldering combustion during the storage, there is also the risk of fire and explosions with flammable gases.

Since the smoldering reaction is a slow combustion reaction, the material will continue burning for days or weeks until its detection and extinction. This justifies the latest studies related to the emission of gases and the detection of smoldering. Although documents from the Scopus database search date back to 1970, there is still a need for deeper studies for a full understanding of how the characteristics of each material influence the smoldering reaction rate. As well as there is a need in studies on the detection and extinction of smoldering in particulate material.

Among the experimental works of smoldering, there are several laboratory studies to evaluate the development of smoldering by varying the characteristics of the material and the external conditions of the environment as temperature and humidity. These conditions could influence the adsorption and absortion of moisture from air. The present work has shown two laboratory experiments, the first one evaluated the smoldering velocity with a hot plate and the second one evatuated the time of ignition and extinction with a radiator equipment.

In the experiments with the hot plate, the smoldering process succeeded vertically from the sample base until the point where the heat exchange was not sufficient to sustain the process. The total amount of heat released from the grains was not sufficient to maintain a self-sustaining combustion throughout the entire sample. However, in the case of corn flour the energy was sufficient to feed the entire smoldering 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 cases, with higher speeds and higher temperatures, were the tests with dried corn flours. One of the reasons is the relatively greater surface area than the grains. The smaller the grain size, the greater its 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. The other is that, with the pre-dried at 100ºC, they did not need to absorb energy from the oxidation reaction to heat and evaporate the water inside the particles. Bypassing the evaporation stage of the water inside the cornflour particles, all the heat generated by the hot plate is absorbed by the pyrolysis step.

Regarding air ventilation, there are two possible effects. One would be an increase in the smoldering reaction rate due to the increase in the oxygen flow available for the oxidation reaction. The other would be the heat dissipation generated preventing the occurrence of the heat insulation effect in the sample and slowing down the smoldering. Despite the theories that indicated that a certain ventilation could provide better oxygen access in the samples and thus a higher rate of combustion, in the ventilation system used in the tests the predominant effect was heat dissipation, which provided lower temperatures and burn rates.

The experiments with radiator device assisted in the analysis of the ignition and extinction characteristics. Which indicated a higher potencial of fire propagation to dried materials regardless of material size. 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 materials, when susceptible to the smoldering reaction, can present very high temperatures and can compromise the integrity of the structures that store these materials. Smoldering combustion is a slow reaction and it is difficult to detect. In some cases, it may take weeks for it to be discovered, which can make it difficult to quench. Its characterization and evaluation of the factors that influence it is extremely important to understand the development of this phenomenon. As well as to design storage structures more resistant and effective to prevent these reactions and to facilitate their extinction.

The experimental study of smoldering may provide important information for fire safety and the development of new research about the development of the smoldering process. As future works, experiments can be carried out by varying other influencing factors or adding new factors to those already selected in this work or experiments with other types of material. Other possible future work that can be evaluated is the transition from smoldering combustion to flaming combustion in grains.

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APPENDIX A – DOCUMENTS FROM LITERATURA REVIEW

Table A-1 - Documents from literature review.

YEAR	DOCUMENT TITLE	AUTHORS	SOURCE	INSTITUTION
2018	Self-heating properties of softwood samples investigated by using isothermal calorimetry	Rupar-Gadd K., Forss J.	Biomass and Bioenergy	Linnaeus University, Department of Built Environment and Energy Technology
2018	Differences between a deciduous and a conifer tree species in gaseous and particulate emissions from biomass burning	Pallozzi E., Lusini I., Cherubini L., Hajiaghayeva R.A., Ciccioli P., Calfapietra C.	Environmental Pollution	Institute of Agro-Environmental & Forest Biology (IBAF), National Research Council (CNR); Department of Landscape Design and Sustainable Ecosystems, Agrarian-technological Institute, RUDN University; Institute of Chemical Methodologies (IMC), National Research Council (CNR); Global Change Research Institute, Czech Academy of Sciences
2018	Self-heating characteristics of coal dust deposits by a hot gas flow in oxy-fuel atmospheres	Wu, D., Norman, F., Vanierschot, M., Verplaetsen, F., Berghmans, J.	Applied Thermal Engineering	School of Chemical Engineering, Sichuan Univeristy; Adinex NV; Mechanical Engineering Technology Cluster TC, Campus Groep T, KU Leuven; Department of Mechanical Engineering, KU Leuven
2018	Experimental technique and modeling for evaluating heat of rewetting effect on coals' propensity of spontaneous combustion based on adiabatic oxidation method	Wang, X., Luo, Y., Vieira, B.	International Journal of Coal Geology	Department of Mining Engineering, Liaoning Technical University; Department of Mining Engineering, West Virginia University

2018	Inert hot particle - Unconventional ignition source	Bechem, J., Barth, U.	Institution of Chemical Engineers Symposium Series	Department Methods of Safety Engineering and Incident Research; School of Mechanical Engineering and Safety Engineering, University of Wuppertal
2018	Spontaneous ignition behaviour of coal dust accumulations: A comparison of extrapolation methods from lab-scale to industrial-scale	Wu D., Schmidt M., Berghmans J.	Proceedings of the Combustion Institute	School of Chemical Engineering, Sichuan Univerisity; BAM Federal institute for Materials Research and Testing by Bundesanstalt für Materialforschung und -prüfung (BAM); Department of Mechanical Engineering, KU Leuven
2017	Development of a screening test based on isothermal calorimetry for determination of self-heating potential of biomass pellets	Larsson, I., Lönnermark, A., Blomqvist, P., Persson, H., Bohlén, H.	Fire and Materials	Fire Research, SP Technical Research Institute of Sweden
2017	Lessons learned from fires of the wood caused by the spontaneous combustion of coal dust in underground mines	Shi, B., Zhou, F., Cheng, J.	Journal of Thermal Analysis and Calorimetry	Key Laboratory of Gas and Fire Control for Coal Mines, Ministry of EducationChina University of Mining and Technology; School of Safety EngineeringChina University of Mining and Technology
2017	Insight into effects of pore diffusion on smoldering kinetics of coal using a 4-step chemical reaction model	Song, Z., Fan, H., Jiang, J., Li, C.	Journal of Loss Prevention in the Process Industries	College of Safety Science and Engineering, Nanjing Tech University
2017	Thermo-physical and kinetics parameters determination and gases emissions of self-ignition of sieved rice husk of different sizes on a hot plate	El-Sayed, S.A., Khass, T.M., Mostafa, M.E.	Asia-Pacific Journal of Chemical Engineering	Mechanical Power Engineering Department, Zagazig University
2017	Smoldering spot ignition of natural fuels by a hot metal particle	Urban, J.L., Zak, C.D., Song, J., Fernandez-Pello, C.	Proceedings of the Combustion Institute	Department of Mechanical Engineering, University of California; State Key Laboratory of Fire Science, University of Science and Technology of China

2017	Combustibility determination for cotton gin dust and almond huller dust	Hughs, S.E., Wakelyn, P.J.	Journal of Agricultural Safety and Health	USDA-ARS Southwestern Cotton Ginning Research Laboratory
2017	The relationship between a thermal analysis and a safety-relevant problem	Liske, B., Maiwald, K., Eskenazi, P.L., Barth, U.	Institution of Chemical Engineers Symposium Series	
2017	Self-ignition and smoldering characteristics of coal dust accumulations in O2/N2and O2/CO2atmospheres	Wu, D., Schmidt, M., Huang, X., Verplaetsen, F.	Proceedings of the Combustion Institute	Department of Mechanical Engineering, KU Leuven; Division 2.2 ‘‘Reactive Substances and Systems’’, BAM Federal Institute for Materials Research and Testing; Department of Mechanical Engineering, University of California; Adinex NV
2017	Numerical investigation on the self-ignition behaviour of coal dust accumulations: The roles of oxygen, diluent gas and dust volume	Wu, D., Norman, F., Schmidt, M., (...), Berghmans, J., Van den Bulck, E.	Fuel	Department of Mechanical Engineering, KU Leuven; Adinex NV; Division 2.2 “Reactive Substances and Systems”, BAM Federal Institute for Materials Research and Testing
2017	Determination of the interfacial heat transfer coefficient between forced air and sand at Reynold's numbers relevant to smouldering combustion	Zanoni, M.A.B., Torero, J.L., Gerhard, J.I.	International Journal of Heat and Mass Transfer	Department of Civil and Environmental Engineering, University of Western; School of Civil Engineering, University of Queensland
2016	The influence of coal particles on self-ignition of methane-air mixture at temperatures 950-1200 K	Leschevich, V.V., Penyazkov, O.G., Shimchenko, S.Y., Yaumenchykau, M.L.	Journal of Physics: Conference Series	Heat and Mass Transfer Institute of the National Academy of Sciences of Belarus
2016	Characteristics of thermoplastic powder in an aqueous foam carrier for inhibiting spontaneous coal combustion	Xi, Z., Li, A.	Process Safety and Environmental Protection	School of Environmental Science and Safety Engineering, Tianjin University of Technology

2016	Numerical study on the ignition behavior of coal dust layers in air and O2/CO2atmospheres	Wu, D., Vanierschot, M., Verplaetsen, F., Berghmans, J., Van den Bulck, E.	Applied Thermal Engineering	Department of Mechanical Engineering, KU Leuven; Adinex NV
2016	Dust Explosion Dynamics	Ogle, R.A.	Book	
2016	Effectiveness of Thermoplastic Powder to Retard Self-Heating and Spontaneous Combustion of Coal	Xi, Z., Sun, X.	Combustion Science and Technology	School of Environmental Science and Safety Engineering, Tianjin University of Technology
2016	Ignition Risks of Biomass Dust on Hot Surfaces	Chin, Y.S., Darvell, L.I., Lea-Langton, A.R., Jones, J.M., Williams, A.	Energy and Fuels	Energy Research Institute, School of Chemical and Process Engineering, University of Leeds
2016	Smoldering Combustion as a Treatment Technology for Feces: Sensitivity to Key Parameters	Yermán, L., Wall, H., Torero, J., Gerhard, J.I., Cheng, Y.-L.	Combustion Science and Technology	School of Civil Engineering, The University of Queensland; Department of Civil and Environmental Engineering, University of Western Ontario; Centre for Global Engineering, and Department of Chemical Engineering and Applied Chemistry, University of Toronto
2016	Advection and the self-heating of organic porous media	Aganetti, R., Lamorlette, A., Guilbert, E., Morvan, D., Thorpe, G.R.	International Journal of Heat and Mass Transfer	Aix-Marseille Université, CNRS, Centrale Marseille; Institute for Sustainability and Innovation, College of Engineering & Science, Victoria University
2016	Influence of inert materials on the self-ignition of flammable dusts	Binkau, B., Wanke, C., Krause, U.	Journal of Loss Prevention in the Process Industries	Otto-von-Guericke-University Magdeburg, Dept. of Plant Design and Process Safety
2016	Smoldering spot ignition of natural fuels by a hot metal particle	Urban, J.L., Zak, C.D., Song, J., Fernandez-Pello, C.	Spring Technical Meeting of the Western States Section of the Combustion Institute	

2016	Determination of spontaneous combustion of thermally dried sewage sludge	Medic Pejic, L., Fernandez Anez, N., García Torrent, J., Ramírez-Gómez, A.	Journal of Loss Prevention in the Process Industries	Department of Energy and Fuels (UPM Technical University of Madrid, Spain); Laboratorio Oficial Madariaga, LOM (UPM Technical University of Madrid, Spain); BIPREE Research Group (UPM Technical University of Madrid, Spain)
2016	Determination of the smouldering temperature on the basis of adiabatic tests	Engel, L., Kimpel, S., Franke, J.	Chemical Engineering Transactions	Consilab Gesellschaft für Anlagensicherheit mbh, Industriepark Höchst G830
2016	Detection of smouldering fires by carbon monoxide gas concentration measurement	Vingerhoets, J., Snoeys, J., Minten, S.	Chemical Engineering Transactions	?
2016	Self-Ignition caused by solar radiation	Krause, G.	Chemical Engineering Transactions	?
2016	Determination of the self-ignition behaviour of bulk materials from heat storage tests below atmospheric pressure	Seitz, S., Franke, J., Giebisch, G.	Chemical Engineering Transactions	
2015	Detection of incipient self-ignition process in solid fuels through gas emissions methodology	Fernandez Anez, N., Garcia Torrent, J., Medic Pejic, L., Grima Olmedo, C.	Journal of Loss Prevention in the Process Industries	Department of Energy and Fuels (UPM Technical University of Madrid, Spain); Laboratorio Oficial Madariaga, LOM (UPM Technical University of Madrid, Spain); Department of Geological and Mining Engineering (UPM Technical University of Madrid, Spain)
2015	Experimental investigation on the self-ignition behaviour of coal dust accumulations in oxy-fuel combustion system	Wu, D., Huang, X., Norman, F., (...), Berghmans, J., Van Den Bulck, E.	Fuel	Department of Mechanical Engineering, KU Leuven; Department of Mechanical Engineering, Imperial College London; Adinex NV

2015	Low temperature ignition of biomass	Jones, J.M., Saddawi, A., Dooley, B., (...), Weatherstone, S., Williams, A.	Fuel Processing Technology	Energy Research Institute, School of Process, Environmental and Materials Research, University of Leeds; University of Lincoln, School of Engineering, Brayford Pool; Alstom Power, Excelsior Road, Ashby-de-la-Zouch; E.ON, Technology Centre, Ratcliffe-on-Soar; Energy Technology Innovation Institute, School of Process Environmental and Materials Research, University of Leeds
2014	Experimental study and discrete element method modeling of temperature distributions in rapeseed stored in a model bin	Rusinek, R., Kobyłka, R.	Journal of Stored Products Research	Institute of Agrophysics, Polish Academy of Sciences
2014	Smouldering fire signatures in peat and their implications for palaeoenvironmental reconstructions	Zaccone, C., Rein, G., D'Orazio, V., (...), Belcher, C.M., Miano, T.M.	Geochimica et Cosmochimica Acta	Department of the Sciences of Agriculture, Food and Environment, University of Foggia; Department of Renewable Resources, University of Alberta; Department of Mechanical Engineering, Imperial College London; Department of Soil, Plant and Food Sciences, University of Bari “Aldo Moro”; School of Engineering, University of Edinburgh; Department of Geography, University of Exeter
2014	Self ignition of layers of metal powder mixtures	Dufaud, O., Bideau, D., Le Guyadec, F., (...), Perrin, L., Caleyron, A.	Powder Technology	Laboratoire Réactions et Génie des Procédés, Université de Lorraine; CEA, DEN, DTEC/SDTC/Laboratoire de

				Technologies Avancées des Procédés du cycle
2014	Thermal analysis of milling products and its implications in self-ignition	Bufalo, G., Costagliola, C., Mosca, M., Ambrosone, L.	Journal of Thermal Analysis and Calorimetry	Dipartimento di NapoliINAIL (ex ISPESL) Settore Ricerca, Certificazione e Verifica; Bioscience and Territory (DIBT)University of Molise; Department of Medicine and Health SciencesUniversity of Molise
2014	Experimental analysis of minimum ignition temperature of coal dust layers in oxy-fuel combustion atmospheres	Wu, D., Norman, F., Verplaetsen, F., Berghmans, J., Van Den Bulck, E.	Procedia Engineering	Katholieke Universiteit Leuven, Department of Mechanical Engineering; Adinex NV
2014	Assessing thermal stability -the challenge of powders	Umbrajkar, S., Rowe, S.	29th Center for Chemical Process Safety International Conference 2014, CCPS 2014 - Topical Conference at the 2014 AIChE Spring Meeting and 10th Global Congress on Process Safety	
2014	Calorimetric and heat transfer studies of the spontaneous combustion of two low carbon fuels	Leslie, G., Pollard, A., Matovic, D.	Journal of Loss Prevention in the Process Industries	Department of Mechanical and Materials Engineering, Queen's University at Kingston
2014	Effects of fuel properties on the natural downward smoldering of piled biomass powder: Experimental investigation	He, F., Yi, W., Li, Y., Zha, J., Luo, B.	Biomass and Bioenergy	Shandong University of Technology, Zibo
2013	Determination of measurement uncertainties in adiabatic hot-storage experiments for reactive dusts	Schmidt, M., Wanke, C., Krause, U.	Chemical Engineering and Technology	?
2013	Smoldering combustion of rice husk dusts on a hot surface	El-Sayed, S.A., Khass, T.M.	Combustion, Explosion and Shock Waves	Zagazig UniversityEl-SharkiaEgypt

