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Development of Speditive Explosibility Test (SET): a statistical reliable method for combustible dust explosibility investigation

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PhD Thesis

Development of Speditive Explosibility Test (SET):
a statistical reliable method for combustible dust explosibility investigation

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"For a long time, people were saying that most accidents were due to human error and this is true in a sense but it's not very helpful. It's a bit like saying that falls are due to gravity." **– Dr Trevor Kletz**

Out of this nettle, danger, we pluck this flower, safety.

William Shakespeare

Abstract

The present work of thesis investigate the explosibility sensitivity and behavior of combustible solid materials, in the form of dusts.

The first phase of the work has focused on the ignition sensitivity of combustible dusts, both in form of clouds than deposed as layers. Standard test methods has been used to assess ignition parameter of the samples, i.e. UNI EN 50821: 1999.

MITC and MITL were measured for pure combustible dusts and for mixtures of different dusts. In particular mixtures of combustible and inert dusts were analyzed. The experimental results underlined the increase of the ignition temperature (both in clouds than in layers) as the weight content of inert material increase. Extinguishing powders with chemical heat depletion effect had demonstrated to be more effective in increasing the ignition temperature with respect to inert acting with physical inertia mechanism. MITC of two combustible dusts mixture have been analyzed and results have shown the poor accuracy of the standard procedure in defining the ignition temperature, particularly when there is little difference between ignition temperatures of the pure materials.

The author has attempted to reduce this lack of accuracy, inadequate when a high level of details is required (like for scientific purposes) in ignition measurements through different statistical analysis of experimental data.

This study has generated a statistical approach to the investigation of dust ignition temperature with the definition of the SIT (Statistical Ignition Temperature) and the definition of a Probability Escalation Interval (PEI); within PEI ignition probability of dusts vary between 0 and 1 with respect to temperature.

The results obtained show that standard methods adopted worldwide could fail in determine the MITC of dusts and bring a new, more accurate methodology as a contribution on the definition of ignition concept of dusts. The statistical study could also apply to ignition criteria of the explosibility test proposed here, in order to give to SET procedures and results a more reliable statistical value.

The Speditive Explosibility Test has the form of a screening procedures for defining explosibility of dust samples, with the aim of avoiding time and cost-consuming standard procedures. The test consist in different procedures derived from standards (mainly UNI EN 50821 and UNI EN 13821) which ignition conditions are modified to be "as worst as possible", in order to avoid marginally explosive dusts to be classified as non explosible.

Tests were performed on different dusts samples (by chemical composition, nature, PSD) and results were validated through explosion test in the 20 L Siwek-sphere. The test classification shows, for the majority of the samples collected, good agreement with literature standard explosibility tests. The SET could be assumed as standard test for the explosibility determination of a dust.

A second experimental campaign was performed on non-traditional dusts coming from textile industries. All samples PSD was analyzed and 15 over 100 total were submitted to the SET with the aim to obtain more data on explosive behavior of those type of dusts.

Keywords: ignition probability escalation, dust explosibility screening, non-traditional dusts

Acknowledgments

The objective of this Thesis has matured along these three years of my PhD career.

During this time, I was lucky to be accompanied by people that appreciate my work and encourage my efforts.

I would like to thank especially Luca and Daniela for their precious scientific support and suggestions, but also for the grateful relationship we have cultivated as human beings.

The global acknowledgments goes to all my friends, familiars and each person has crossed my paths and revealed to be a positive encounter.

I hope , finally, that all this people thought the same about me.

Introduction

1.1   Generality on dust explosion

The term "explosion" is declined in dictionary in different acceptations:

- "a rapid increase in volume and release of energy in an extreme manner, usually with the generation of high temperatures and the release of gases";
- " A violent bursting as a result of internal pressure";
- "The loud, sharp sound made as a result of either of these actions".

As gas and dust explosions are concerned, these different meanings are both used, depending on context whether the causes or the consequences of the phenomena are analyzed.

While gas explosion is worldwide known as phenomenon and its hazards have been deeply investigated since early technological era, dust explosion study is still a challenge for those who are concerned with.

Thus, nevertheless the first documented dust explosion dated as old as 1795: Count of Morozzo (Northwest Italy) reconstructed a flourmill deflagration occurred in Turin on 14 December of that year. The most probable cause was indicated in the unaware act of a mill worker that enlightened the inside of one flour silo with a lamp; this caused the ignition and consequential explosion of the amount of dust lifted by the miller boy and his injuring.

Dust explosion study had made several steps from that moment on.

People experienced in everyday lifetime small and confined dust explosion (actually rapid combustion): the sawdust injection in stove to sustain its early combustion is a typical example.

Dusts are described as solid particles generated by handling, crushing, grinding, rapid impact, detonation, and decrepitation (breaking apart by heating) of organic or inorganic materials, such as rock, ore, metal, coal, wood, and grain. (Industrial Hygiene)

For many years, NFPA 68 defined combustible dust as a "finely divided solid material 420 microns or smaller in diameter (material passing a U.S. No. 40 Standard Sieve) that presents a fire or explosion hazard when dispersed and ignited in air. Other NFPA standards, however, have changed their combustible dust definition to remove the size criterion, but discuss size in their explanatory notes. In general, dusts generated from combustible material with a particle size of less than 420 microns could be defined as combustible dusts. However, certain particles, such as fibers, flakes, and agglomerations of smaller particles, may not pass a No. 40 sieve but still have a surface-area-to-volume ratio sufficient to pose a deflagration hazard. In the most recent revisions, the explanatory notes in many of the NFPA standards have moved from a 420 to 500 micron size threshold.

European regulations according to the EN 13327 set this threshold at 500 um.

Dust explosion could be defined as the rapid combustion of a combustible solid substance divided into small particles. As a combustion reaction, this requires an amount of energy to occur; reagents had to be at a certain energy level, called activation energy to be converted into products.

This gap to reach the activation energy in combustion reaction is fulfilled with the energy of an ignition source that is transferred to the reagents.

Dust and gas explosion has the "fire triangle" in common: both need an ignition, a combustible and an oxidant to occur. Dust explosion actually need two more components to happen: a certain confinement of the fuel involved and its adequate dispersion in air: here literature speaks about the "dust explosion pentagon" (OSHA).
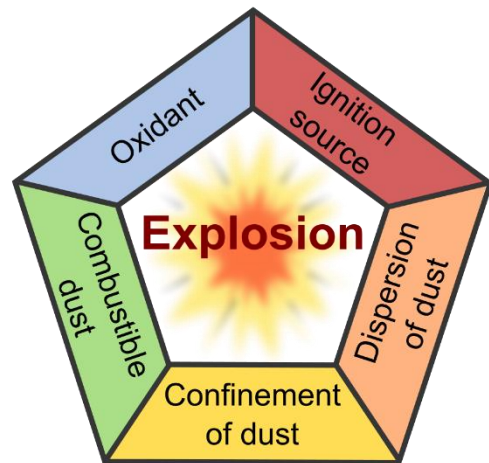
Figure 1: Dust explosion pentagon
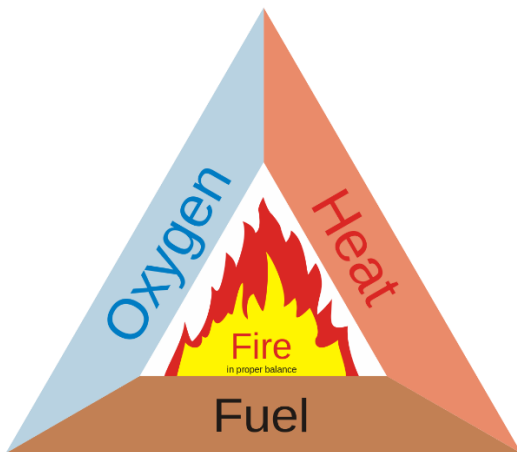
Figure 2: Fire triangle

The first aspect of the pentagon to take in account should be the possibility of a dust being combustible: this could be actually considered the generic aim of the screening test proposed here in this work. In these terms the words combustible, flammable and explosible are considered the same. Nevertheless in the followings explosible is referred to dust that result positive in the screening test and to indicate LEL and UEL (lower and upper explosibility limits).

Dust must be airborne and beforehand disperdible in air. This aspect deals with the so called dustiness, as defined in Klippel et al. (2015) work, as the capacity of a dust sample to be dispersed and stay airborne (definition in VDI 2263-3). Dustiness is relevant with respect to the correct procedure of standard test: e.g., only dust sample that could be efficiently injected in the furnace could be submitted to the MIT test.

Ignition sources that could be found in a working place are several, the most probable to find within a dust handling or processing plant are correlated to the processing apparatus. These could be represented by welding, cutting, heating elements. Furthermore, the generation of sparks by mechanical impacts, burning materials or embers could initiated an explosion, as well as electrical sources like electrostatic discharges or electric arcs. Each different type of ignition must have a specified experimental setup with which dust sample sensitivity to it could be examined.
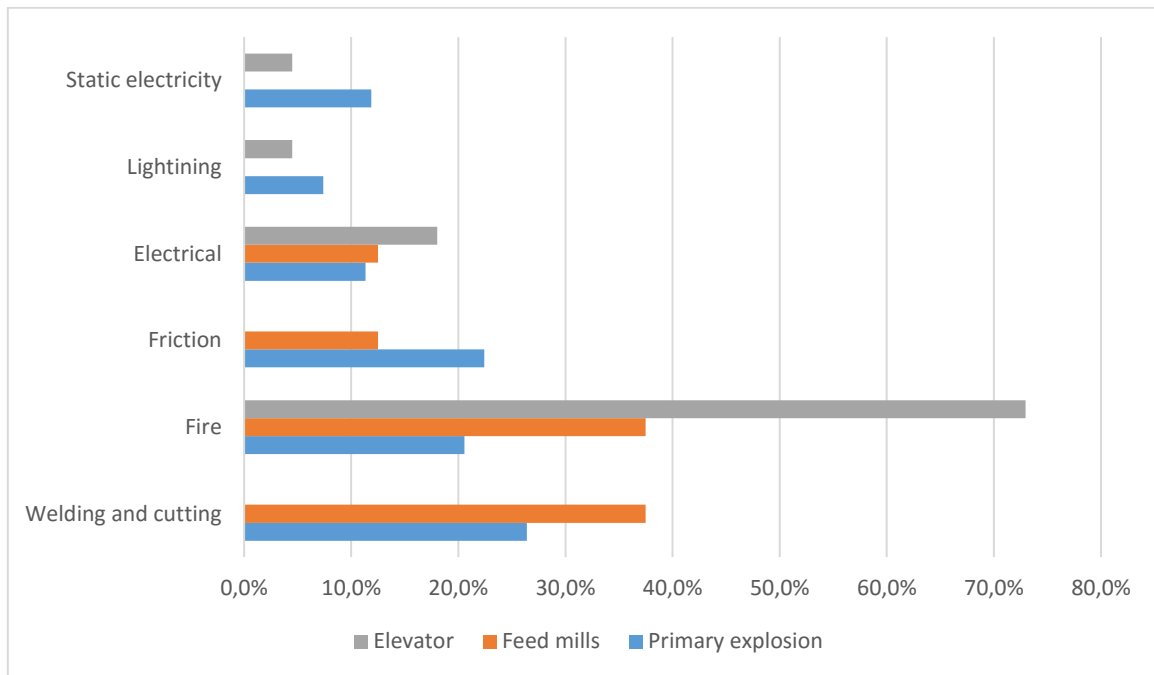
Figure 3: Major dust explosions ignition sources with respect to different industrial equipment, from Abbasi (2007).

1.2 Dust explosion influencing factors

Specific surface area is defined as the ratio between the surface and the volume of a single particle in the dust clouds. If as an example we consider spherical particle (traditional dusts could be mainly assumed as spheres) then we have:

$$S = \frac{\pi x^2}{\frac{\pi}{6}x^3}$$

(1)

Where x is diameter of spheres.

Therefore the specific surface area available for the combustion reaction will increase as the x linear dimension decrease; the specific surface being the site where oxygen and combustible mix and reaction take place.

In this context the smaller the dust particle are the higher the combustion rate, in general up to a certain limit value below whom any further increase in the specific surface has no significant influence on the reaction.

Aluminum dust particles could be at first instance not combustible at all, as aluminum is subjected to superficial oxidation at extremely low rate (if the specific area is reduced); actually when size of aluminum particles is reduced combustion rate increase quickly.

Figure 4: Schematic drawing that shows the influence of particle size with respect to combustion kinetics, from Echkoff (2003).

Specific surface area of dust particles is one of the variables that affect dust explosion sensitivity and violence; among them:

- Dust chemical composition and nature;
- Physical conditions of dust and gas phase before the explosion (temperature and pressure)
- Moisture content of dust
- Dust particles size distribution (PSD) and shape
- Dust particle distribution in dust cloud
- Degree of dispersion or agglomeration of dust particles
- Distribution of dust concentration in cloud
- Initial turbulence of dust cloud

All of these factors do contribute to the dust explosion process (causing and be consequences of it). Actually, dust chemical composition, physical conditions and particle size distribution could be considered as parameters proper of the dust cloud, while degree of dispersion or agglomeration and turbulence are associated more to the mechanism of dust generation in the actual industrial process.

Any solid material susceptible to react with an oxidant element and generate a relevant amount of heat in the evolution of the combustion could give birth to a dust explosion if conditions of the "pentagon" are satisfied.

Oxygen is generally the main oxidizer for such type of reactions: element already stables as oxides are therefore excluded in the list of those susceptible to rise an explosion (as example silicates, carbonates, sulphates ecc...).
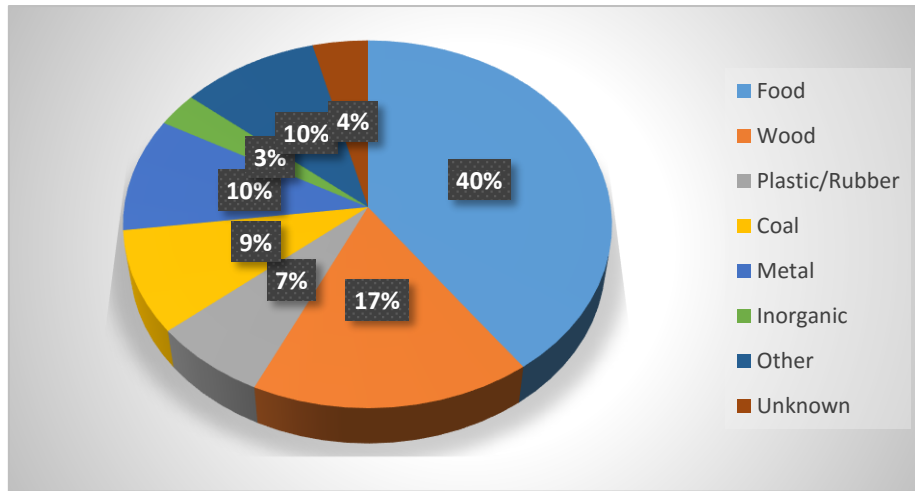
Figure 5: Occurrence of explosion dust according by industrial area during the period 1986-2004, from Abbasi (2007).

Mainly combustible dusts are divided in organic and inorganic materials.

The heat of combustion is therefore the main variable when the assessment of the explosibility of a dust is envisaged.

Great heat of combustion means great heat release during the reaction; Echkoff (2003) underlined that also the oxygen consumption is a key-factor and has to be considered together with the heat of combustion when a comparison between different combustible dusts is performed.

$$Combustible\ dust + Oxygen = Oxides + Heat \qquad (2)$$
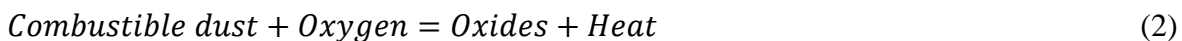
Inorganic elements follow the reaction scheme simplified of Eq. 1.1; the reaction products are the stabilized oxides of the element and the heat amount released.

While organic compounds follow a slight different scheme:

$$Organic dust + Oxygen = CO_2 + H_2O + Heat \qquad (3)$$
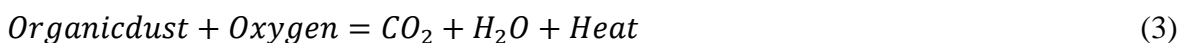
In this way, the number of moles generated in the reaction are greater and the pressure rise (i.e. the violence of the explosion) is reduced, according to the gas equation

$$p = \frac{TRv}{n} \qquad (4)$$

Where T and v are temperature and volume of gases and n is the number of moles in this volume.

Nitrogenous compounds are instead prone to liberate nitrogen in the reaction and thus is depleting the oxygen fraction available for the combustion, which is sometimes compromised or completely avoided (as example natural elements containing proteins or carbohydrates, azo dyes etc.)

In this study several dusts samples of different chemical composition are tested and their behavior to ignition and explosion potential is analyzed. In particular experimental campaign of flammability screening test is been performed with dust samples coming from the Italian industrial reality, spacing form metallic dusts to natural organic and synthetic organic ones.

It's worthy to underline that dust samples in industrial context are rarely pure elements: the great majority consists of mixture of different elements, depending on the type of manufacturing to which materials are subjected. This mixture could be also inert – combustible dust mixture if dust collecting is performed in a part of process where some thermal treatment has been done on it.

This study deals mainly with these types of samples, that are the more representative in the actual industry, but unfortunately the more difficult to examine, as scientific fundaments on dust mixture behavior are not yet well understood and several works are in progress with this aim in the academic and research context all-around the world.

1.3 Motivation of the Thesis work

Nowadays there's no standard conceived to simply determine if the dust can cause an explosion, i.e. a test questioning about the explosibility capacity of a dust sample that returns a yes/no result.

The work of this Thesis starts from here: how it is possible to characterize the explosibility of a dust sample without drawing upon standards specific methods?

In order to fully describe dust behavior a great number of parameters can be measured according to several technical standards. In general, each standard describes how to measure a single parameter, which characterize a specific dust behavior.

The main problematics rise from different aspects of the study:

- Standard methods are often expensive in terms of time and laboratory expenses;
- Dust that "could not explode" are not tested through standard methods;
- The definition of a test require a great data set to rely on.

Literature provided different approach to the explosibility determination; these varying one from another depending on the apparatus device used in the test or in the detail of the investigation and number of variables monitored.

A more detailed overview of all this methods is provide in Chapter 4.

The ongoing work of development of this type of test came mainly from the activity planned in the C.S.S.I.AT.Ex in Politecnico. Within it, dust explosible properties are tested through standard test methods. The laboratory offers entrepreneurs the possibility to examine whether the dust produced in their industrial operation is out of risk or not. The actual industrial characterization of dusts samples is different: as said before rarely dust samples could be found in form of pure element, but mainly mixtures of peculiar composition are present.

The chance to work with several dust samples coming from different industrial realities (and from different operational processes) let to found the reliability of the screening test procedure on a relative high-inclusive data set.

Moreover an intensive work has been performed with non-traditional dusts. These are dust samples coming from the textile industrial reality of Northern Italy. Among them 15 were tested with the screening test and 10 to 15 resulted explodible. The limitations related to non-traditional dusts applied also to the screening test; here limited dispersibility due to the fibrous nature of samples do not allow to perform the screening test on some of them. This campaign's results are described in chapter ### and reveals how the screening test has to be improved when non-traditional dusts are involved.