2013	Ignition behavior of magnesium powder layers on a plate heated at constant temperature	Chunmiao, Y., Dezheng, H., Chang, L., Gang, L.	Journal of Hazardous Materials	Fire & Explosion Protection Laboratory, Northeastern University; Liaoning Nonferrous Metal Prospecting Institute; Department of Civil Engineering, Shenyang Jianzhu University
2013	Prediction of coal stockpile autoignition delay time using micro-calorimeter technique	Zhao, X., Wang, Q., Xiao, H., (...), Chen, P., Sun, J.	Fuel Processing Technology	State Key Laboratory of Fire Science, University of Science and Technology of China; State Key Laboratory of Coal Resources and Safe Mining, China University of Mining & Technology
2013	Self-heating of powder materials during high-temperature drying	Rafique Qureshi, M.M.	ASM International - 27th Heat Treating Society Conference 2013	
2013	Effects of fuel properties on natural downward smoldering of piled biomass powder: Experimental investigation	He, F., Gao, Z., Luo, B., Li, Z., Yi, W.	Forest and Plant Bioproducts Division 2013 - Core Programming Area at the 2013 AIChE Annual Meeting: Global Challenges for Engineering a Sustainable Future	
2012	Analysis of an explosion in a wool-processing plant	Salatino, P., Di Benedetto, A., Chirone, R., Salzano, E., Sanchirico, R.	Industrial and Engineering Chemistry Research	Dipartimento di Ingegneria Chimica, Università degli Studi di Napoli Federico II and Istituto di Ricerche sulla Combustione− CNR

2012	Analysis of the effects of storage conditions on the preservation of soybean quality and the prevention of the self-heating process and the occurrence of fires.	Milanko, V.J., Gavanski, D.G., Laban, M.D.	Hemijska Industrija	Visoka tehnička škola strukovnih studija u Novom Sadu, Novi Sad, Srbija; Univerzitet u Novom Sadu, Fakultet tehničkih nauka, Novi Sad, Srbija
2011	Effect of weathering of coal and organic dusts on their spontaneous ignition	Joshi, K.A., Rangwala, A.S., Raghavan, V.	28th Annual International Pittsburgh Coal Conference 2011, PCC 2011	
2011	Ignition of dust layers by mechanical sparks	Van Wingerden, K., Hesby, I., Eckhoff, R.	AIChE Annual Meeting, Conference Proceedings	
2011	Preventing self-heating and ignition in drying operations	Zeeuwen, P., Ebadat, V.	Chemical Engineering	
2011	Self ignition of layers of powder mixtures: Effect of solid inertants	Bideau, D., Dufaud, O., Le Guyadec, F., (...), Corriou, J.-P., Caleyron, A.	Powder Technology	CEA, DEN, DTEC/SDTC/Laboratoire d'études des matériaux en environnement, Laboratoire Réactions et Génie des Procédés, Nancy-Université
2011	Self-sustaining smoldering combustion for NAPL remediation: Laboratory evaluation of process sensitivity to key parameters	Pironi, P., Switzer, C., Gerhard, J.I., Rein, G., Torero, J.L.	Environmental Science and Technology	Department of Civil and Environmental Engineering, University of Western Ontario; Department of Civil Engineering, University of Strathclyde; Institute for Infrastructure and Environment, School of Engineering, University of Edinburgh
2010	Influence of the degree of carbonization and granulation of fuel on combustion process	Cornea, N.	Revista de Chimie	

2010	Experimental determination of self-heating and self-ignition risks associated with the dusts of agricultural materials commonly stored in silos	Ramírez, A., García-Torrent, J., Tascón, A.	Journal of Hazardous Materials	BIPREE Research Group, Universidad Politécnica de Madrid; Laboratorio Oficial Madariaga, Universidad Politécnica de Madrid; Departamento de Ingeniería y Ciencias Agrarias, ETSI Agraria, Universidad de León
2009	Comparison of natural upward and downward smoldering using the volume reaction method	He, F., Behrendt, F.	Energy and Fuels	School of Agricultural Engineering, Shandong University of Technology; Department of Energy Engineering, Berlin Institute of Technology
2009	The fire-retarding effect of inorganic phosphorus compounds on the combustion of cellulosic materials	Liodakis, S., Fetsis, I.K., Agiovlasitis, I.P.	Journal of Thermal Analysis and Calorimetry	Laboratory of Inorganic and Analytical Chemistry, Department of Chemical EngineeringNational Technical University of Athens (NTUA)
2009	Heat accumulations and fire accidents of waste piles	Shimizu, Y., Wakakura, M., Arai, M.	Journal of Loss Prevention in the Process Industries	School of Engineering, The University of Tokyo; Kanagawa Industrial Technology Center; Environmental Science Center, The University of Tokyo
2009	Measurement of the heat of smoldering combustion in straws and stalks by means of simultaneous thermal analysis	He, F., Yi, W., Zha, J.	Biomass and Bioenergy	School of Light Industry and Agricultural Engineering, Shandong University of Technology
2008	Correlation between self-ignition of a dust layer on a hot surface and in baskets in an oven	Janes, A., Carson, D., Accorsi, A., (...), Tribouilloy, B., Morainvillers, D.	Journal of Hazardous Materials	INERIS - Parc ALATA

2008	Optimized spray dryer temperature by applying an adapted smouldering test	Steensma, M.	Bulk Solids Handling	
2007	Spontaneous ignition in storage and production lines: Investigation on wood pellets and protein powders	Pauner, M.A., Bygbjerg, H.	Fire and Materials	
2006	Experimental results contributed to early detection of smouldering milk powder as an integrated part of maintaining spray drying plant safety	Chong, L.V., Chen, X.D., Mackereth, A.R.	Drying Technology	
2006	A numerical model to simulate smouldering fires in bulk materials and dust deposits	Krause, U., Schmidt, M., Lohrer, C.	Journal of Loss Prevention in the Process Industries	Federal Institute for Materials Research and Testing (BAM), Division II.2 'Reactive Substances and Systems'
2005	Applications of the VSP2 in process safety	Greenfield, W.	3rd International Symposium on Runaway Reactions, Pressure Relief Design, and Effluent Handling	
2005	Self-ignition of combustible bulk materials under various ambient conditions	Lohrer, C., Krause, U., Steinbach, J.	Institution of Chemical Engineers Symposium Series	
2005	Self-ignition of combustible bulk materials under various ambient conditions	Lohrer, C., Krause, U., Steinbach, J.	Process Safety and Environmental Protection	Federal Institute for Materials Research and Testing (BAM), Working Group "Flammable Bulk Materials and Dusts, Solid Fuels"; Technical University of Berlin, Faculty of Process Sciences, Chair of Plant and Safety Engineering
2003	Ignitions of explosive dust clouds by smouldering and flaming agglomerates	Gummer, J., Lunn, G.A.	Journal of Loss Prevention in the Process Industries	Health and Safety Laboratory, Harpur Hill, Buxton, Derbyshire
2003	Self-ignition of dust at reduced volume fractions of ambient oxygen	Schmidt, M., Lohrer, C., Krause, U.	Journal of Loss Prevention in the Process Industries	Federal Institute for Materials Research and Testing (BAM), Laboratory II.22 "Dust Fires and Dust Explosions"

2002	Self-ignition of dusts at reduced volume fractions of oxygen	Krause, U., Schmidt, M.	Journal De Physique	
2001	Self-heating behaviour of low moisture content particles -modelling the basket-heating of solid particles and some aspects of the cross over behaviour using milk powder as an example	Chen, X.D.	ANZIAM Journal	
2001	The influence of initial conditions on the propagation of smouldering fires in dust accumulations	Krause, U., Schmidt, M.	Journal of Loss Prevention in the Process Industries	Federal Institute for Materials Research and Testing (BAM), Laboratory II.22 “Dust Fires and Dust Explosions”
2000	Smoldering combustion of dust layer on hot surface	El-Sayed, S.A., Abdel-Latif, A.M.	Journal of Loss Prevention in the Process Industries	Department of Mechanical Power Engineering, Zagazig University
2000	Propagation of smouldering in dust deposits caused by glowing nests or embedded hot bodies	Krause, U., Schmidt, M.	Journal of Loss Prevention in the Process Industries	Federal Institute for Materials Research and Testing (BAM); Otto-von-Guericke University
1999	Safe handling of renewable fuels and fuel mixtures	Wilén, C., Moilanen, A., Rautalin, A., (...), Timmers, P., Brehm, K.	VTT Publications	
1999	A mathematical model of the self-heating of spray-dried food powders containing fat, protein, sugar and moisture	Chong, L.V., Chen, X.D.	Chemical Engineering Science	Food Science and Process Engineering Group, Department of Chemical and Materials Engineering, The University of Auckland
1999	Laboratory study of spontaneous combustion of coal: the influence of inorganic matter and reactor size	Sujanti, W., Zhang, D.-K.	Fuel	CRC for New Technologies for Power Generation from Low-rank Coal, Department of Chemical Engineering, The University of Adelaide

1999	Study of spontaneous combustion for automobile shredder residue (ASR)	Horii, M., Iida, S.	JSAE review	Social & Environmental Research Division, Japan Automobile Research Institute; Environmental Affairs Division, Toyota Motor Corporation
1998	Extinguishing smouldering fires in silos: BRANDFORSK project 745-961	Tuomisaari, M., Baroudi, D., Latva, R.	VTT Publications	
1998	Effect of inerts on layer ignition temperatures of coal dust	Reddy, P.D., Amyotte, P.R., Pegg, M.J.	Combustion and Flame	Department of Chemical Engineering, DalTech (formerly The Technical University of Nova Scotia), Dalhousie University
1997	Initiation of smouldering fires in combustible bulk materials by glowing nests and embedded hot bodies	Krause, U., Schmidt, M.	Journal of Loss Prevention in the Process Industries	Federal Institute for Materials Research and Testing (BAM); Otto-von-Guericke University
1995	Explosion part three	Pilkington, Gary, Cartwright, Paul	Chemical Engineer	
1993	Risks associated with drying and storage of powders	Maddison, N.	Powder Handling and Processing	
1992	Research on dust explosions at the University of Michigan	Kauffman, C.W., Sichel, M., Wolanski, P.	Powder Technology	The University of Michigan
1992	Studies relating to ignition of the fire at King's Cross Underground Station	Wharton, R.K.	Fire Safety Journal	Explosion and Flame Laboratory, Health and Safety Executive
1988	Influence of sample shape and size on self-ignition of a fat-filled milk powder	O'Mahony, J.G., Synnott, E.C.	Journal of Food Engineering	Dairy and Food Engineering Department, University College Cork
1988	Hot-surface ignition temperatures of dust layers	Miron, Y., Lazzara, C.P.	Fire and Materials	
1987	Simulation of Spontaneous Heating for Evaluating Ignition Temperature and Induction Time	Liang, H., Tanaka, T.	KAGAKU KOGAKU RONBUNSHU	Department of Chemical Process Engineering, Hokkaido University

1987	The Spontaneous Ignition of Dust Deposits — Ignition Temperature and Induction Time—	Liang, H., Tanaka, T.	KONA Powder and Particle Journal	Department of Chemical Process Engineering, Hokkaido University
1985	Thermal ignition of self-heating porous slab: Comparison with experiment	Kordylewski, W., Krajewski, Z.	Combustion and Flame	Technical University of Wroclaw
1985	Smoldering combustion in horizontal dust layers	Leisch, S.O., Kauffman, C.W., Sichel, M.	Symposium (International) on Combustion	Department of Aerospace Engineering The University of Michigan
1981	Combustion inhibition of cellulose by powders: Preliminary data and hypotheses	McCarter, R.J.	Fire and Materials	
1981	Spontaneous combustion in beds of small fuel particles	Lawn, C.J., Street, P.J., Baum, M.M.	Symposium (International) on Combustion	Central Electricity Generating Board, Marchwood Engineering Laboratories
1977	Self-ignition and mechanisms of interaction of coal with oxygen at low temperatures. 2. Changes in weight and thermal effects on gradual heating of coal in air in the range 20-300 °C	Marinov, V.N.	Fuel	Energoproekt, Department of Scientific Research
1970	Checks on the Possibilities for the Spontaneous Combustion of Wood Checks on the Possibilities for the Spontaneous Combustion of Wood-Pillar as Heated Periodically by the Neighboring Chimney at Relatively Low Temperature for the Long Period.	Handa, T., Suzuki, H., Origasa, R., Takahashi, A.	Bulletin of Japan Association for Fire Science and Engineering	Science University of Tokyo

APPENDIX B – TEMPERATURE EVOLUTION GRAPHS OF EXPERIMENTS

Experiments with the samples of dried corn flour without a ventilation system.

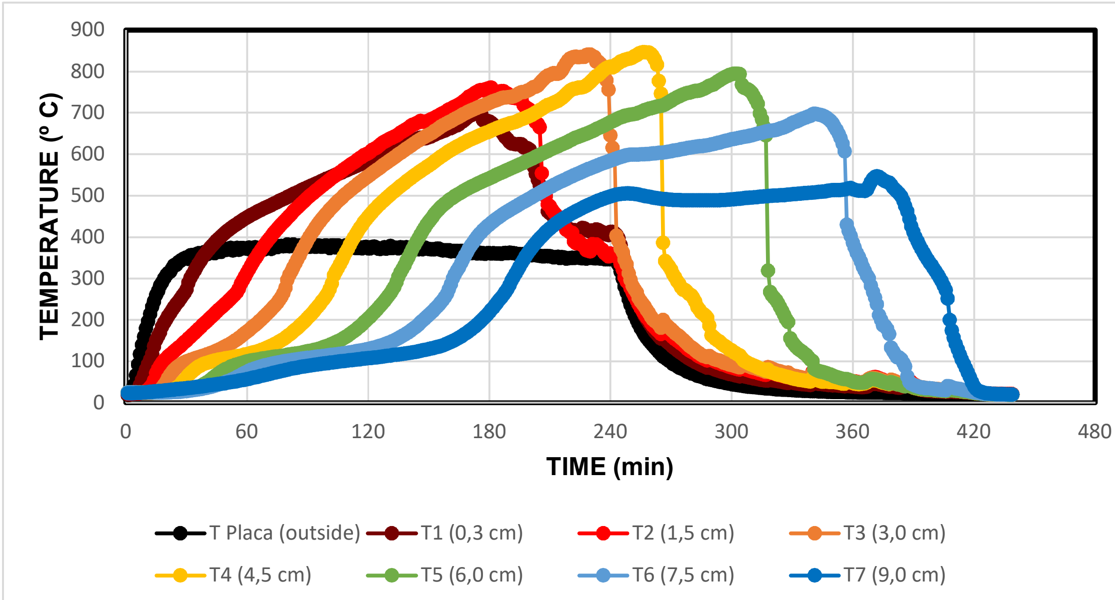

Figure B-1 - Dried corn flour without a ventilation system (F-Dried-NV_2).

Experiments with the samples of dried corn flour with the ventilation system.

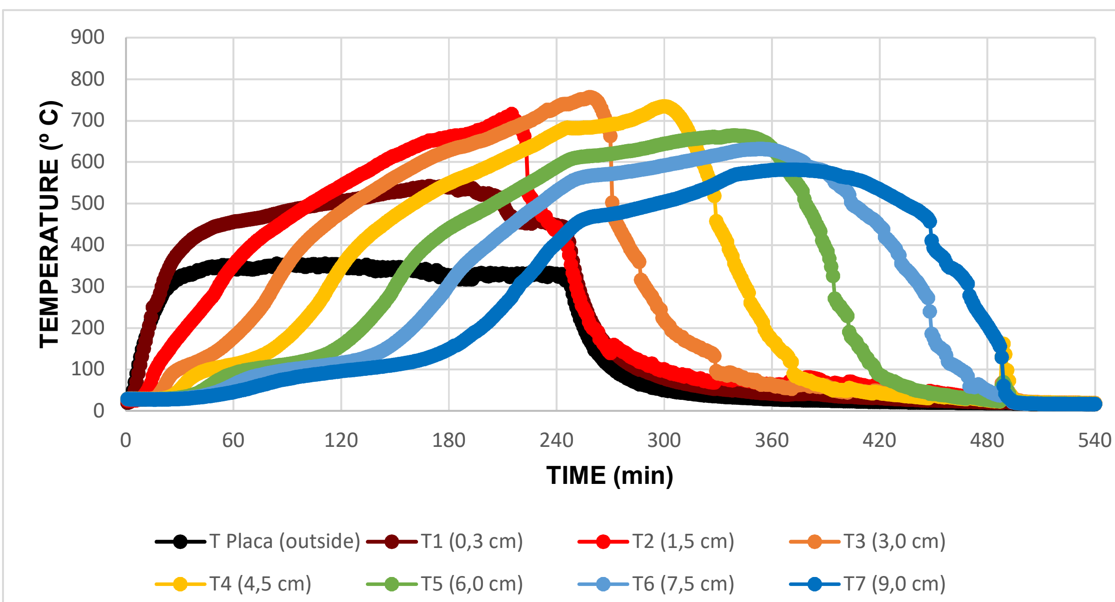

Figure B-2 - Dried corn flour with a ventilation system (F-Dried-V_2).