As what concern costs of standard methods, these are related to the great number of tests (meant as single injection of a specific dust concentration in any apparatus) and consequently in terms of test time duration. As an example for the MIT determination procedure described in the UNI EN 50281, at least 11 injections of the dust sample are required to assess the final value.

In the LIT procedure, problematics are related to the long execution of one single test: the confirmation or not of an ignition has to be investigated up to 30 minutes from the starting of the test. Again, no ignition tests are to be confirmed with a series of three test at the same temperature.

This need of reducing the time (and economics) costs of tests has to be compared to the need of performing an adequate number of tests in order to sustain the statistical reliability of the results.

Chapter 2 of this thesis deals with this problematics related to the determination of a "statistical" ignition temperature for dust sample, investigating the evolution of the probability of ignition in the temperature range between the "0%" ignition probability temperature and the 100%.

This existence of this probability range with respect to a certain variable (temperature, energy as examples) influences also the procedures of the SET and the definition of a number of attempts for each tests that could consider this probability evolution in its totality.

Actually the European standard EN 1127-2011 (Explosive atmospheres. Explosion prevention and protection. Basic concepts and methodology) defines main possible ignition sources for a dust explosion. This regulation could be usefully integrated with the possible implications of the results obtained by the SET and the implications related to the ignition probability definition on statistical basis.

Measurement of explosible parameters of dust

2.1   Preliminary phase and sampling of dusts

Laboratory tests constitute the first step for the hazard evaluation in working places where combustible powders are handled or produced.

An efficient investigation should start from the analysis of the working place where the dust to be tested is found: here the operator of the analysis should individuate the presence of areas where dust clouds could formed (in normal process operations but also in case of accidents).

Furthermore, it's relatively important to individuate on situ the presence of possible effective ignition sources nearby the areas where dust cloud could be found.

After this preliminary phase the evaluation starts from the collection of the samples of dust: these have to be the more representative as possible of the actual characterization of the possible dust cloud that could be ignited and explode.

The sampling phase plays an important role: even the more precise and accurate testing is misleading if the dust samples tested are not representative of the real dust characteristics found in situ.

Factors that could influencing dust ignition sensitivity like moisture content, size distribution and chemical composition (among others) should not be altered from the original sampled dust from industrial sites to the one tested in laboratory. The initial part of sampling is the collection of dusts from the section of the process plant in which is deposed: this could be part of a moving stream, contained in a conveyor bucket or cumulated in a bag or in a heap. Different procedures of sampling should be applied depending on the sites where the collection has to be done.

The second part of sampling is the sub-sampling practice performed in laboratory, down to the adequate amount of dust sample to perform the measurement (in some case down to 0.1 grams).

The attention here should be focused on the grade of segregation of the great amount of dust that could be found in sampling bags and compared to the poor representation of the dust sample that could be found in a little picked up amount from it. For instance, particle with higher density will fall in the lower part of the container, while lighter particles will stay in the surface.
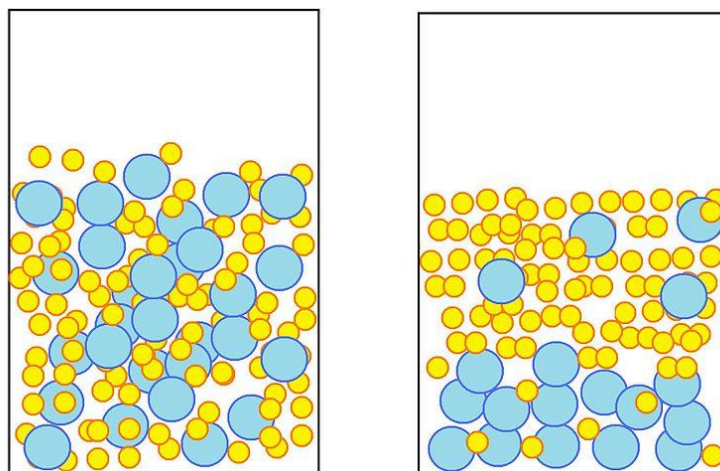
Figure 6: Schematic example of segregation of particles with different sizes in containers.

In laboratory the coning-quartering sampling method is adopted, this is described in the standard UNI 10802:2013.

Figure 7: Schematic representation of coning-quartering sampling method

Classification of hazards related to dust explosions falls in two categories: the "likelihood of an explosion" tests and the "consequence of explosion" tests.

The first are related to the sensitivity of the dust sample to be ignited and starts the evolution to the explosion. These tests should provide the adequate measures of prevention of the occurrence of a dust explosion, among them:

- Control measures on the existing ignition sources;
- Control on the oxidant supply to the combustion reaction;
- Limitation of the disperdibility of dust in air.

The second group defines the severity of an explosion and deals with mitigating and reduction measures:

- Venting, suppression, inerting procedure
- Prevention and housekeeping culture in working place.
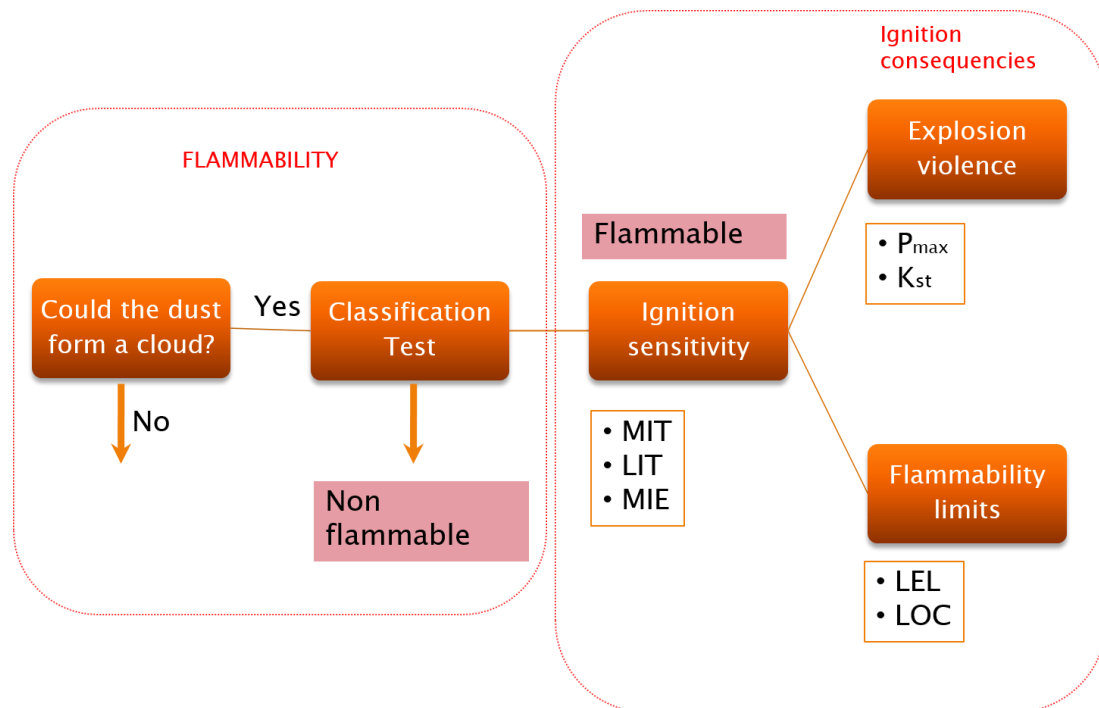
Figure 8: Example of possible proceeding of an explosibility dust screening test.

The parameters measurements related to the ignition sensitivity of dusts are:

- Minimum Ignition Temperature of a dust cloud (MIT);
- Minimum ignition temperature of a dust layer (LIT);
- Minimum Ignition Energy of a dust cloud (MIE);
- Minimum Explosible Concentration (MEC) or Lower Explosibility Limit (LEL);
- Limiting Oxidant Concentration (LOC);
- Electrostatic volume resistivity and chargeability of dust

In the followings, the main parameters with their measurement procedures are described, underlining the problematics related to laboratory scale measurement and the application limits depending on the type of dusts to be tested and the present literature in each field.

2.2 Minimum ignition temperature of a dust cloud

Any hot surfaces, with an energetic level overpassing a critical threshold, could be an efficient ignition source for a combustible dust cloud. In industry it's not rare to come across elements which surfaces are at a higher temperature with respect to ambient, in particular in sites where burners, dryers or furnaces are present.

In those situations if a dust cloud is generated in proximity of these hot surfaces a dust explosion could occur. The Minimum Ignition Temperature of the dust sample involved represents the threshold level that has not to be overpassed by any heated elements in the working place where combustible dust are generated.

Measurements of MIT could be assessed through a G-G Furnace (according to Dorsett, 1960) or with the BAM furnace (Leuschke, 1966).

The Godbert-Greenwald furnace consists of a cylindrical furnace with stainless steel external structure covered internally with silica and able to sustain temperature up to 1000° C. The internal diameter of the furnace is 36 mm. The heated silica tube is vertical and open to the atmosphere at its lower end. The upper end is connected to the dust holder by a glass adaptor. The dust is injected into the furnace by an air pulse obtained as the air contained into a reservoir is discharged by opening a solenoid valve. The device is mounted on a metal mirror plate enabling the operator to observe the interior of the furnace.

A pair of thermocouples is placed in contact with the interior part of the furnace; the first is linked to a PID temperature controller for furnace control, the other is intended for monitoring the internal temperature.

The BAM furnace is similar to the G-G one, but it is positioned horizontally with respect to the working desk and the injection of dust sample in the test chamber is manual, through a rubber bulb. Echkoff reported that differences in the MIT values of dust samples are found with the two experimental setup. While in fact, the G-G furnace shows a relevant dependence on the particle settling velocity and therefore on the residential time of it inside the furnace, the BAM furnace allows dust "weak to ignite" to settle and the ignition of smouldering gases from these particles could occur.