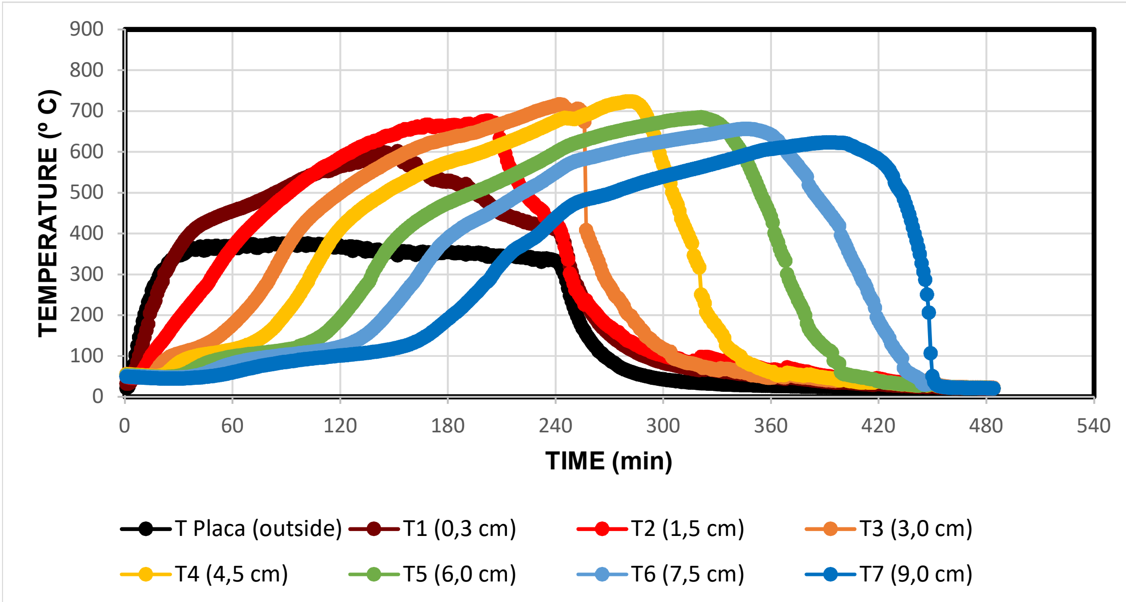

Figure B-3 - Dried corn flour with a ventilation system (F-Dried-V_3).

Experiments with the samples of wet corn flour without a ventilation system.

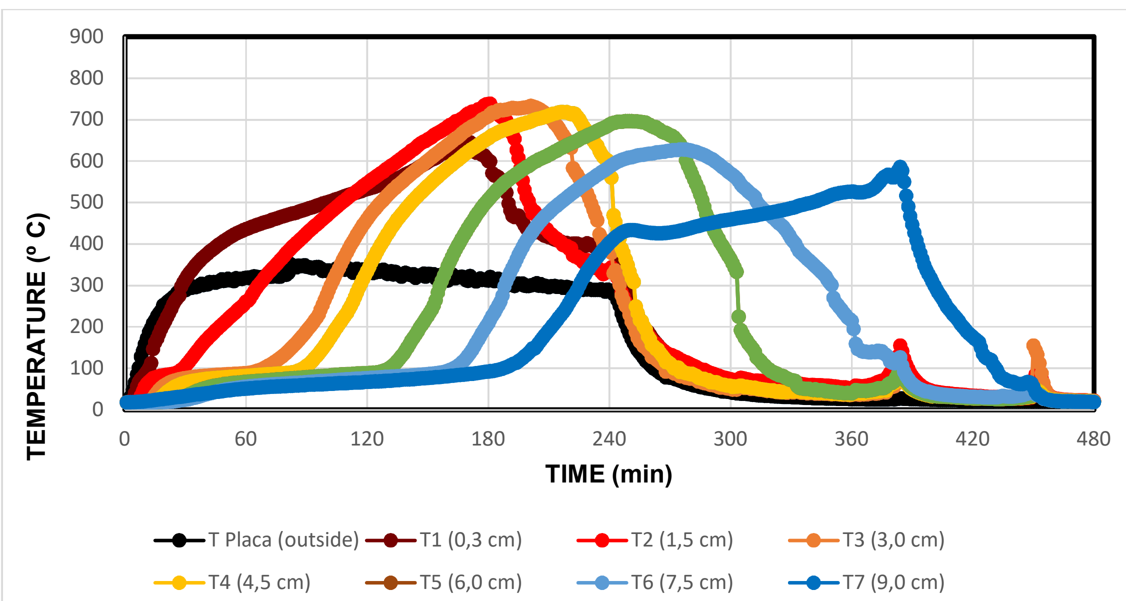

Figure B-4 - Wet corn flour without a ventilation system (F-Wet-NV_2).

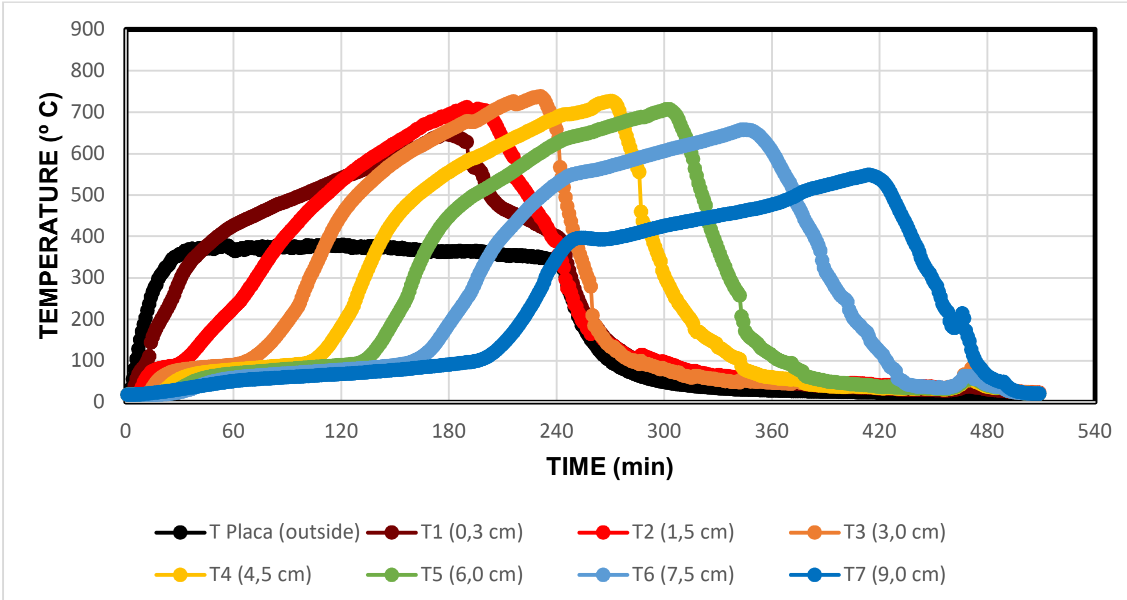

Figure B-5 - Wet corn flour without a ventilation system (F-Wet-NV_3).

Experiments with the samples of wet cornflour with the ventilation system.

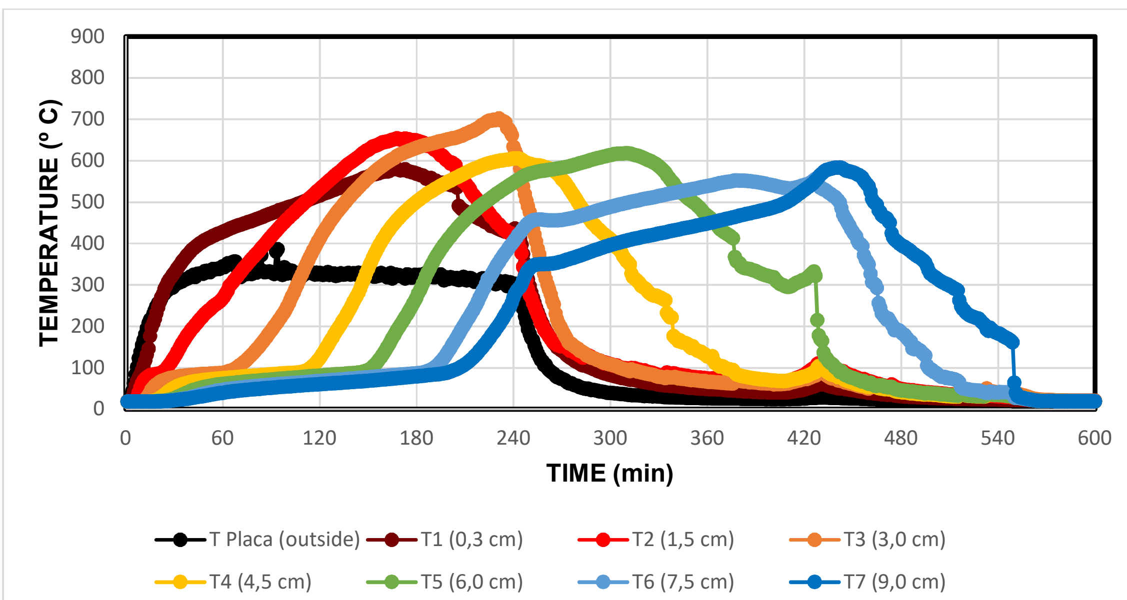

Figure B-6 - Wet corn flour with a ventilation system (F-Wet-V_1).

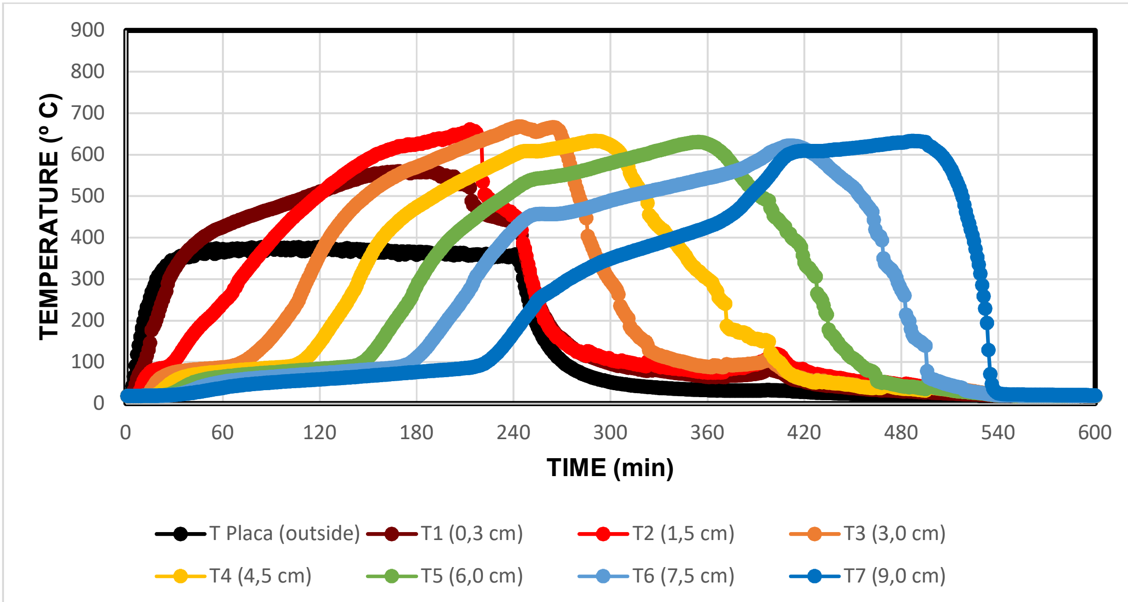

Figure B-7 - Wet corn flour with a ventilation system (F-Wet-V_3).

Experiments with the samples of dried grain 0.5 without a ventilation system.

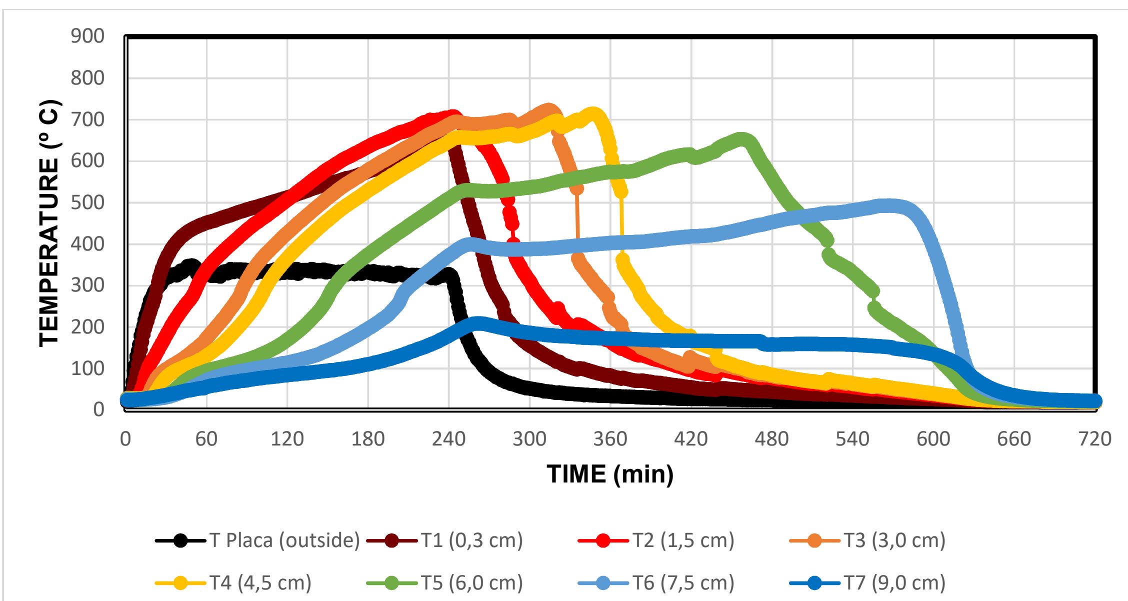

Figure B-8 - Dried grain 0.5 mm without a ventilation system (GS-Dried-NV_1).

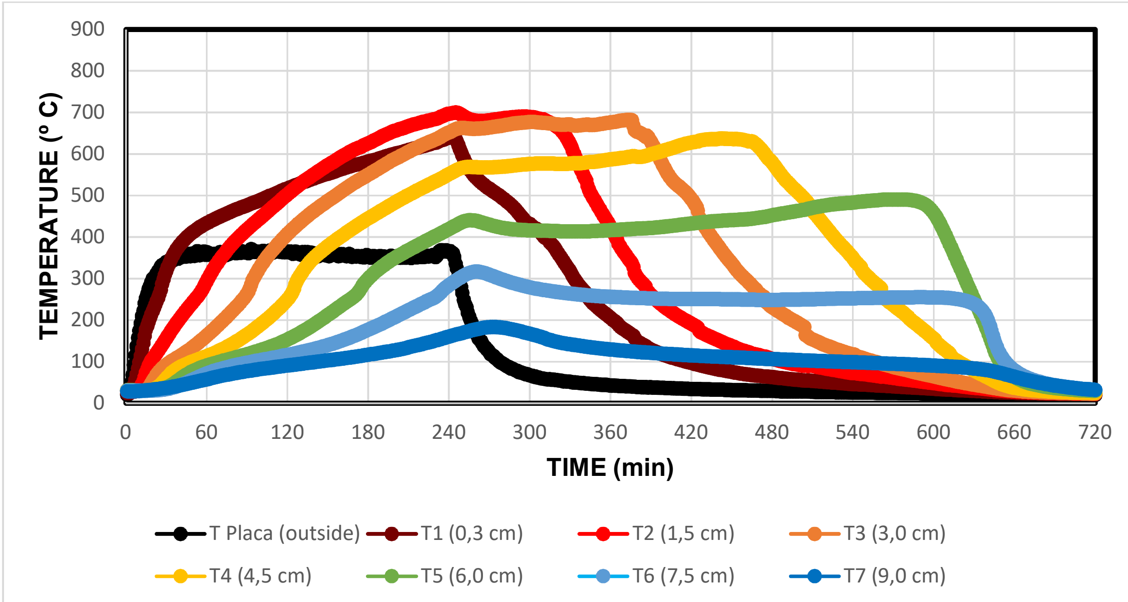

Figure B-9 - Dried grain 0.5 mm without a ventilation system (GS-Dried-NV_2).

Experiments with the samples of dried grain 0.5 with the ventilation system.