Hensel (1984) conducted a comparison study between the results of ignition in a modified G-G furnace with the length doubler than the standard and a BAM furnace with the automatic injection (instead of manually). The results reported that same as or smaller MIT were found with the BAM furnace (as an example Lycopodium dust MIT is 30°C lower).

More recently Wolanski (1992) has demonstrated that the ignition temperature of dusts could vary relevantly with scale. Many authors have dealt with the task of validating numerical models of temperature ignition of clouds of dusts with experimental data. Among others, Di Benedetto et al. (2015) proposed a model for the computation of minimum ignition temperature of polyethylene dust clouds. Addai et al. (2015) compared experimental data obtained from the ignition temperature of hybrid mixture (combustible dust and gas) in a modified G-G furnace with seven mathematical models for MIT prediction.

Another theoretical model was proposed by Chunmiao et al. (2012) for the determination of minimum ignition temperature of a magnesium powder cloud under conditions simulating a Godbert–Greenwald (GG) furnace.

Yuan et al. (2014) in their work, adopted the BAM furnace with a slight modification to assess ignition temperatures of Titanium nanopowders.

Gummer and Lunn (2003) tested the ignition sensitivity of dust clouds in the temperature range around MIT to smouldering nests of the same dusts, in a vertical tube apparatus.

In the present work MIT is measured by means of a Godbert Greenwald (G-G) furnace (Chilworth Technologies, model MITC-L, serial number J303161B). Measurement procedure was developed according to CEI EN 50281-2-1:1999 as far as air sample injection overpressure and sample quantity are considered.

According to CEI EN 50281-2-1 several runs were made at decreasing temperature ($\Delta T = 20$ K above 300 °C and 10 °K below) in order to assess the temperature range (T_1; T_2) as described above. The air pressure in the dispersion reservoir was set at 1.1 bar. A 0.1 g sample of dust or mixture was blown into the furnace.

The standard ignition criteria is the presence of a visible flame coming out of the furnace bottom. The presence of sparks in absence of flames is not considered proof of ignition. The T_1 is accounted when no ignition occurs over 10 consecutive test. At this temperature, further investigations was made varying both the injection pressure and the sample weight (varying values of pressure release between 0.02 and 0.5 bar and sample weight (ranges from 0.01 to 1 g). In case of ignition the temperature is lowered by 20 °K and 10 test runs are repeated.

The procedure described above to assess ignition temperature of dust clouds returns T_2 as the MIT_C value. The real MIT_C falls in the 20° C interval between T_2 and T_1.

Figure 9: Evidence of ignition in the G-G furnace test.

The standard methods, described above, defines the MIT value with an accuracy that stands between this 20°C temperature range. If a more detailed analysis is required, another procedure has to be adopted. The reliability of this procedure is call into question in this Thesis.

The applicability limit of the G-G furnace is evident when dealing with non traditional dusts. These flocks-like or plate-shaped particles present usually very low tendency to disperse in air and generate clouds. The study with textile waste dusts, described in Chapter 5 clearly underlined this problematics In particular the injection apparatus of the G-G furnace, air blow from reservoir operated by a solenoid valve, result inefficient to disperse these samples into the furnace.

2.3   Minimum ignition temperature of a dust layer

Smoldering layers of combustible dusts, even if do not represent the proper primary dust explosion fuel themselves, could be typical initiator of a secondary dust explosion. The smoldering combustion igniter could be a metallic piece overheated by impact or a hot ember coming from another combustion process; the glowing fire on the dust layers is generally a slow process and could burn unbothered until it reaches its thermal peak with consequent ignition of the dust cloud coming in contact.

The critical threshold in this case it is represented by the Layer Minimum Ignition Temperature (or LIT according to UNI EN 50281-2-1:1999).

The (MIT$_L$) test in this study was performed with a hot plate apparatus as described in the standard procedures. The apparatus is made up of an aluminum plate over which the dust layer is placed and an electric resistance (1200 W) that allows to heat up the plate at the test set point. A sample holder made of a metallic ring enables to shape a 5 mm dust layer on the hot plate. Alternative holders allow to settle 12.5 and 25 mm height samples. A thermocouple is placed to monitor the temperature in the middle of the dust layer. A PID controller interfaced with a second thermocouple located inside the plate controls the plate temperature.

The dust layer is dropped on the hot plate once set point temperature (T$_{SP}$) was reached. Ignition criteria was assumed in accordance with CEI EN 50281-2-1 as the hot plate temperature that provoked one of the following conditions: visible glowing or flaming; a temperature of 450°C in the dust layer; a temperature rise of above 250 K respect to the set for the hot plate. If no ignition occurred within 30 minutes, the run was stopped. In case of ignition, the test proceeded until the sample temperature diminished below the plate temperature.

According to CEI EN 50281-2-1 measurement procedure for the MIT$_L$, several runs were made at decreasing temperature $\Delta T = 20$ K each step in order to assess the temperature range (T$_1$; T$_2$), where T1 is the lower temperature at which no ignition occurred and T$_2$ is the upper temperature at which at least one ignition occurred. Further measurement at (T$_1$+T$_2$)/2 was performed to assess the minimum temperature at which an ignition occurred.

The ignition criteria for the ASTM E2021 is slightly different from the UNI EN standard: glowing, flaming and a temperature rise above 50°C are considered proof of combustion in process in the dust layer examined.

Furthermore it has found a slight decrease of the LIT of dust mixture, when an amount of inert material up to 20% is added to the combustible dust, thus in counter tendency with literature data.

Moreover, dust sample composed of materials that could undergo to melting process could not be analyzed efficiently with this procedure.

Many studies are present in literature dealing with minimum ignition temperature assessment of dust layers. Bideau et al. (2010) build up a model simulating the distribution temperature and evolution in time in the combustion of inert-combustible mixture dust layers.

On the other hand Krause and Schmidt (2000) have inveditgate the propagation of smouldering in dust deposits through the adoption of cylindrical mesh wire baskets where the dust was layered. Recently Ajrash et al. (2016) focused their investigation on the influence of PSD and moisture content on the auto ignition temperature of coal dust layer, in hot and humid ambient.

Figure 10: Evidence of ignition in the hot plate standard test for LIT, glowing combustion within the dust layer sample.

2.4   Minimum ignition energy of a dust cloud

Electric sparks and arc generated from short circuit of electrical engines or damaged cables could trigger a dust cloud explosion. Electrical arc generated by electrostatic energy could also develop an efficient explosion igniter for some type of dust.

The first experimental apparatus devoted to measure the hazards related to electrical arc in contact with clouds of combustible dusts was developed by Dorsett et al. (1960).

The standard apparatus is described in the UNI EN 13821:2004 and in the present work is carried out by an Hartmann tube of 1.2 l volume.

Hartmann tube is plastic hollow cylinder with a 9 cm internal diameter and 34.5 cm high.

The ignition source is provided by a couple of Titanium electrodes placed within the tube at 11.5 cm high from the basement. Electrodes are placed horizontally and with a 6 mm gap between each other.

The electrodes are included in a electric circuit that provide a capacitive discharge between them through a series of capacitors with different energy stored. The original apparatus was developed by Dorsett et al. (1960), actual setup differs from it for the discharge circuit that has been provided by an inductance; the induction coil makes the spark more effective as an ignition source by increasing the discharge duration of the spark. Dust dispersion mechanism remained the same: an air blow activated by a solenoid valve injects the dust sample into the tube (7.0 bar pressure).

The more recent standard for the minimum ignition energy measurement procedures is the UNI 13821:2004. According to this standard (and the correspondent ASTM E 2019-03) electric circuits for the spark generation could be of different kind.
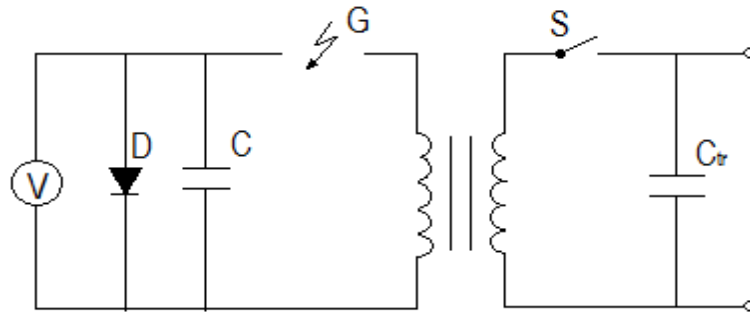
Figure 11: Transformer-generated spark circuit

In Figure 11 C is the discharge condenser, having initial tense between its walls equal to U. Theoretical values assumed by energy generated from the circuit are equal to $^1/_2\,CU^2$.

EN13821 declines the characteristic of the circuit:

- Discharge circuit inductance has to be between 1 and 2 mH;
- Discharge circuit ohmic resistance has to be smaller than 5 Ω;
- Electrodes has to be made of Tungsten, Alloy INOX, Copper or brass
- Electrode peak has to be 1.5 to 2.5 mm large;
- Electrodes must be adequately insulated to avoid stray currents.

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It will be necessary to take account of the possible influences of dust concentration, dust cloud turbulence, and degree of dust dispersion on the test result. Preliminary tests must be carried out to adjust the dust dispersion conditions and the ignition delay, so that prescribed minimum ignition energies are actually measured for three specified reference dusts. Starting with a value of spark energy that reliably causes ignition of a given concentration of the dust being tested, the dust concentration being itself a variable, the test energy is successively halved until no ignition occurs in 10 successive tests.

The minimum ignition energy is defined to lie between the highest energy at which ignition fails to occur in at least ten successive attempts to ignite the dust-air mixture and the lowest energy at which ignition occurs within 10 successive attempts.