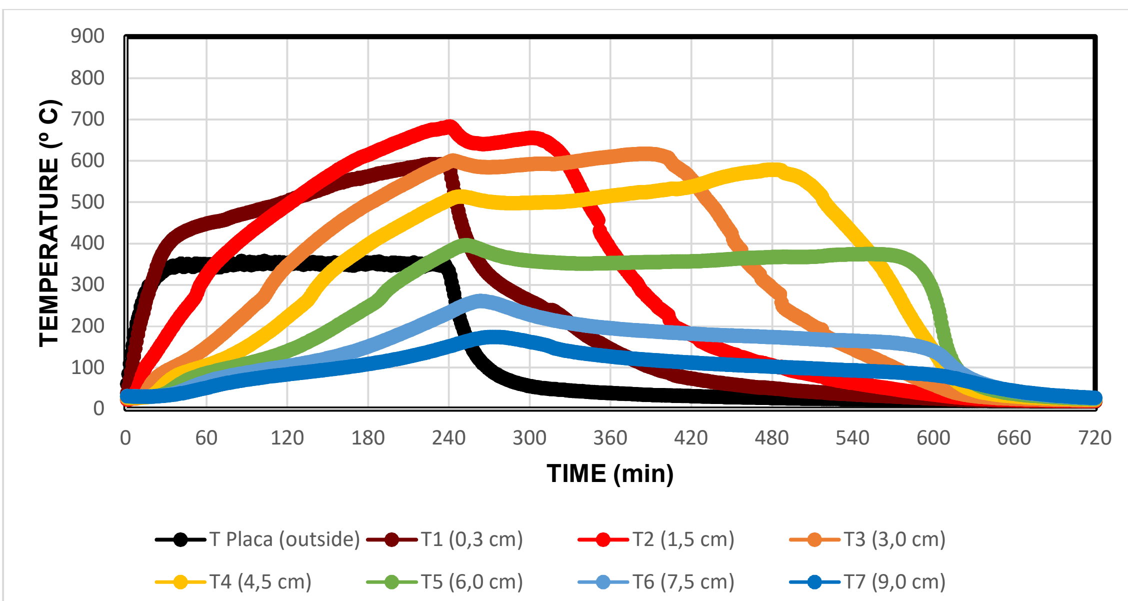

Figure B-10 - Dried grain 0.5 mm with a ventilation system (GS-Dried-V_1).

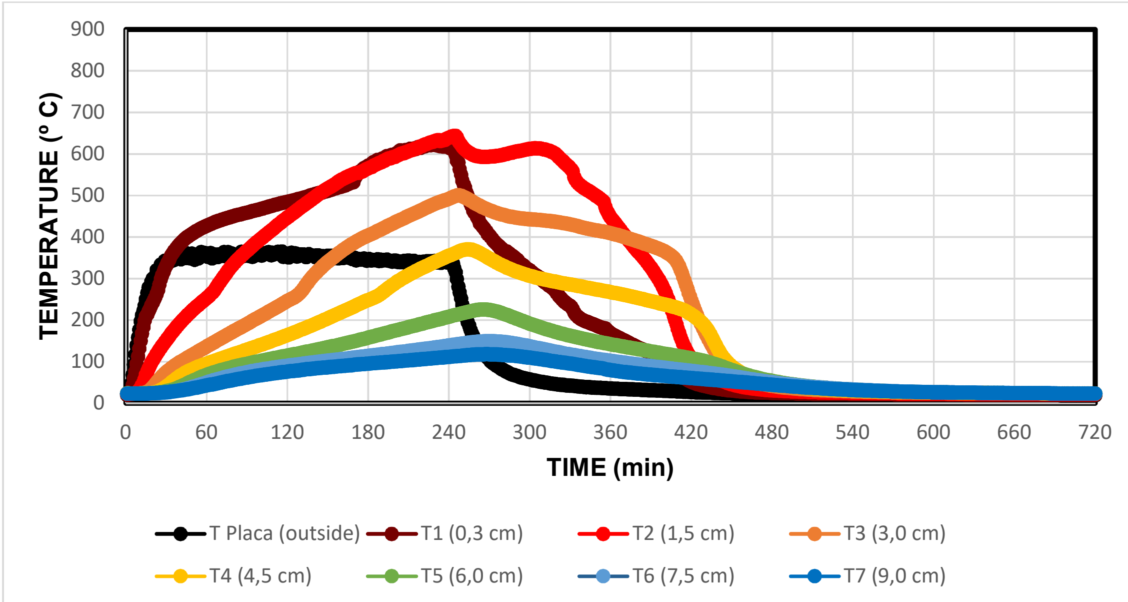

Figure B-11 - Dried grain 0.5 mm with a ventilation system (GS-Dried-V_2).

Experiments with the samples of wet grain 0.5 without a ventilation system.

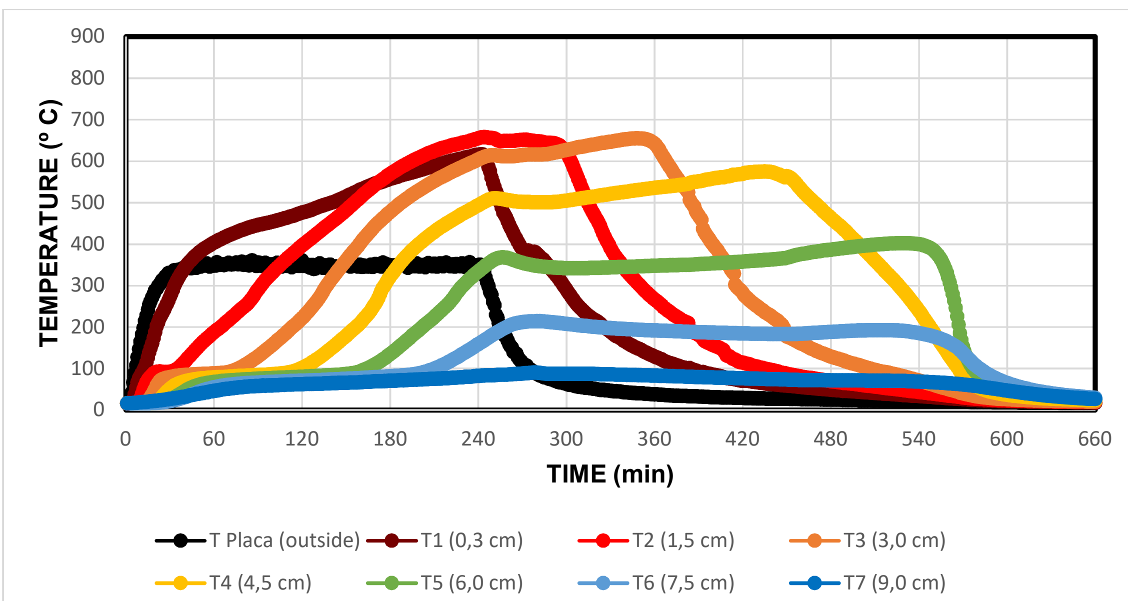

Figure B-12 - Wet grain 0.5 mm without a ventilation system (GS-Wet-NV_1).

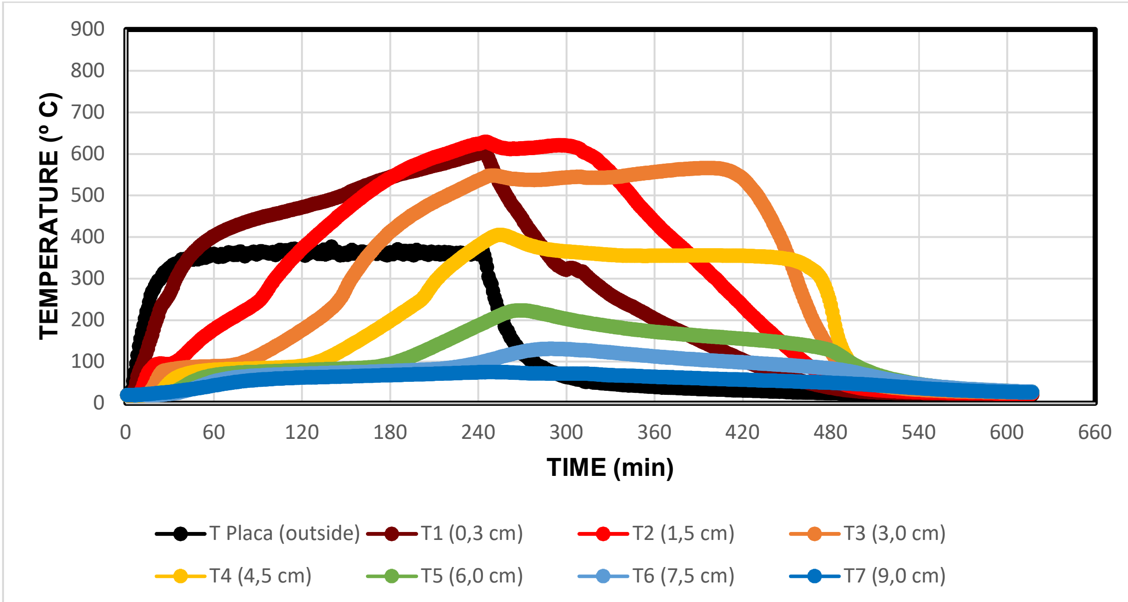

Figure B-13 - Wet grain 0.5 mm without a ventilation system (GS-Wet-NV_3).

Experiments with the samples of wet grain 0.5 with the ventilation system.

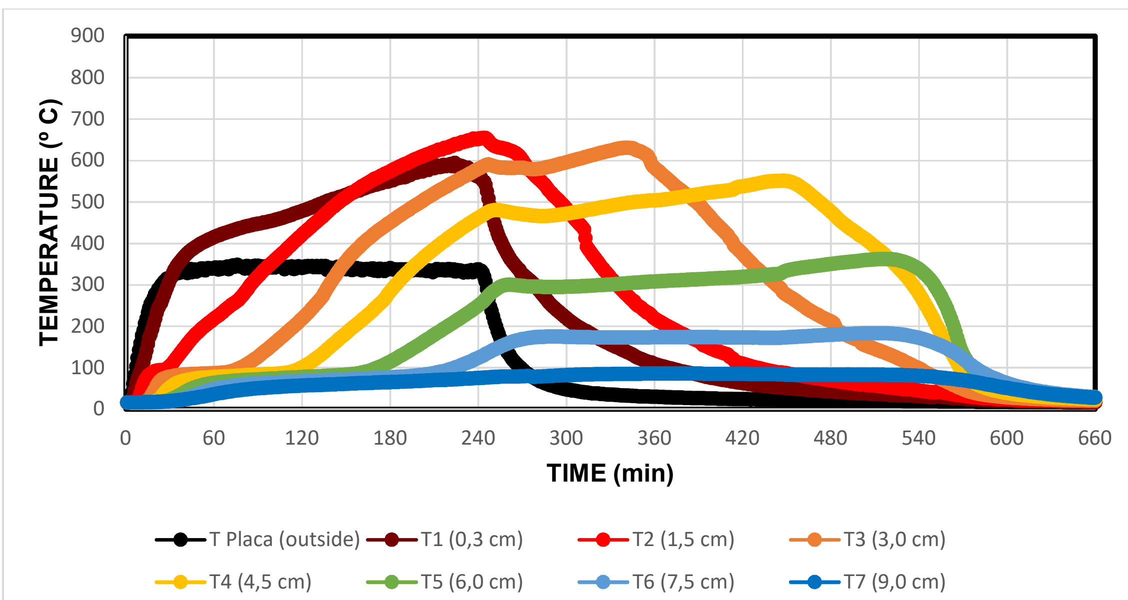

Figure B-14 - Wet grain 0.5 mm with a ventilation system (GS-Wet-V_1).

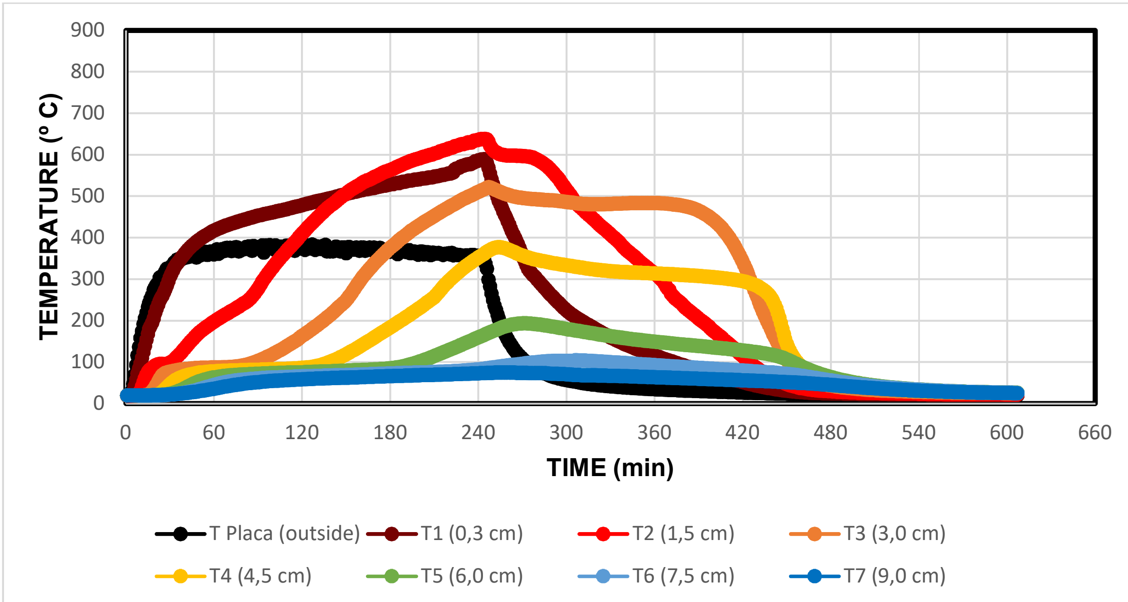

Figure B-15 - Wet grain 0.5 mm with a ventilation system (GS-Wet-V_3).

Extra experiments

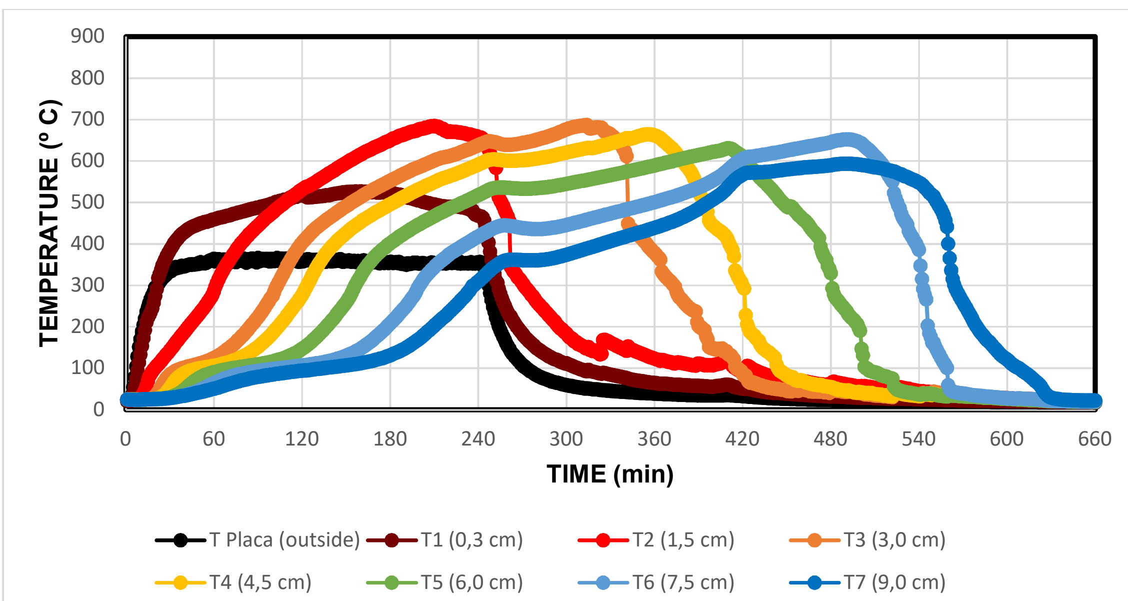

Figure B-16 - Dried cornflour with a 1h ventilation system (F-Dried-V(1h)_1).