When the ignition energy of non traditional dusts has to be measured the Hartmann tube dispersion setup could not be efficient to generate a well-dispersed dust cloud and to disaggregate the flocks typical of this type of samples. In this case, as in the G-G setup for the MITC measurement, an alternative dispersion unit has to be developed for adequate testing.

Many studies on the MIE of dusts and on variables influencing it have been carried out worldwide. Recently new measurement methods were proposed by Echkoff and Randeberg (2007) dealing with electrostatic discharge impact on sensible dusts (with MIE < 1 mJ) and Choi et al. (2001) with the introduction of an apparatus for the MIE testing employing a vibrating mesh to generate the dust cloud.

Janes et al. (2008) compared the results of MIE measured both by Hartmann tube apparatus and MIKE 3 apparatus.

A statistical method was proposed by Bernard et al. (2010) for the estimation of MIE; some hypothesis of this model have been adopted in this work.

2.5 Minimum Explosibility Concentration (MEC), Lower Explosibility Limit (LEL)

Similarly to combustible gas, combustible dusts have a lower concentration in volume in air at which the occurrence of an explosion could be considered reasonably impossible. The difference with respect to gas, is that while it's simple to define gas concentration in a confined room, it's not so easy to measure it as what concern an airborne of dust disperse inside a closed space. Actually industrial equipment handling dusts often deal with great amount of it and a dilution in air is not practice. Nevertheless, this operation is simpler to carry out for some apparatus: the typical example being the dust extractor system mounted on machineries that generate fine materials in their operations. The MEC is respected in the extraction system once the air velocity in the ducts is sufficient to obtain a dust concentration inside it below this threshold.

MEC is first measured by Dorsett et al. (1960) of US Mines Bureau in a 1.2 L cylindrical container where the dust to test was dispersed and the ignition source was provided by an electric continuous spark. The procedure starts with low amount of dusts, then increasing it until an ignition occurs.

This method was influenced by the dispersion and ignition mechanism: the first at the bottom of the tube do not let the cloud to developed before the ignition, thus resulting in an underestimation of the MEC, the second was not very effective as ignition source, resulting in an overestimation of MEC.

Siwek vessel (Siwek, 1985) was adopted for P_{max} and k_{st} measuring, as well as for the MEC. In this case, however, depending on the excessive energy release with respect to the volume of the sphere in which was released, lower than expected MEC values are found. This phenomenon is defined as the overdriving of the explosion (discussed also for the k_{st} measurement in the SET results of Chapter 4) and could be avoided within the 1 m^3 chamber, due to its larger volume, the ignition source do not interfere with the first phase of the dust cloud propagation.

The 20 l sphere method is described in standard UNI EN 14034-3:2011 and adopted internationally due to its smaller scale, more feasible in laboratory setups. The explosion criterion here is a value of maximum explosion pressure equal to at least 1.5 bar (g). This includes the pressure of 1.1 bar (g) generated by the chemical 10 kJ ignitor only, without dust. Tests are conducted with successively decreasing dispersed dust masses in steps of 0.2 g until a mass is reached at which the maximum pressure is lower than 1.5 bar(g) in three consecutive tests with the same dispersed dust mass. The minimum explosible concentration is then assumed to lie between the highest nominal concentration (dispersed mass divided by vessel volume) at which the maximum explosion pressure was less than 1.5 bar(g) in three successive tests and the lowest nominal concentration at which the explosion pressure was 1.5 bar(g) or more in one of up to three successive tests.

Among other works, Chatrathi and Cashdollar (1992) have compared MEC values obtained from 20 L Sphere and 1 m^3 chamber.

The study of Parnell et al. (2013) made a critical evaluation of testing methods, including MEC measurement with a comparison of tests performed on gin dusts with 10 both kJ chemical igniters and stationary coil as ignition sources. Amyotte et al. (2014) used MEC values as parameter for the implementation of a model assessing the dust explosion occurrence probability.

LEL measurements of hybrid mixture (combustible dust and gases) were obtained by Garcia Agreda et al. (2011) in a 20L Siwek sphere adapted for such type of materials.
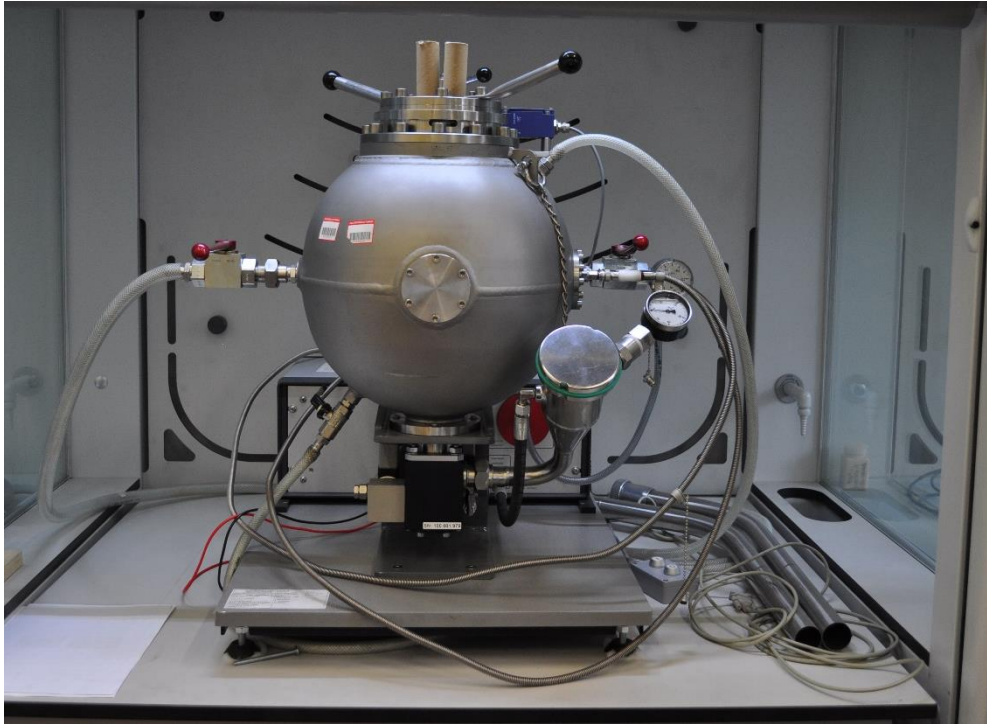
Figure 12: Siwek 20 L sphere of the C.S.S.I.AT.Ex, in Turin

2.6   Limiting Oxygen Concentration (LOC)

The LOC indicates the minimum oxygen percentage in volume in air at which a dust explosion is yet possible. Below this threshold the amount of oxidant is inadequate to sustain a combustion process and therefore to develop an explosion.

LOC is measured according to UNI EN 14034-4 and tests are performed in the 20 L Siwek sphere.

Generally an inert gas (nitrogen mostly) is added to air to defines several mixture with different oxygen content. The procedure to follow is describe below; a couple of chemical igniters of 1 kJ has to be used in the tests.

By increasing gradually the ratio of inert gas to air and varying the dust concentration, the oxygen concentration shall be reduced to a level, at which explosions no longer occur for any dust concentration. An ignition of the dust (dust explosion) shall be considered to have taken place, when the measured overpressure (influence of chemical igniters included) relative to the initial pressure p_i is $\geq 0,5$ bar [$p_{ex} \geq (p_i+0,5$ bar)].

The highest oxygen concentration where no ignition occurs in three consecutive tests shall to be reported as the limiting oxygen concentration LOC.

LOC determination could apply to the safety protection measurement of substitution or partial inerting, depending on the type of dusts and inert gas considered. The possibility of dust explosions in process equipment can, in principle, be effectively eliminated by substituting the air by a gas that prevents flame propagation in the dust cloud. Thus could rise to economic problematics, since large amount of inert gases in a plant could weigh greatly on the process costs.

For most dusts, it is not necessary to substitute the entire atmosphere in the actual area by, for example, nitrogen, carbon dioxide, or other inert gas to obtain inerting.

In some cases, it may even be of interest to use smaller fractions of inert gas than required for completing inerting, because this reduces both the ignition sensitivity of the dust cloud and the maximum pressure and rate of pressure rise at constant volume. Metal dusts, like aluminum and magnesium, exothermic reactions with C02 could occur and the use of rare gases may have to be considered in certain cases.

In practice, several experiments were made on the influence of inerting gases on ignition sensitivity and explosion mitigation of combustible dusts.

Echkoff (2003) reported some results obtained by other authors in their research, mainly:

- explosion pressure and the rate of pressure rise decreased with decreasing oxygen content and the reduction of the maximum pressure is approximately proportional to the reduction of the oxygen content, (Wiemann, 1984);
- As particle size increased, the influence of reducing the oxygen content became pronounced (Hertzberg and Cashdollar, 1987)

More recently significant results were found by Wilen (1998) and Schwenzfeuer (2001). The first found an increase in the LOC of biomass dust, when the initial pressure is increased. The second found that that LOC for ignition of dust clouds by electrostatic discharges were significantly higher than the conservative limit determined in standard tests, using a very strong pyro- technical ignition source. On the other hand, Walther and Schacke (1986) reported that the LOC of clouds of a polymer powder was independent of the initial cloud pressure over the range 1–4 bar (abs.).

Echkoff (2004) experiments showed that the maximum rate of pressure rise decreased approximately linearly with decreasing oxygen content, down to zero at 11 vol.% oxygen, the limiting oxygen concentration (LOC) for sustained flame propagation through the dust cloud.