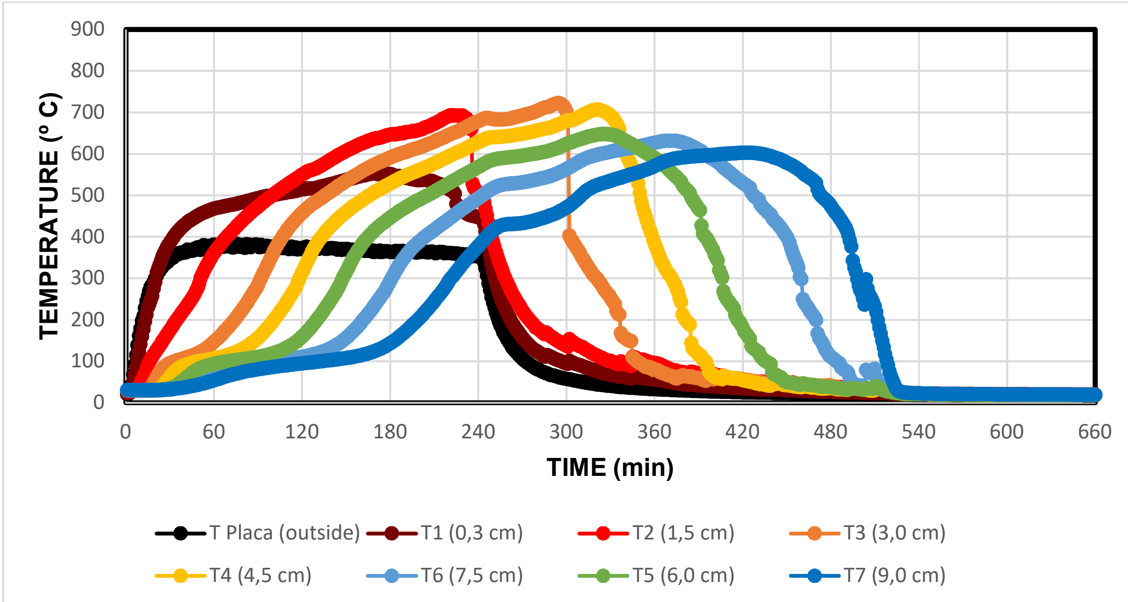

Figure B-17 - Dried cornflour with a 2h ventilation system (F-Dried-V(2h)_1).

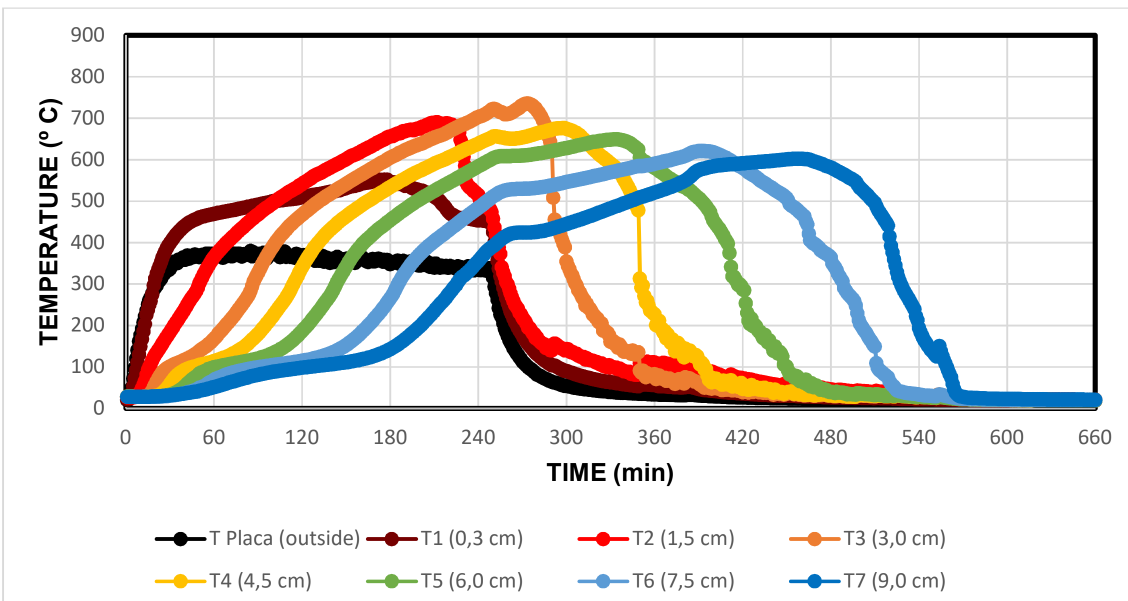

Figure B-18 - Dried corn flour with an 8h ventilation system (F-Dried-V(8h)_1).

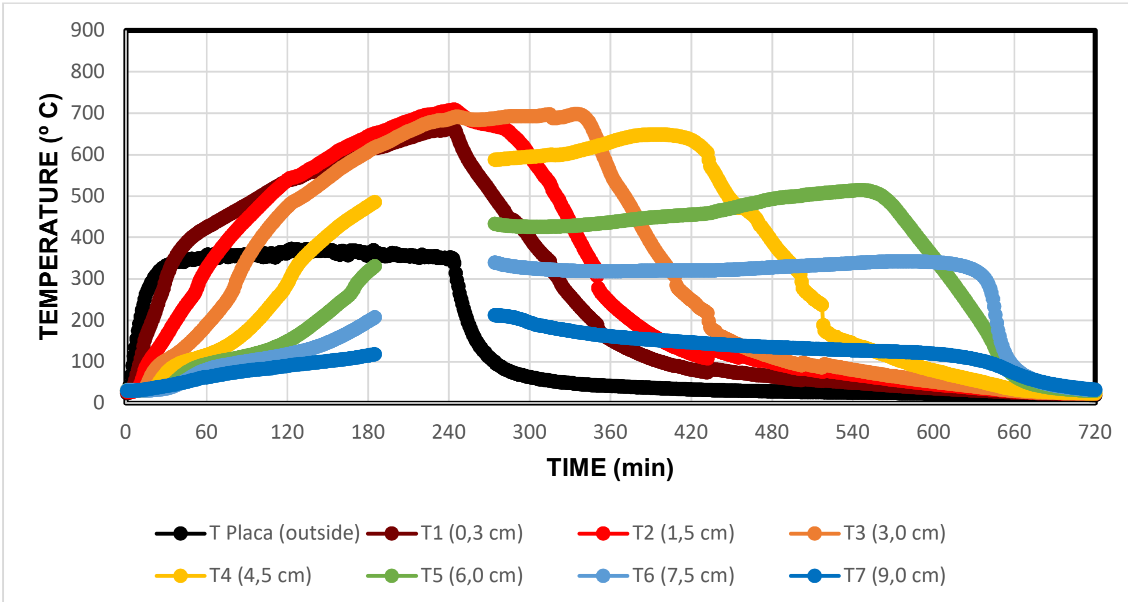

Figure B-19 - Dried grain 0.5 mm with 1h ventilation system (GS-Dried-V(1h)_1).

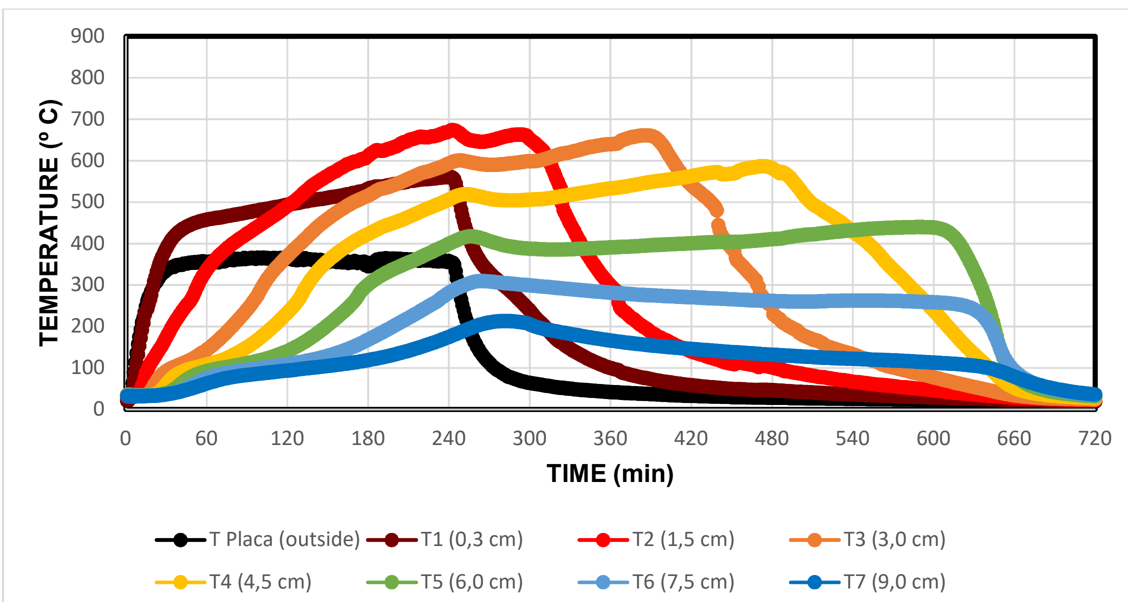

Figure B-20 - Dried grain 0.5 mm with a 2h ventilation system (GS-Dried-V(2h)_1).

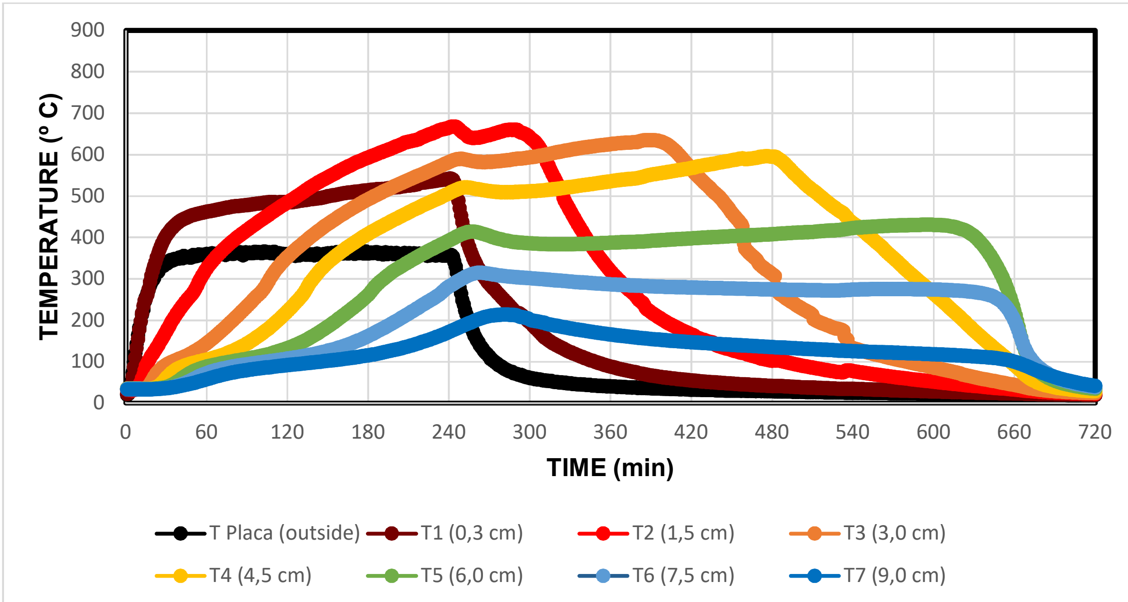

Figure B-21 - Dried grain 0.5 mm with 8h ventilation system (GS-Dried-V(8h)_1).

APPENDIX C - RESULTS OF EXPERIMENTS PERFORMED

Graphs with the relationship between the position of each thermocouple and the time it takes for the smoldering front to reach the temperature of 350º C.

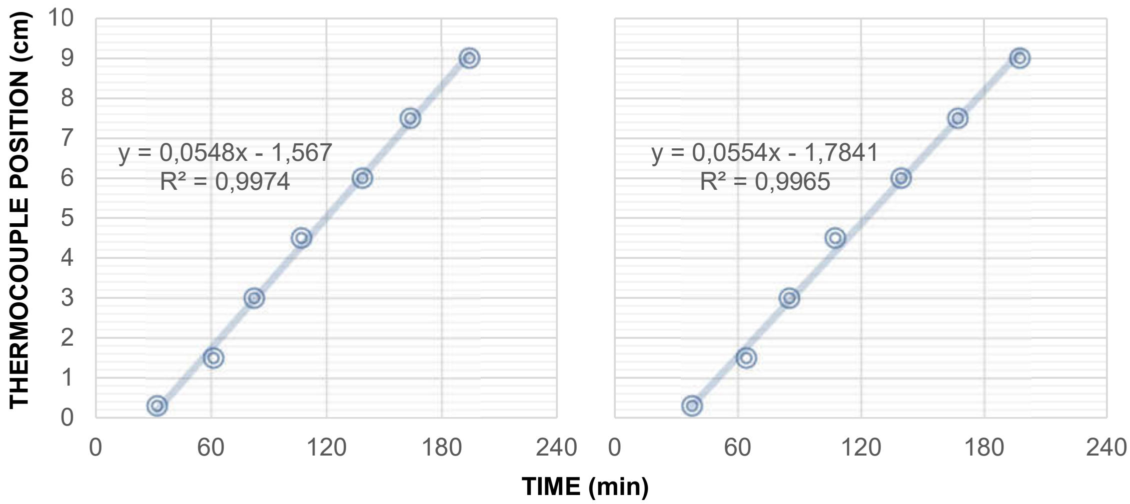

Figure C-1 - Dried corn flour without the ventilation system (F-Dried-NV) - sample 1 and sample 2.

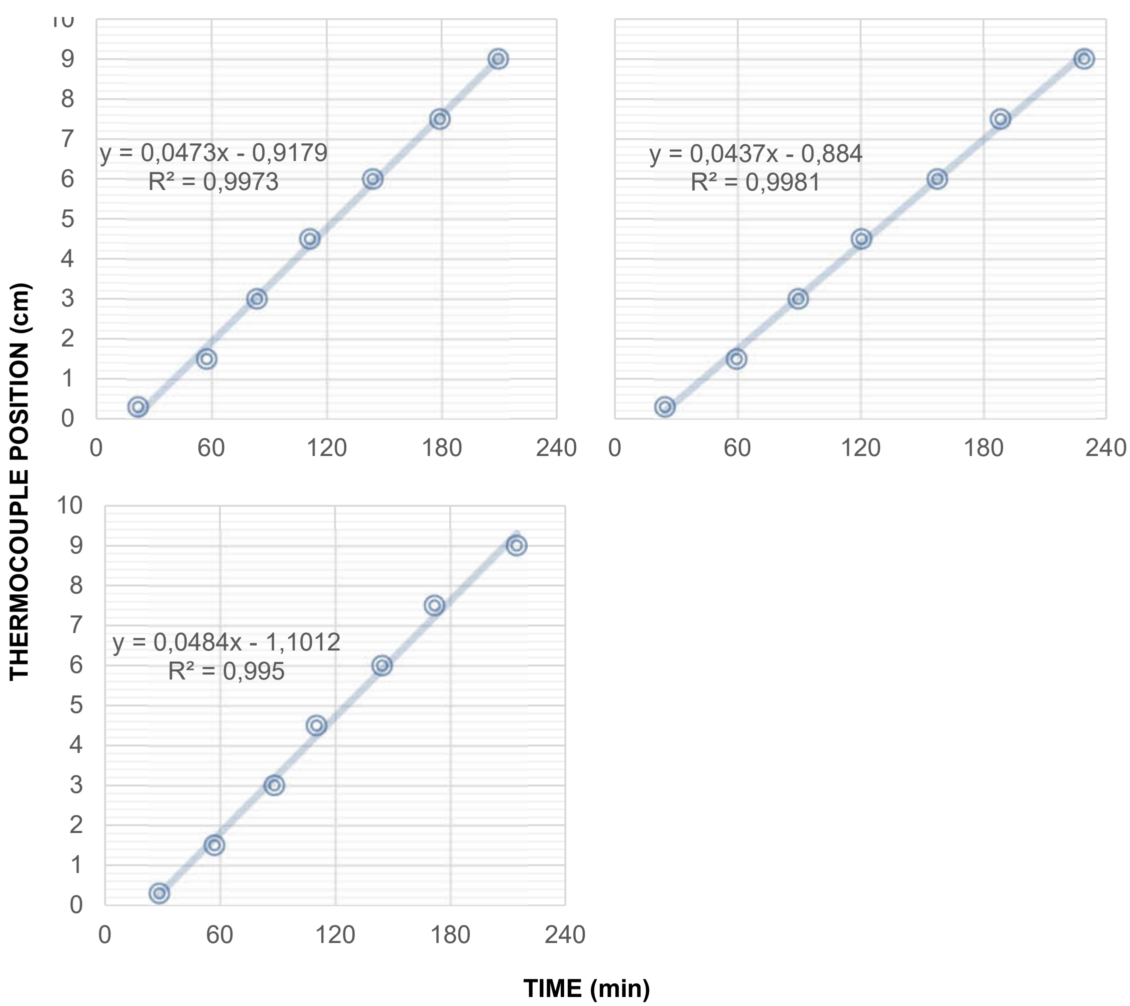

Figure C-2 - Dried corn flour with the ventilation system (F-Dried-V) - sample 1, sample 2 and sample 3.