2.6   Explosion violence parameters

The parameters that evaluate the consequences of a dust explosion are defined as explosion violence parameters and are:

- P_{max} maximum explosion pressure (bar)
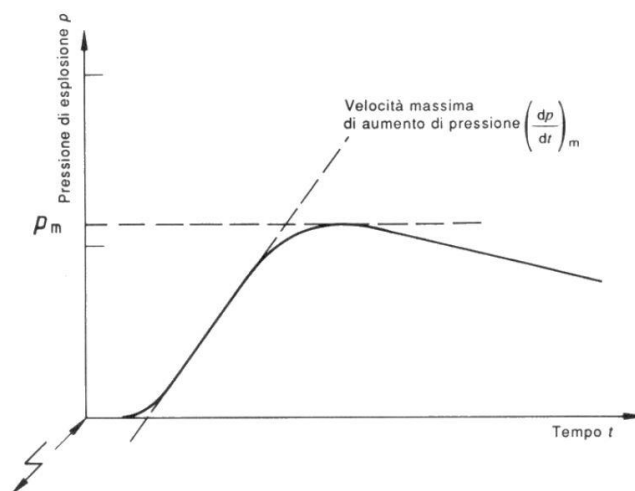- $(dP/dt)_{max}$ maximum rate of pressure rise (bar/s)

Figure 13: Typical pressure.time curve of a dust explosion, the pressure peak and the maximum rate of pressure rise with time are indicated.

The procedure to obtain those value is the standard UNI EN 14034-1 and the equipment in which the test is performed is the Siwek 20 L sphere, as in the case of MEC, LEL and LOC determination.

The procedure of test consider different dust ignition attempts at different dust concentrations. For this type of test only the more powerful 10 kJ igniters are used.

From the pressure-time curve obtained , the value of kst is derived.

This index report the rate of pressure rise to a 1 m3 reference volume, through the following correlationship propsed by Bartknecht (1971, 1978):

$$K_{st} = \left(\frac{dP}{dt}\right)_{max} * \sqrt[3]{V_{vessel}} \tag{5}$$

Where Vvessel is the volume of vessel test where the dust is ignited.

Values of kst classified the dusts according to their explosion severity in classes, reported in the table below.

Kst values [bar·m·s^{-1}]	Explosibility St Class
0	St0
K_{st} < 200	St1
200 < K_{st} < 300	St2
K_{st} > 300	St3

Kst and pmax values have specific meanings in the building of explosion mitigation equipment in process plants handling combustible dusts.

The investigation of those parameters has been intensively performed worldwide and since the very beginning of dust explosion research.

The general theory of explosion parameters is well described in the works of Cashdollar (2000) which gave an overview of dust explosion characterization, Echkoff and Amyotte (2010) dealing with explosion causation and mitigation and the recent book by Amyotte (2013) that gave a complete introduction of the dust explosion research.

Recent studies have focused on the occurrence of the overdriving phenomenon (described in detail later, in Chapter 4): among them the studies of Clooney et al. (2013) and of Myers et al. (2013).

2.7   Tests at Temperature and Pressure different from ambient conditions

Echkoff (2003) reported results obtained by Glarner (1983) and Wiemann (1987) on explosion tests performed at initial temperature higher than ambient.

They found decreasing MEC with the increasing of the initial temperature, below to a certain value close to 0, when the data seemed to converge to a asymptotic value of temperature. From the data collected by the two authors this convergence point is found at around 300-500°C. This is relative low with respect to the same temperature value found for hydrocarbon gases (1200°C). They justify

this behavior with the more complex physics and chemistry dealing with organic dusts with respect to gases.

Glarner (1984) studied the influence of the initial temperature on the dust minimum ignition energy generated from an electric spark. Also in this case a convergence appeared at 0.088 mJ and 1000°C.

On the other hand explosion parameters k_{st} and P_{max} are influenced also by the initial temperature.

Maximum pressure rise tends to decrease with the increasing temperature due to the reduction oxygen content being the pressure at a constant level, while temperature increase. The trend of the rate of pressure rise with respect to initial temperature is more complex and could not be explained by a linear relationship.

Wiemann (1987) studied also the influence of the increasing initial pressure on explosion parameters. Clearly the increasing of pressure resulted in increasing pressure peaks in the explosions, with a proportional trend with the initial value. Wiemann's experiments found a linear correlation between initial pressure and rate of pressure rise, even if with initial pressure greater than 2 bar the correlation level out.

The most recent effort in this investigation is due to Gribson and Rogers (1980) with their study on ignition of dust clouds in hot environments.

More experimentations are needed also in this context, in particular aiming to the development of practical equipment to generate initial temperature or pressure different from ambient in safety conditions.

The dust ignition concept

3.1   Literature context

The original motivation in the early years of dust explosion research to develop standard tests was simply to determine in the more adequate manner some specific parameter useful to design appropriate protection measures for plants that handled combustible dust.

In particular, a test was developed to measure explosion violence parameters (k_{st} and P_{max}) in order to obtain data on the venting requirements for plant apparatus in which an explosion, and then a pressure rise over design value, was likely to occur. Later, data collected with these standards, were adopted as physical constants of the specific dust without further investigation and misunderstanding their original scope.

More recently research has pointed out the diversification of the parameters and the need of arbitrary tests related to different parameters. The scenario, which researchers have to face currently, is complex.

Only a few parameters could be considered physical or chemical constants of the specific examined dust sample.

Keywords for an efficient and practice approach to the measurement of dust ignitability and explosibility parameters has to be:

- differentiation;
- resolution;
- applicability.

As Echkoff reported (2003) one first approach to the differentiation is to arrange several tests for every different parameter in order to define the more appropriate for different practical issues.

If this approach is adopted, two parallel operations are possible. The example relates to the ignition sensitivity of dust to electric sources.

From one hand experimental tests could be performed, in which different kind of electric discharge (and of different energy) are used as ignition sources, to test the sensitivity of dust sample to every one of them. On the other hand a detailed in situ survey is necessary to evaluate the actual ignition sources present in the industrial context on which dust samples are located and in order to discuss the ignition test results with them. For example in the case of the ignition energy measurement the introduction of an inductive component in the discharge circuit, resulting in more incendive sparks, could be used to increase the ignition effectiveness. If, however, electrostaticity hazards are to be proved, this arrangement would not be useful, because discharge of lower energy order are examined. Glor (2003) investigated furtherly the difference between the effectiveness of electric hazards to ignite a combustible dust and concluding that more work is needed to cover all cases related to electrostatic phenomena and a standard solution is yet to be found.

An alternative approach to diversification is using the measured parameters as input for calculations in suitable theories in order to simulate the best as possible actual industrial conditions. From k_{st} values, laminar cloud combustion velocity could be found and the cloud combustion could be simulated with fluid dynamics models, thus in practice increasing the complexity of the task.

Resolution focused on the scale of the measurement test. Real industrial situations could be simulated through a full-scale setup test in which dust could be introduced and occurrence of ignition registered. This is obviously not practical to build up with respect to small-case laboratory equipment. Investigation should focus on what level of resolution could be the more suitable for the parameter to be measured.

As Eckhoff reported (2015) effective difference in dust explosion violence has been observed whether the test was performed in the 20 l Siwek sphere or in experimental 500 m3 silo. This was because these parameters (k_{st} and P_{max}) are not influenced only by inherent dust parameter (like moisture content, size of particles) but also by parameters related to the specific process in which the dust is generated. Among those, the dust dispersion system and the degree of turbulence of the dust cloud and its concentration, are relevant and have to be considered when tests with different scale resolution are performed.

Another point to be examined is the applicability of laboratory test procedures suitable for all type of dusts susceptible to combustion in the actual industrial reality.

Standard tests in use for dust explosibility parameters present by their same nature some limitations with respect to certain type of dust samples. As underlined in Chapter 5, these measurement procedures are hardly suitable for all non-traditional dusts (for example flock-like aggregate coming from textile industry) because of their poor disperdibility in air. This condition itself should grant that such type of dust are rarely capable of generate an explosive atmosphere in air; however other laboratory test, with more energetic ignition sources, or with more effective mechanism of dust dispersion, have proved the explosive nature of these samples. This is reported by Amyotte et al. (2011) and detailed again in Chapter 5.

Recently some revision on standard measurement test have been done, aiming to go towards more differentiated and consequently efficient procedures.

Nifuku and al. (2001) have developed an ignition test method in which a sieving tap is used to create the dust cloud over a Hartmann tube to investigate the Minimum Explosible Concentration.

Dahn and Dastidar (2007) proposed a test to determine the threat posed by potential incendive electrostatic discharge from fiberglass reinforced plastic pipes.

In the meantime data collected by the BIA was published in Beck (1997), grouping combustible dusts in different classes according to their chemical nature, reporting for each element (where measured) LEL, MIE, St Classification, LOC, MIT (cloud and layer) and BZ combustibility class.

Table 1: Recent tests developed for dust explosive properties assessment.

Parameter	Equipment	Proposer, developer (date)
Dustiness	Rotating drum tester	Breum, 1999
Ignition Energy	Circuit for lower than 1 mJ spark generation	Siwek & Cesana (2014), Eckhoff & Randenberg (2007)
Ignition Energy	Movable electrode, energy down to 1μJ	Zhou, Tan & Yu (1994)
Ignition Energy	Vibrating mesh to generate dust cloud	Choi et al. (2001)
Dispersibility	Measuring powder flowability	Jong et al. (1999)
Dustiness	Standard methods	VDI 2263-9
LOC	New test method	ASTM E 2931, Dastidar ()
K_{st} and Pmax	Exploding wire in 20 l sphere	Scheid et al. (2013)

Finally, if an investigation of ignitability of a dust is planned, figure ### (reported from Eckhoff, 2003) is relevant.
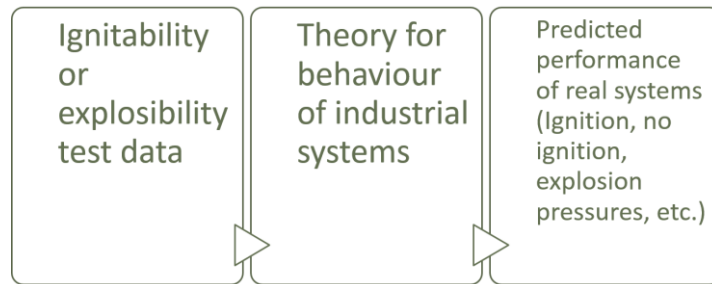
Figure 14: From experimental data to real systems, derived from Echkoff (2003).