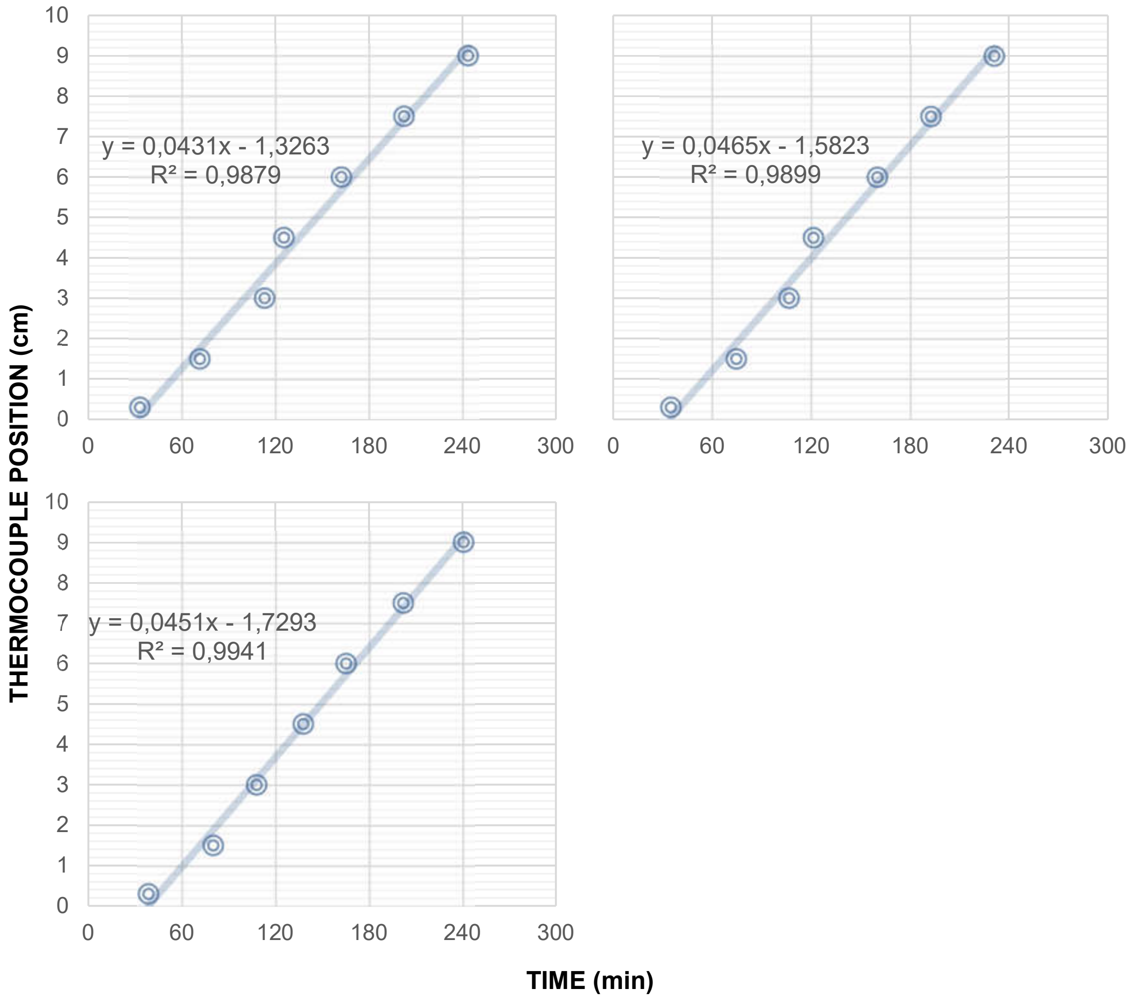

Figure C-3 - Wet corn flour without the ventilation system (F-Wet-NV) - sample 1, sample 2 and sample 3.

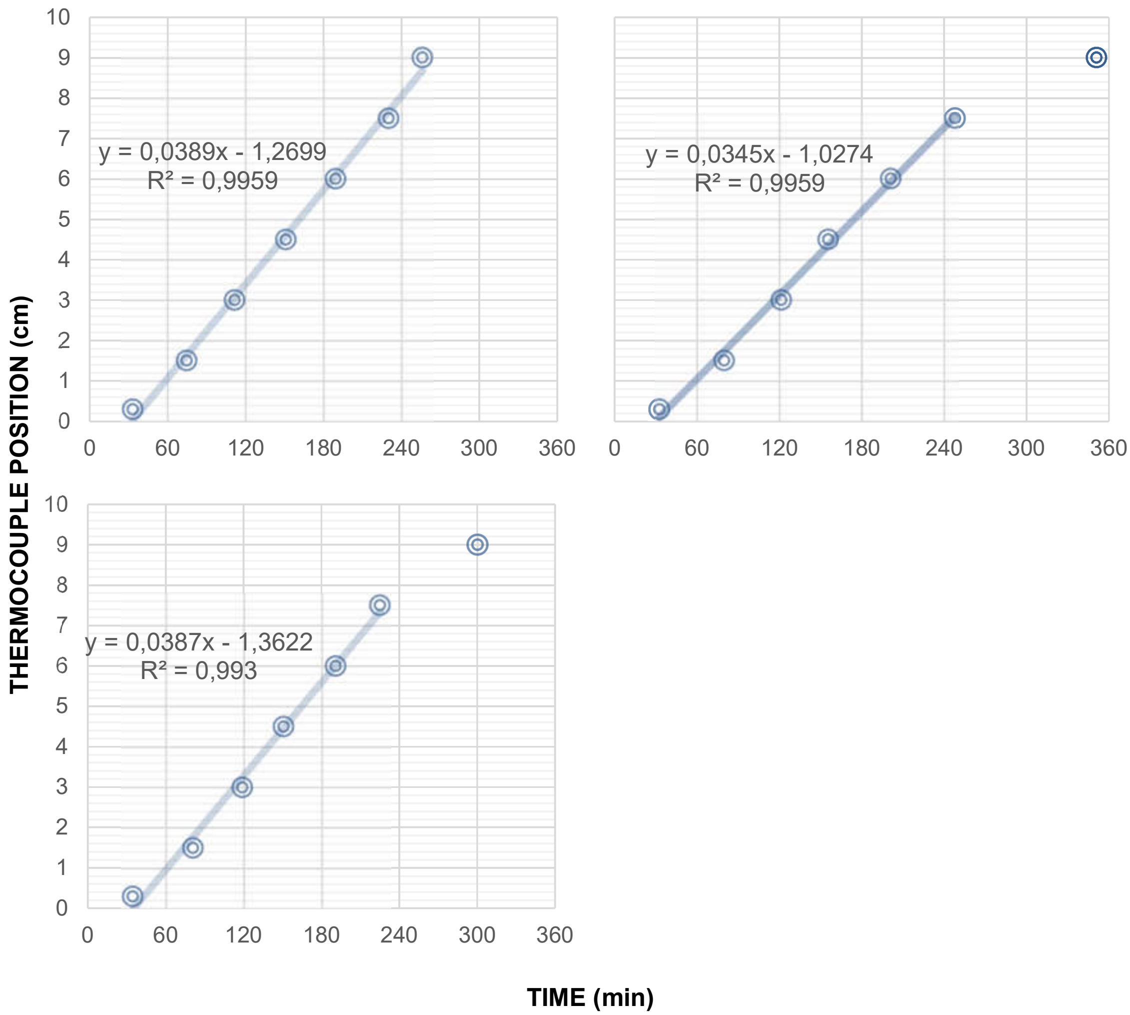

Figure C-4 - Wet corn flour with a ventilation system (F-Wet-V) - sample 1, sample 2 and sample 3.

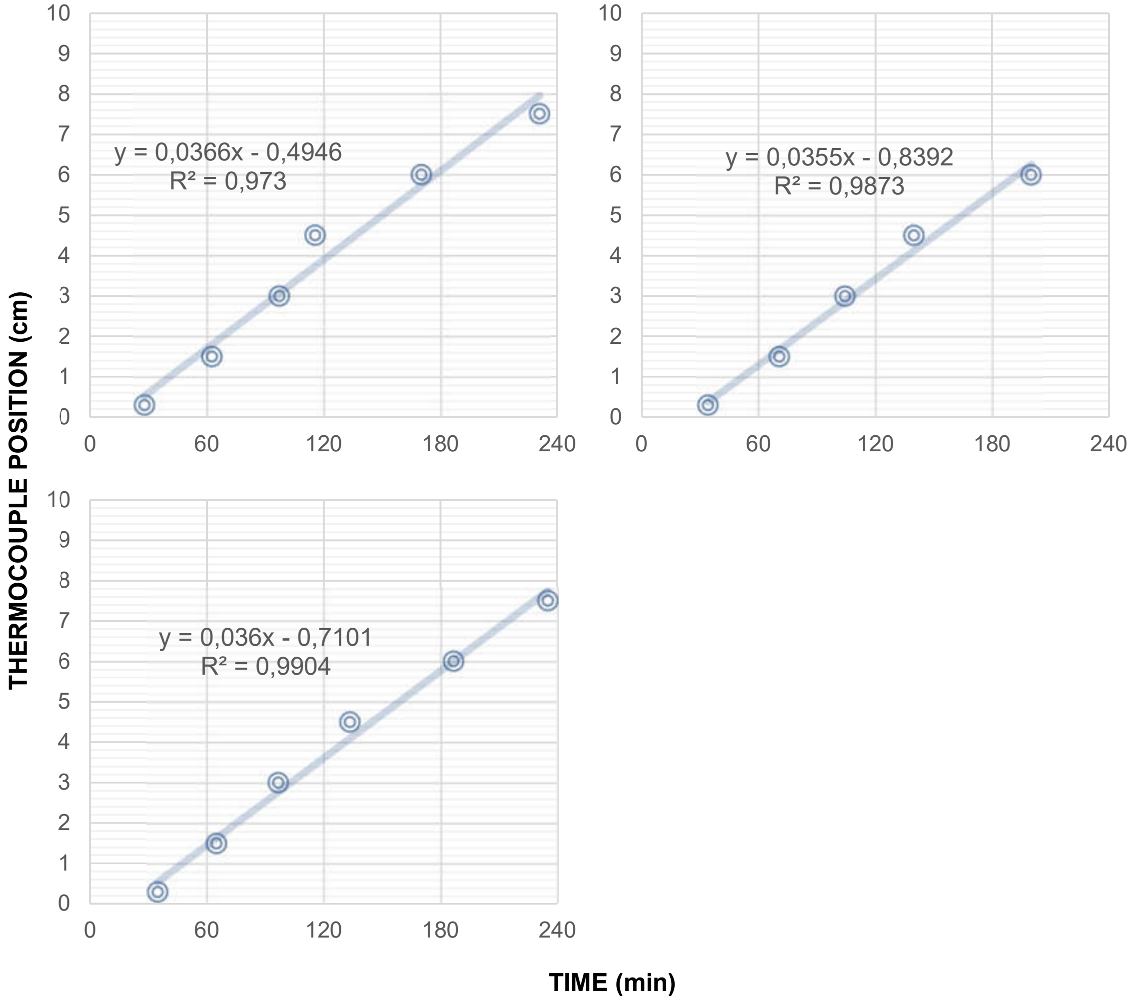

Figure C-5 - Dried grain (776.4µm) without the ventilation system (GS-Dried-NV) - sample 1, sample 2 and sample 3.

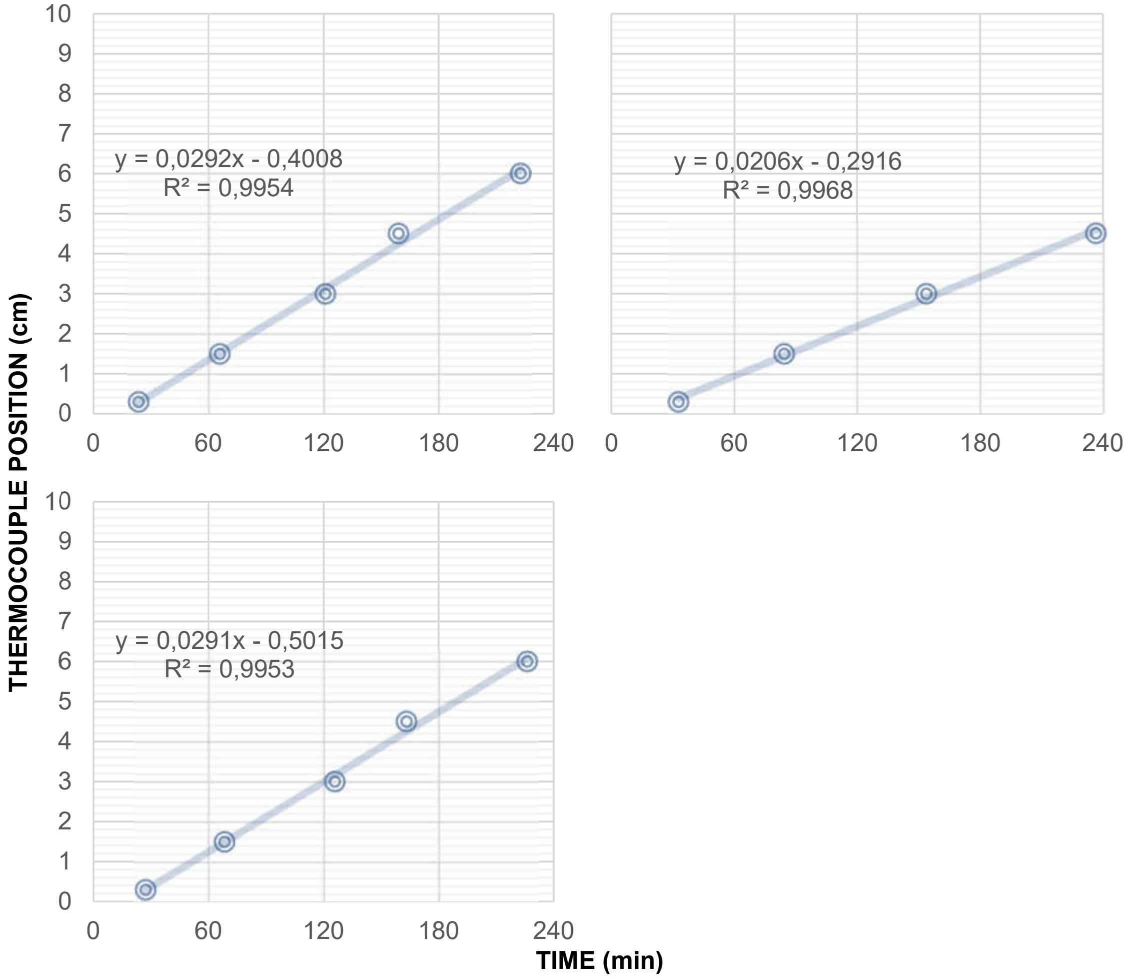

Figure C-6 - Dried grain (776.4 µm) witht a ventilation system (GS-Dried-V) - sample 1, sample 2 and sample 3.

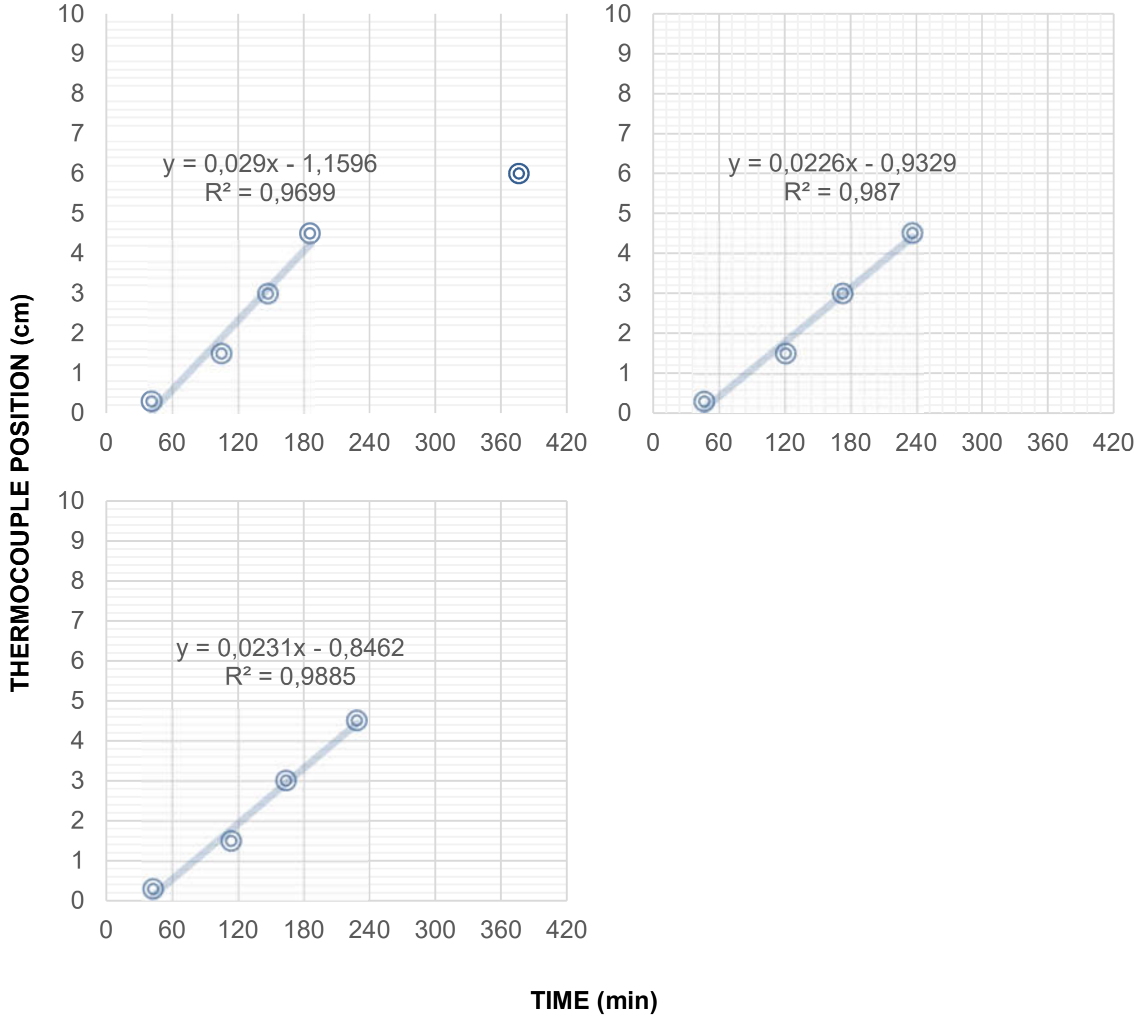

Figure C-7 - Wet grain (776.4 μm) without the system ventilation (GS-Wet-NV) - sample 1, sample 2, and sample 3.

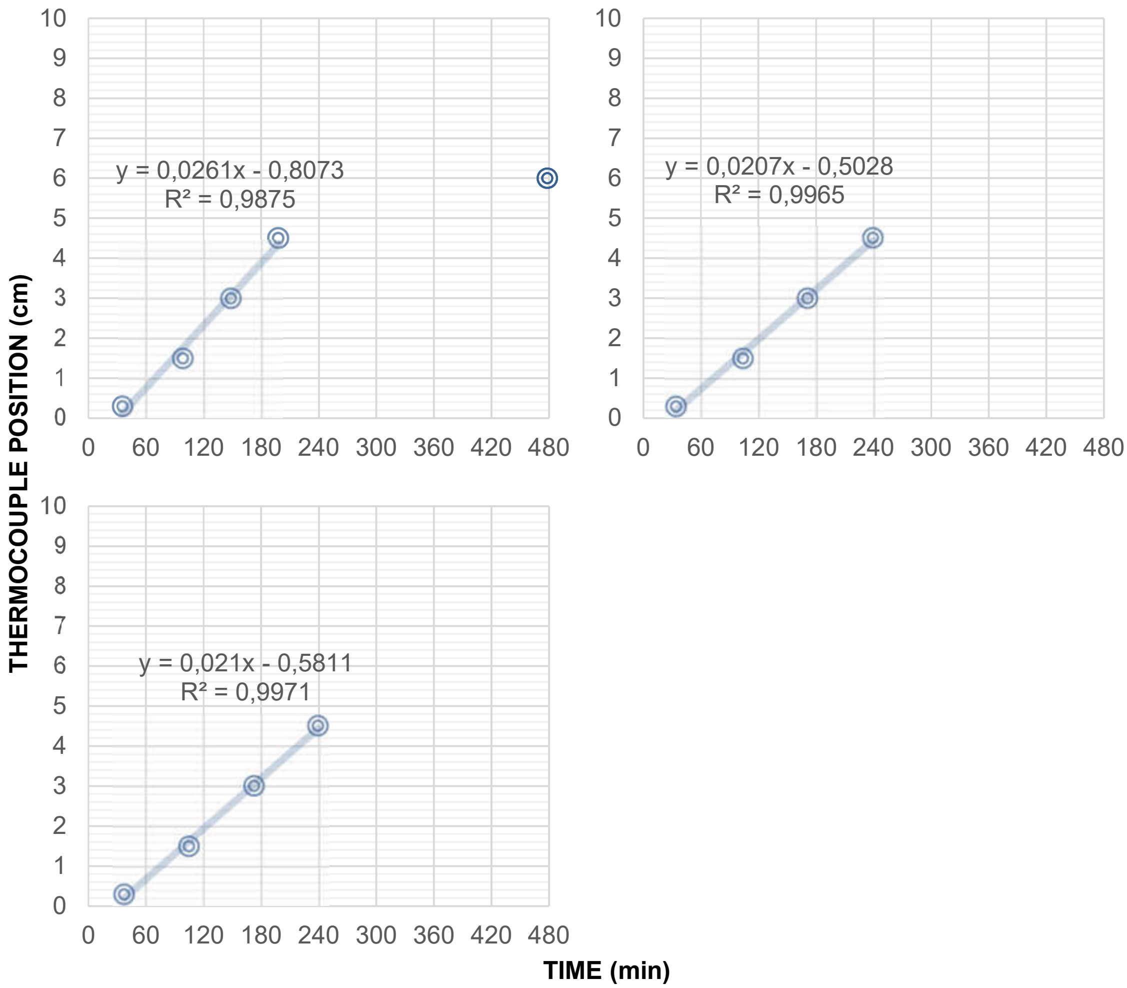

Figure C-8 - Wet grain (776.4 µm) with a system ventilation (GS-Wet-V) – sample 1, sample 2, and sample 3.

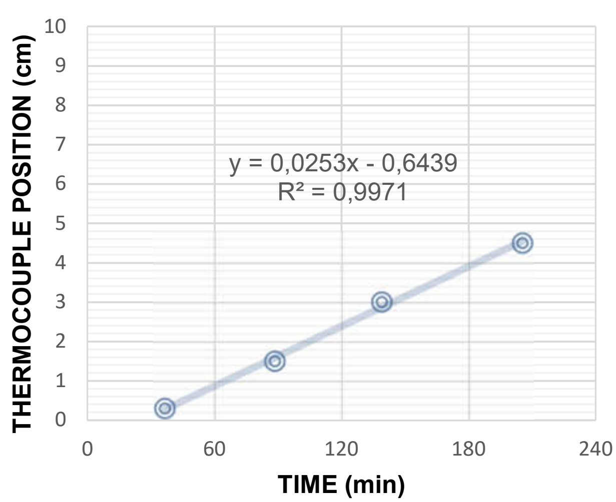

Figure C-9 - Dried grain (1411 µm) without a ventilation system (GL-Dried-NV) – sample 1.

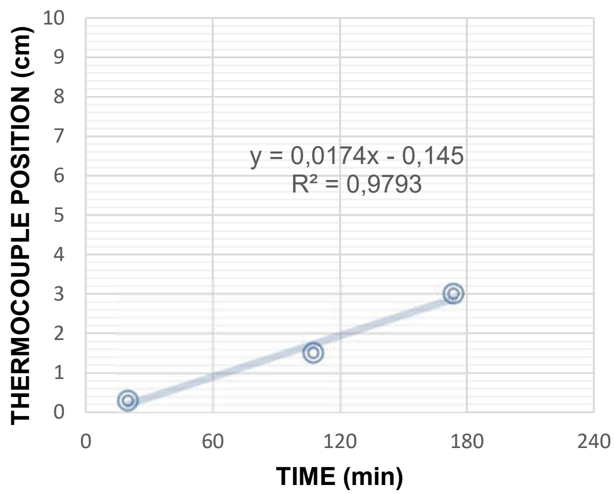

Figure C-10 - Wet grain (1411 μm) without a ventilation system (GL-Dried-NV) – sample 1.

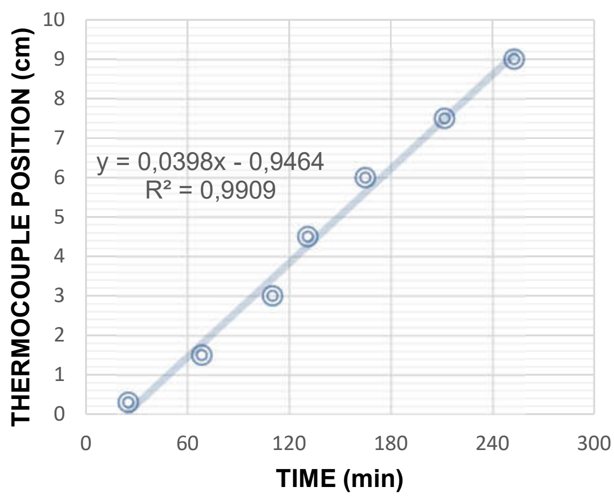

Figure C-11 - Dried corn flour with 1h ventilation system (F-Dried-V(1h)) – sample 1.

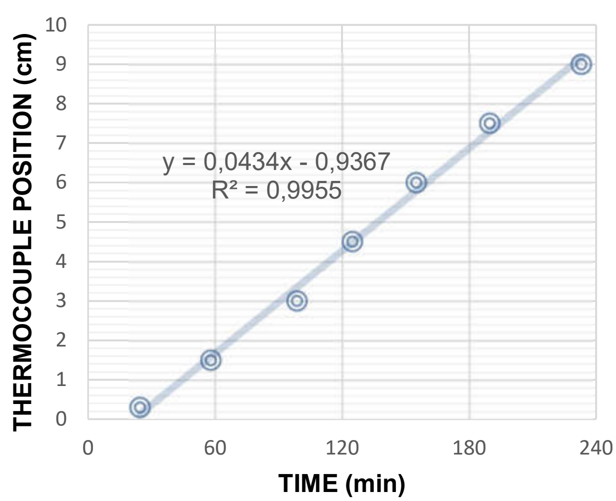

Figure C-12 - Dried corn flour with 2h ventilation system (F-Dried-V(2h)) – sample 1.

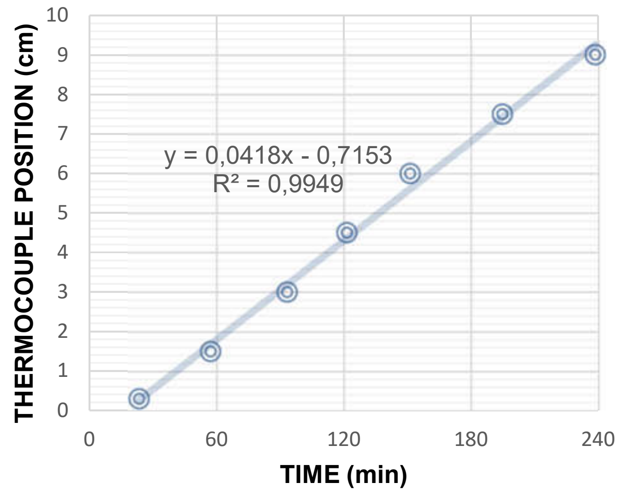

Figure C-13 - Dried corn flour with 8h ventilation system (F-Dried-V(8h)) – sample 1.

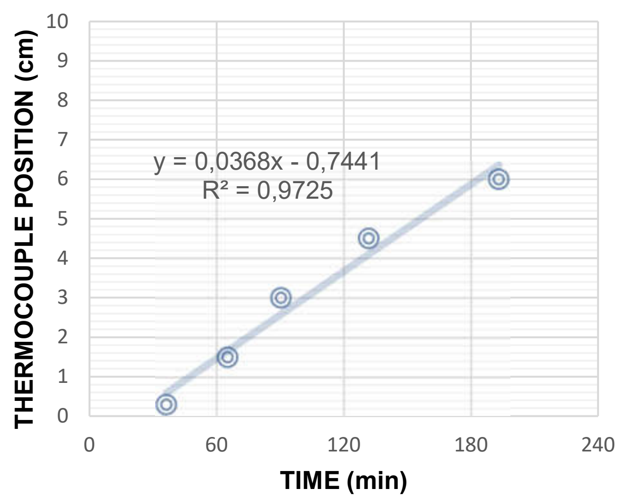

Figure C-14 - Dried grain (776.4 μm) with 1h ventilation system (GS-Dried-V(1h)) – sample 1.

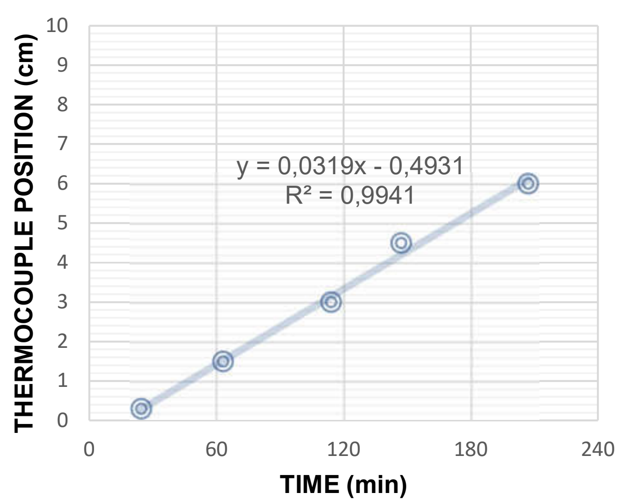

Figure C-15 - Dried grain (776.4 μm) with 2h ventilation system (GS-Dried-V(2h)) – sample 1.

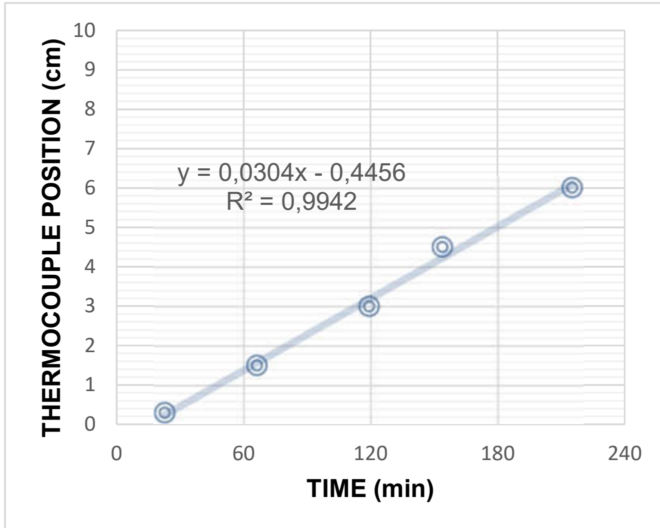

Figure C-16 - Dried grain (776.4 μm) with 8h ventilation system - (GS-Dried-V(8h)) – sample 1.