The left side of the scheme stands for testing procedures, while predicted results are present in the right side. If a full scale testing campaign is planned, then what results from the first box is the same in the right-side box. However if laboratory scale tests are adopted, some correlations through theory and suitable models is required to transform test results to actual data for real system performance.

The main questions are:

- Which level of quality is obtained from the actual available measurement techniques?
- Is there a suitable theory beside each of them?
- Which level of resolution is the optimal one?

These questions should been applied every time an investigation on a dust explosion parameter is started, thus depending on the type of dusts, on the industrial site and setup where the dust is handled.

3.2 The concept of the Probability Escalation Interval (PEI)

The attempt to answer to the previous questions could start from the identification of a demarcation line between the evaluation of the explosion hazard concerning a dust and the quantification of the explosion risks associated to it, once hazard is demonstrated to exist.

In fact, while ignition sensitivity measurements could deal with the identification of hazards, the quantification of explosion parameters (mainly kst and pmax) is more a matter of risk estimation.

This work will focus primarily on the determination of the explosion hazard correlated to a specific dust sample; clearly this is related with the ignition sensitivity of the material to a certain external stress application (an independent variable) in terms of transferred energy.

The more the dust sample is "sensible" to this stress the higher the probability of an explosion to occur.

Consequently two further questions emerged:

- How could we correlate explosion probability of a dust to the intensity of the stress applied to it?
- What is the lower value of this stress at which we are sure (statistically based) to avoid an explosion?

Several tests (presented in Chapter 1) has been defined by technical and scientific literature to determine the dust explosibility parameters to which technicians can refer to assess the characteristics of a dust sample. Some parameters describe a countable variable, which is defined by a numerical value (e.g. P_{max}, k_{st}) while others define a threshold above (or below) which the explosion should not occur (MIE, LOC, autoignition temperature). These latter should be regarded as a limit at which the probability of occurrence of ignition (or explosion propagation in case LOC or LEL are considered) is low enough to be considered negligible.

This is the main point of our investigation and is reported in the two previous questions, to which this Thesis will try to answer.

Technical standards indicate the procedure to be followed to measure the different parameters. The Cloud Autoignition Temperature (AIT), according to the European standard CEI EN 50821 is the temperature at which 10 consecutive ignition attempts in a defined apparatus result negative (no ignition). Similar assumption is made by UNI 13821 as MIE is considered.

This work will focus on the temperature as independent variable influencing the ignition probability of dusts.

As MIT is concerned the ignition probability at minimum ignition temperature is supposed to be zero, but to date this has to be considered just as an assumption.

Effectively the relationship between the probability of occurrence of the studied phenomenon (ignition for example) and the independent variable (the temperature, in the case of AIT) is not yet well established.

MIT is determined in order to assess what is a "safe" condition respect to what condition should be considered "unsafe", being "safe" and "unsafe" defined as those conditions at which ignition "should not occur" or "could occur" respectively, assumed that ignition is an undesired event. If autoignition were a deterministic event with respect to T, a well-defined boundary temperature (say T*) could be found such that the ignition probability would be 0 at T<T* and 1 at T>T*. Process safety management would take great advantage of this phenomenon as ignition could be assumes as

impossible at T<T*. If this were the case, the same conclusion could be drawn as the spark ignition were considered.

It is well known that real systems behave in a very different manner as there is a temperature (or energy, in case of spark ignition) interval where ignition is not a deterministic event but rather a probabilistic event.

Literature data (Marmo et. Al. 2011, Bernard et al., 2010) demonstrate that a temperature interval exists over which the ignition probability varies from almost 0 to almost 1 continuously.

This range is defined here as the "probability escalation interval" (PEI). For many different dusts PEI seems to span about 30 °C. Conceptually the boundary between "safe" and "unsafe" as stated above should then be assumed as the temperature at which the ignition probability is low enough to be considered negligible. From an idealized point of view, this boundary should be intended as the MIT (or MIE if spark ignition is considered). In terms of risk analysis, the limit for an undesired event probability to occur depends first on the magnitude of the consequences. By considering the ignition as the starting event (or root cause) of a chain of events leading to the explosion, an order of magnitude of the acceptable probability p^*_{acc} could be assumed as 10^{-3}.

The PEI definition would take central position both in the determination of the explosibility of a dust sample, than in the determination of the minimum temperature at which the dust could ignite.

Starting from this concept this Thesis include the development of an explosibility screening dust test, named SET (Speditive Explosibility Test), as a tool to define the explosion hazard related to a dust, with a certain level of confidence; this being related again to the ignition probability concept described above.

Experimental data presented here can indeed be used to identify the threshold below which the ignition probability is lower than a suitably predefined level with prescribed confidence, providing a more solid ground on which the definition of the AIT could be based

Statistical models had been applied on this experimental database with the twofold aim to:

- investigate mathematical relationship between MIT_C and the temperature field in its neighborhood
- evaluate the reliability of the MIT_C value determined by the procedure defined by technical standards and to propose a new definition of the AIT, based on statistically sound methodology

Three different statistical approaches and definitions of Ignition Temperature are experimented in the attempt to increase the accuracy and reliability of this value, especially required when dust mixture are involved and more defined data are needed for scientific purposes.

The first definition of SIT derives from UNI EN 13821 definition of MIE; the second bases on a normal law distribution while the last bases on Generalized Linear Models. This last approach has proved to be the more reliable for our work and authors will adopt its definition of SIT in future works.

3.2.1 Ignition of combustible-inert dust mixtures: state of the art

A brief section is presented here with the purpose of collect all main literature findings in the field.

The study of dust explosions has to deal with the influence of inerts or impurities included in the combustible dust bulk. Indeed industrial products of several processes are frequently encountered as powders, in many cases in the form of mixture among different elements.

In the pharmaceutical production actives principles are intimately added with excipients or coating materials; in food industry grains are always mixtures of different cereals and the same could be said about sugar powders; in chemical factories paintings are often mixture of pigments and also the waste recycling industry treats mixtures of powdered materials (for example oxidized and non-oxidized metals mixed together).

A dust mixture with an inert could be defined in different ways, the "mixing" depending on samples' chemical composition (i.e. a combustible dust and a sand), but also on dust size distribution. Thus is the case of a sample of dust with very large size distribution (from 10^2 um to 10^0 um) that could be considered a mixture between two different materials with sensitive difference in ignition sensitivity (the higher the smaller are dust particles).

In the process industry quite often dust is processed in non-homogenous conditions, with a variable content of humidity and impurities related to process evolution. All these parameters contribute to rise (or decrease) the ignition sensitivity of the dust mixture as well as the explosion severity. Nevertheless several studies on the likelihood of explosion occurrence with combustible dusts has been made (and comparison with different ignition sources). However only recently studies on combustible dust mixtures (with inert or another combustible element) have been carried on (Dufaud et al. 2012, Marmo and Luzzi 2009, Reddy, Amyotte and Pegg, 1998, Amyotte 2006, Hamad and Qubbaj 1998) and literature data are relatively scarce with respect to pure combustible dust investigations.

The partial inerting technique aims to reduce the potential of the explosion in term of heat release. It differs from a complete inerting process where the amount of inert is considerably greater and no explosion occurs at all. The amount of inert is sufficient to mitigate the ignition sensitivity of dusts samples, influencing their MIT or MIE, or effects on their explosion parameters, like k_{st}(explosion violence index).

Echkoff (2004) states that partial inerting offers an additional degree of freedom in dust explosion protection: that is the possibility to, have the effect of explosion or even the occurrence of it reduced by it.

Additional protection systems (like venting size) could be drastically reduced thus implying a decrease in economic and design costs, leaving unmodified the safety condition for the process in the case.

The partial inerting is a prevention procedure that could also be included in the concept of a design inherent safety, a concept becoming more and more relevant recently.

As Amyotte (2005) reports the use of solid inertants is more applicable in terms of suppressing agents in a context aimed not to prevent but to mitigate the consequences of an inevitable explosion. Inerting indeed requires that fuel dusts has been intimately mixed with the inert material in the first stage of the process, while suppression consists in inject the inert powder into the just-ignited combustible dust to limit the generation of overpressure, even if the main intent is the same in both the techniques, i.e. the heat removal from the combustion reaction.

Practically the inerting method, with the need to premix combustible and inert dust, is less applicable than suppressing, because the contamination of process dust with foreign solids is often undesirable in terms of quality, hygiene or food safety. This contamination is less relevant in coal

mining industry where coal combustible dust generated during the process is extracted mixed with inert rock sand dust(limestone or dolomite for instance).

Main studies have been carried out on rock dust inerting coal combustible dust and investigations have focused on explosion severity and flammability limit (NiansengKuai et al. 2011), on minimum explosible concentration (MEC) and flame propagation velocity (Sapko et al., 2000), on dust layer ignition (Reddy, Amyotte and Pegg, 1998) and on flammability behavior and minimum inerting concentration (Chatrathi and Going, 2000).
More recent studies has been made also on other dust mixtures, involving other combustible powders largely diffused in industry process. Dufaud et al. (2010) investigated MIT cloud, LIT and MIE of several mixtures of combustible dust in addition with inerts like alumina, silica and sodium bicarbonate.