ANNEX A – PARTICLE SIZE ANALYSIS

Beckman Coulter LS Particle Size Analyzer

UNIVERSITAT DE BARCELONA

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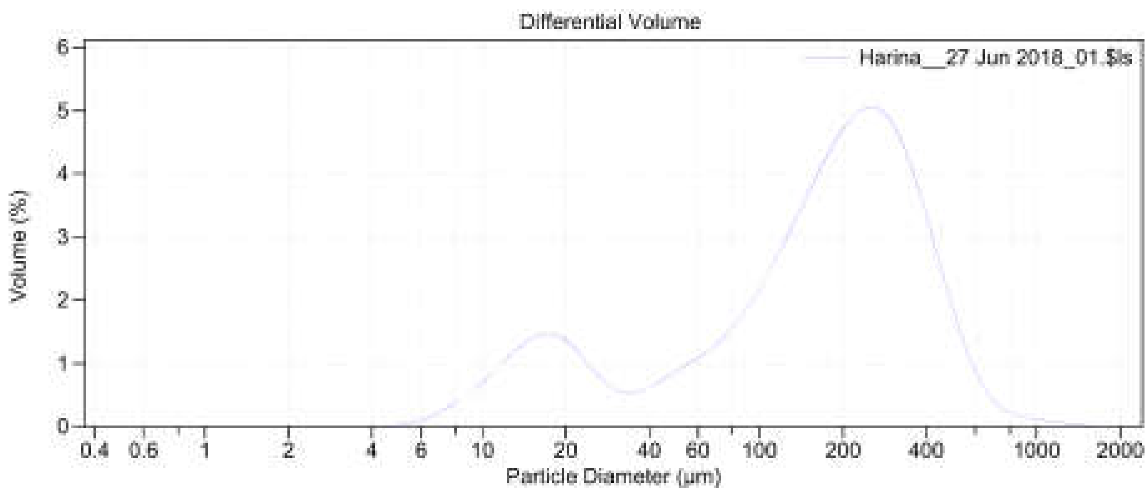
27 Jun 2018

Unitat de Citometria - Centres Científics i Tecnològics - UB

File name:	D:\Resultados 2018\p250\P250_2018060587\Harina__27 Jun 2018_01.$ls Harina__27 Jun 2018_01.$ls		
File ID:	Harina		
Operator:	Ricard Alvarez Arpal		
Reg CCiT:	20180602587		
Run number:	1		
Optical model:	Fraunhofer.rfd		
Residual:	0.67%		
LS 13 320	Dry Powder System		
Start time:	12:42 27 Jun 2018	Run length:	27 seconds
Average Vacuum:	17.3" H2O		
Obscuration:	6%		
Software:	6.01	Firmware:	2.02



Volume Statistics (Arithmetic) Harina__27 Jun 2018_01.$ls

Calculations from 0.375 µm to 2000 µm

Volume:	100%		
Mean:	205.1 µm	S.D.:	161.1 µm
Median:	181.6 µm	C.V.:	78.6%
D(3,2):	62.06 µm	Skewness:	1.405 Right skewed
Mode:	245.2 µm	Kurtosis:	4.220 Leptokurtic

d_{10}: 18.62 µm d_{50}: 181.6 µm d_{90}: 408.7 µm

<10%	<25%	<50%	<75%	<95%
18.62 µm	81.77 µm	181.6 µm	292.8 µm	490.1 µm

<1 µm	<10 µm	<100 µm	<1000 µm
0%	2.02%	29.1%	99.7%

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Harina__27 Jun 2018_01.$ls

Channel Diameter (Lower) µm	Diff. Volume %	Cum. < Volume %
0.375124	0	0
0.411798	0	0
0.452057	0	0
0.496252	0	0
0.544768	0	0
0.598027	0	0
0.656493	0	0
0.720675	0	0
0.791132	0	0
0.868477	0	0
0.953383	0	0
1.04659	0	0
1.14891	0	0
1.26123	0	0
1.38454	0	0
1.5199	0	0
1.66849	0	0
1.83161	0	0
2.01068	0	0
2.20725	0	0
2.42304	0	0
2.65993	0	0
2.91998	0	0
3.20545	0	0
3.51883	0	0
3.86284	2.28525E-5	0
4.24049	0.000976509	2.28525E-5
4.65506	0.00820505	0.000999361
5.11017	0.0337607	0.00920441
5.60976	0.0822856	0.0429651
6.1582	0.150405	0.125251
6.76025	0.231389	0.275655
7.42117	0.330073	0.507044
8.14669	0.44959	0.837117
8.94315	0.590869	1.28671
9.81748	0.75136	1.87758
10.7773	0.922822	2.62894
11.8309	1.09564	3.55176
12.9876	1.25518	4.6474
14.2573	1.38407	5.90258
15.6512	1.46003	7.28665
17.1813	1.46308	8.74668
18.861	1.3829	10.2098
20.705	1.22603	11.5927
22.7292	1.02117	12.8187
24.9513	0.811626	13.8399
27.3906	0.642526	14.6515
30.0685	0.542262	15.294
33.0081	0.517789	15.8363
36.2352	0.560478	16.3541
39.7777	0.650612	16.9145
43.6665	0.763357	17.5651
47.9356	0.877478	18.3285
52.622	0.985817	19.206
57.7666	1.09517	20.1918
63.4141	1.21963	21.287
69.6138	1.37316	22.5066
76.4196	1.56424	23.8798
83.8907	1.79511	25.444
92.0923	2.06358	27.2391
101.096	2.36563	29.3027
110.979	2.69712	31.6683
121.829	3.05245	34.3654
133.74	3.42287	37.4179
146.815	3.79547	40.8408
161.168	4.15418	44.6362
176.925	4.48134	48.7904

Channel Diameter (Lower) µm	Diff. Volume %	Cum. < Volume %
194.222	4.75795	53.2717
213.21	4.96198	58.0297
234.054	5.06703	62.9917
256.936	5.04487	68.0587
282.056	4.87299	73.1036
309.631	4.54423	77.9766
339.902	4.07348	82.5208
373.132	3.49661	86.5943
409.611	2.8625	90.0909
449.657	2.22281	92.9534
493.617	1.62533	95.1762
541.876	1.11133	96.8015
594.852	0.712029	97.9129
653.008	0.438883	98.6249
716.849	0.276679	99.0638
786.932	0.190915	99.3405
863.866	0.145978	99.5314
948.322	0.115456	99.6774
1041.03	0.0678954	99.7928
1142.81	0.0624937	99.8807
1254.54	0.0367061	99.9432
1377.19	0.0162143	99.9799
1511.83	0.00354083	99.9961
1659.63	0.000347676	99.9996
1821.88	0	100
2000		100

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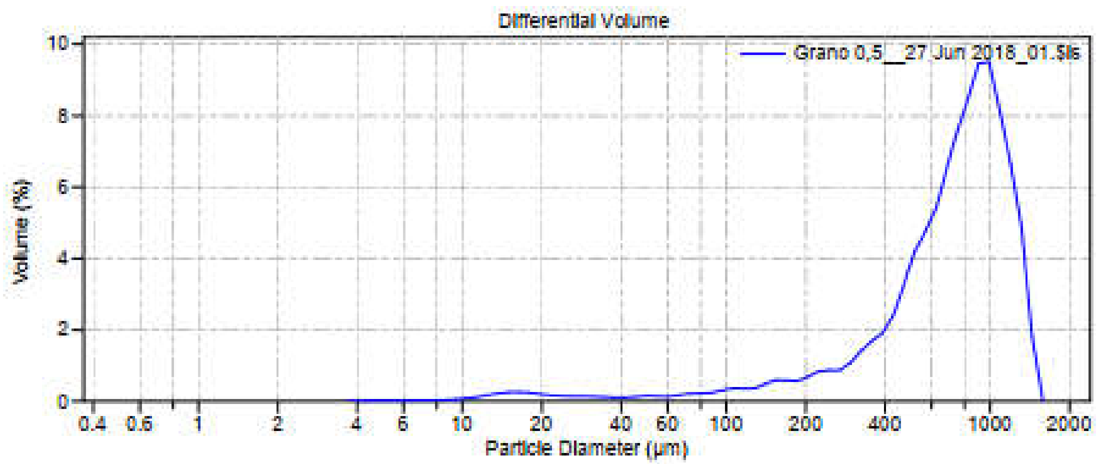
File name:	D:\Resultados 2018\p250\P250_20180602587\Grano 0,5__27 Jun 2018_01.$ls
	Grano 0,5__27 Jun 2018_01.$ls
File ID:	Grano 0,5
Operator:	Ricard Alvarez Arpal
Reg CCIT:	20180602587
Run number:	1
Optical model:	Fraunhofer.rfd
Residual:	0.91%
LS 13 320	Dry Powder System
Start time:	12:24 27 Jun 2018
Run length:	27 seconds
Average Vacuum:	16.4" H2O
Obscuration:	8%
Software:	6.01
Firmware:	2.02



Volume Statistics (Arithmetic) Grano 0,5__27 Jun 2018_01.$ls

Calculations from 0.375 µm to 2000 µm

Volume:	100%		
Mean:	752.9 µm	S.D.:	343.9 µm
Median:	776.4 µm	C.V.:	45.7%
D(3,2):	286.2 µm	Skewness:	-0.229 Left skewed
Mode:	993.6 µm	Kurtosis:	-0.586 Platykurtic

d_{10}: 253.3 µm d_{50}: 776.4 µm d_{90}: 1199 µm

<10%	<25%	<50%	<75%	<95%
253.3 µm	519.6 µm	776.4 µm	1006 µm	1298 µm

<1 µm	<10 µm	<100 µm	<1000 µm
0%	0.16%	4.41%	74.4%

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Grano 0,5__27 Jun 2018_01.$ls

Channel Diameter (Lower) µm	Diff. Volume %	Cum. < Volume %
0.375124	0	0
0.411798	0	0
0.452057	0	0
0.496252	0	0
0.544768	0	0
0.598027	0	0
0.656493	0	0
0.720675	0	0
0.791132	0	0
0.868477	0	0
0.953383	0	0
1.04659	0	0
1.14891	0	0
1.26123	0	0
1.38454	0	0
1.5199	0	0
1.66849	0	0
1.83161	0	0
2.01068	0	0
2.20725	0	0
2.42304	0	0
2.65993	0	0
2.91998	0	0
3.20545	0	0
3.51883	0.0019334	0
3.86284	0.00634798	0.0019334
4.24049	0.010649	0.00828138
4.65506	0.0129226	0.0189303
5.11017	0.012706	0.0318529
5.60976	0.0111736	0.0445589
6.1582	0.0103099	0.0557326
6.76025	0.0118186	0.0660425
7.42117	0.0169804	0.0778611
8.14669	0.0270921	0.0948415
8.94315	0.0442046	0.121934
9.81748	0.071712	0.166138
10.7773	0.112569	0.23785
11.8309	0.164833	0.350419
12.9876	0.218255	0.515252
14.2573	0.256166	0.733507
15.6512	0.263966	0.989673
17.1813	0.240886	1.25364
18.861	0.201928	1.49452
20.705	0.166098	1.69645
22.7292	0.144909	1.86255
24.9513	0.138859	2.00746
27.3906	0.140097	2.14632
30.0685	0.136424	2.28642
33.0081	0.11897	2.42284
36.2352	0.0991959	2.54181
39.7777	0.101424	2.64101
43.6665	0.130142	2.74243
47.9356	0.151968	2.87257
52.622	0.139118	3.02454
57.7666	0.13447	3.16366
63.4141	0.172589	3.29813
69.6138	0.211373	3.47072
76.4196	0.219979	3.68209
83.8907	0.24335	3.90207
92.0923	0.305312	4.14542
101.096	0.364269	4.45073
110.979	0.358241	4.815
121.829	0.363073	5.17324
133.74	0.476049	5.53632
146.815	0.592436	6.01236
161.168	0.585973	6.6048
176.925	0.565347	7.19077

Channel Diameter (Lower) µm	Diff. Volume %	Cum. < Volume %
194.222	0.670058	7.75612
213.21	0.838123	8.42618
234.054	0.873464	9.2643
256.936	0.87181	10.1378
282.056	1.06917	11.0096
309.631	1.41704	12.0787
339.902	1.68523	13.4958
373.132	1.90504	15.181
409.611	2.39324	17.0861
449.657	3.27614	19.4793
493.617	4.17179	22.7554
541.876	4.74927	26.9272
594.852	5.39996	31.6765
653.008	6.49176	37.0765
716.849	7.57113	43.5682
786.932	8.46837	51.1394
863.866	9.45898	59.6077
948.322	9.49916	69.0667
1041.03	8.07837	78.5659
1142.81	6.63291	86.6442
1254.54	4.89404	93.2771
1377.19	1.82882	98.1712
1511.83	0	100
1659.63	0	100
1821.88	0	100
2000		100

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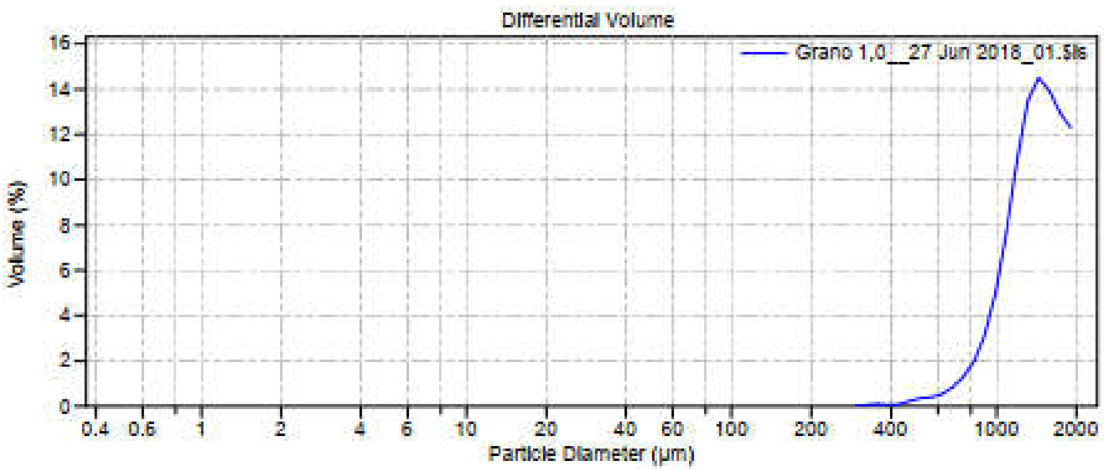
File name:	D:\Resultados 2018\p250\P250_2018060587\Grano 1,0__27 Jun 2018_01.$ls
	Grano 1,0__27 Jun 2018_01.$ls
File ID:	Grano 1,0
Operator:	Ricard Alvarez Arpal
Reg CCIT:	20180602587
Run number:	1
Optical model:	Fraunhofer.rfd
Residual:	1.38%
LS 13 320	Dry Powder System
Start time:	12:22 27 Jun 2018
Run length:	19 seconds
Average Vacuum:	16.8" H2O
Obscuration:	6%
Software:	6.01
Firmware:	2.02



Volume Statistics (Arithmetic) Grano 1,0__27 Jun 2018_01.$ls

Calculations from 0.375 µm to 2000 µm

Volume:	100%		
Mean:	1403 µm	S.D.:	333.0 µm
Median:	1411 µm	C.V.:	23.7%
D(3,2):	1304 µm	Skewness:	-0.324 Left skewed
Mode:	1443 µm	Kurtosis:	-0.393 Platykurtic

d_{10}: 964.1 µm d_{50}: 1411 µm d_{90}: 1855 µm

<10%	<25%	<50%	<75%	<95%
964.1 µm	1174 µm	1411 µm	1662 µm	1928 µm

<1 µm	<10 µm	<100 µm	<1000 µm
0%	0%	0%	12.0%

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Grano 1,0__27 Jun 2018_01.$ls

Channel Diameter (Lower) µm	Diff. Volume %	Cum. < Volume %
0.375124	0	0
0.411796	0	0
0.452057	0	0
0.496252	0	0
0.544768	0	0
0.598027	0	0
0.656493	0	0
0.720675	0	0
0.791132	0	0
0.868477	0	0
0.953383	0	0
1.04659	0	0
1.14891	0	0
1.26123	0	0
1.38454	0	0
1.5199	0	0
1.66849	0	0
1.83161	0	0
2.01068	0	0
2.20725	0	0
2.42304	0	0
2.65993	0	0
2.91998	0	0
3.20545	0	0
3.51883	0	0
3.86284	0	0
4.24049	0	0
4.65506	0	0
5.11017	0	0
5.60976	0	0
6.1582	0	0
6.76025	0	0
7.42117	0	0
8.14669	0	0
8.94315	0	0
9.81748	0	0
10.7773	0	0
11.8309	0	0
12.9876	0	0
14.2573	0	0
15.6512	0	0
17.1813	0	0
18.861	0	0
20.705	0	0
22.7292	0	0
24.9513	0	0
27.3906	0	0
30.0685	0	0
33.0081	0	0
36.2352	0	0
39.7777	0	0
43.6665	0	0
47.9356	0	0
52.622	0	0
57.7666	0	0
63.4141	0	0
69.6138	0	0
76.4196	0	0
83.8907	0	0
92.0923	0	0
101.096	0	0
110.979	0	0
121.829	0	0
133.74	0	0
146.815	0	0
161.168	0	0
176.925	0	0
194.222	0	0
213.21	0	0
234.054	0	0
256.936	0	0
282.056	0	0
309.631	0.0627594	0
339.902	0.0986017	0.0627594
373.132	0.0490537	0.161361
409.611	0.0732639	0.210415
449.657	0.229418	0.283679
493.617	0.358072	0.513097
541.876	0.401189	0.871169
594.852	0.536615	1.27236
653.008	0.832945	1.80897
716.849	1.28963	2.64192
786.932	2.00585	3.93155
863.866	3.19836	5.9374
948.322	5.07438	9.13576
1041.03	7.72909	14.2101
1142.81	10.8855	21.9392
1254.54	13.5183	32.8247
1377.19	14.5016	46.343
1511.83	13.9325	60.8446
1659.63	12.919	74.7771
1821.88	12.3039	87.6961
2000		100