Mintz, Bray, Zuliani, Amyotte and Pegg (1996) studied MEC and MOC of mixtures of fine metallic powders with MgO as inert. Janes and Carson (2013) analyzed ignition sensitivity of fuel dusts like lycopodium, rice and coffee husks, powdered sugar with respect to different amounts of inerts (alumina and kieselguhr).

Hamdan and Qubbaj (1997) investigated oil shale dust in addition with calcium carbonate, stone and clay.

Marmo,Fois and Luzzi (2011) performed a research on MIE of flour, lycopodium and grain dust with silica, limestone and calcium carbonate.

The experimental campaign presented in this work (Danzi et al. 2014) deal with the investigation of LIT and MIT of mixtures (flour, sucrose, lactose as combustible and limestone, silica and extinguishing powders as inerts).

3.3   Ignition Temperature measurements

The procedure described by UNI EN 50821 is shown in Figure 15; measurements proceed in steps of 20 K until a value of temperature at which no ignition occur after 10atempts is found.

The MIT correspond to this value of temperature, at which 20K are furtherly subtracted as conservative approach required. (10K if the temperature found is below 300°C).

As said before, MIT represent a non-countable variable, not measurable directly, but that has to be estimated through sensitivity trials.
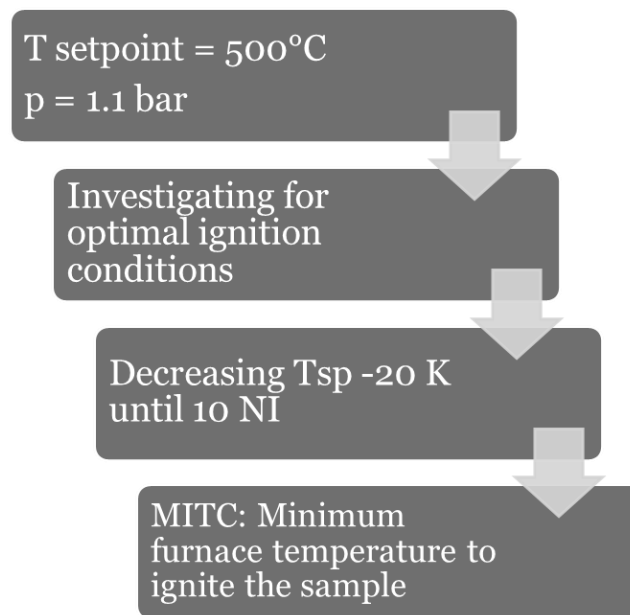

Figure 15: MIT according to UNI EN 50281-1:1999, Schematic procedures.

The dust sample subjected to test will respond to a certain applied stress, in the MIT case, a value of temperature, in order to highlight a specific critical threshold, in this case the ignition temperature.

If this threshold is overpassed by the stress applied to the sample, then a positive response will come (ignition), otherwise, a negative response will occur (no ignition). As already reported the transition between the positive and the negative response depending on the applied stress is not a straight demarcation, but consist in a probability range between 0 and 1. This probability value will increase and have a specific evolution with stress (temperature) rising.

Similarly to MIT procedure, the MIE measurement standard is described according to UNI EN 13821.

The sample is submitted to a certain electric discharge energy that will cause with a high degree of confidence an ignition. Then the test is repeated over a range of decreasing energy at the same concentration until no ignition occur in 10 consecutive tests.

When this energy is found, different concentrations are tested. If an ignition occurred with a certain concentration of dust, the first phase is repeated at this concentration, until 10 negative tests are found. The procedure is repeated until the maximum energy at which no ignition occur is found defined as E1, while E2 is the minimum energy at which ignition occur in 10 consecutive tests.

The MIE lays between these 2 values and it's calculated according to:

$$\text{MIE} = 10^{\log E_2 - \frac{I[E_2]*(\log E_2 - \log E_1)}{N[E_2]+1}}$$

(6)

Where:

$I(E_2)$ is the number of test with ignition at $E = E_2$;

$N(E_2)$ is the total number of tests performed at $E = E_2$.

Same assumptions about the ignition probability made on MIT apply on MIE, while in this case the variable will be the discharge energy and not the temperature.

The assessment of the minimum ignition temperature of dust layer, or LIT, is also reported in the UNI EN 50281.

The probability ignition problematic studied in this section applied also at this measurement since the investigation range of the ignition temperature is again 20K, and any intermediary results could be found, nevertheless this application is not part of this work and will be included in future perspectives.

3.3.1 Experimental setup and procedures

In the following paragraphs, the experimental setup adopted for all of the experimental measurements of MIT is described. When slight modifications of the standard procedures were required, these are reported and described in details.

MIT_C was measured by means of a Godbert Greenwald (GG) furnace (Chilworth Technologies, model MITC-L, serial number J303161B). Measurement procedure was developed according to CEI EN 50281-2-1:1999 as far as air sample injection overpressure and sample quantity are considered.

Particles size diameter characterization of the dust samples was made by laser diffractometry. The samples were characterized by the d10, d50 and d90 quantiles of the mass distribution as indicated in Table 1; the d_x diameter being defined as the percentage in weight of particles smaller than the x_{th} diameter.

As regards the inert dust particle, a further classification through mechanical sieving was performed to verify the effectiveness of the inertial effect according to particle size. The sieving system adopted was a tile of standard sieves operated by a sieving machine (model IG3/WET by Giuliani Torino). Different meshes were used, from 32 µm to 500 µm, which allowed the classification of inert dust by size.

The MIT_L test was performed with a hot plate apparatus. This consists of an aluminum plate over which the dust layer is placed and an electric resistance (1200 W) that allows to heat up the plate at the test set point. A sample holder made of a metallic ring enables to shape a 5 mm dust layer on the hot plate. Alternative holders allow settling 12.5and 25 mm height samples.

A thermocouple is placed to monitor the temperature in the middle of the dust layer. The plate temperature is controlled by a PID controller interfaced with a second thermocouple located inside the plate.

A conditioning treatment was performed on each pure material prior to measurements or mixture preparation (3 hours at 65° C in a laboratory stove). After stove drying samples were kept in silica-gel dryers to preserve the humidity residual until measurements. In any case no more than 48 hours lasted between conditioning and measurements.

Mixtures were prepared before each tests series using the conditioned pure material. A batch of some grams was generally prepared. To obtain the proper weight ratio given amount of pure

samples were weighted on an analytical balance and then mixed manually in a glass becker with the aid of a spatula.

Mixing was obtained both by mechanical blending with the spatula and by gentle rotation of the becker while it was kept tilted. The use of a glass container allowed to better observing the mixture behavior. No evident sign of segregation were observed with any material. This was also confirmed by the repeatability of the results obtained with any batch of mixture.

3.3.2 Definition of SIT derived from UNI EN 13821:2002

According to CEI EN 50281-2-1 several runs were made at decreasing temperature ($\Delta T = 20$ K above 300 °C and 10 °K below) in order to assess the temperature range (T1; T2) as described above. The air pressure in the dispersion reservoir was set at 1.1 bar. A 0.1 g sample of dust or mixture was blown into the furnace.

I ignition is accounted if there is a visible flame coming out of the furnace bottom. The presence of sparks in absence of flames is not considered proof of ignition. The T1 is accounted when no ignition occurs over 10 consecutive test. At this temperature, further investigations was made varying both the injection pressure and the sample weight (varying values of pressure release between 0.02 and 0.5 bar and sample weight (ranges from 0.01 to 1 g). In case of ignition the temperature is lowered by 20 °K and 10 test runs are repeated.

The procedure described above to assess ignition temperature of dust clouds returns T2 as the MITC value. The real MITC falls in the 20° C interval between T2 and T1.

A first attempt to define a more precise value can be derived on the basis of the data obtained according to CEI EN 50281-2-1 procedure by calculating the Statistical Ignition Temperature (SIT) according to Eq. () derived from the MIE definition given in UNI EN 13821:2002:

$$T_s = 10^{\log T_2 - \frac{I[T_2]*(\log T_2 - \log T_1)}{N[T_2]+1}} \tag{7}$$

Where:

T1 is the lower temperature at which ignition does not occur after 10 consecutive tests;

T2 is the temperature value at which at least one ignition occurs after 10 consecutive tests;

I(T2) is the number of test with ignition at T = T2;

N(T2) is the total number of tests performed at T = T2.

Since this last approach seem to return more accurate values it has been adopted at first instance in MIT_C measurement for combustibles dust mixtures. On the other hand MIT_C measurement according to CEI EN 50281-2-1 being more conservative give the temperature limit below which dust explosion risk is negligible in operating conditions. The new definition of SIT, described in Section 3.8, would demonstrated the poor accuracy of the standard definition itself.

3.3.3   MITL measurements

The dust layer was dropped on the hot plate once set point temperature (Tsp) was reached.

Ignition criteria was assumed in accordance with CEI EN 50281-2-1 as the hot plate temperature that provoked one of the following conditions: visible glowing or flaming; a temperature of 450°C in the dust layer; a temperature rise of above 250 K respect to the set for the hot plate. If no ignition occurred within 30 minutes the run was stopped. In case of ignition the test was prolonged until the sample temperature diminished below the plate temperature.

According to CEI EN 50281-2-1 measurement procedure for the MITL, several runs were made at decreasing temperature $\Delta T = 20$ K each step in order to assess the temperature range (T1; T2), where T1 is the lower temperature at which no ignition occurred and T2 is the upper temperature at which at least one ignition occurred. Further measurement at (T1+T2)/2 was performed to assess the minimum temperature at which an ignition occurred.

3.4   Materials

The combustible dusts tested were mainly typical materials processed in food industry like flour ("00" type) and three types of carbohydrates: Glucose, Lactose and Sucrose. In addition Sulphur was tested due to its very low MIT.

Inert dusts were Limestone, Silica sand and three different type of extinguishing powders.

For what concerns the MITC study all combustible dusts were sieved at 75 um and the under sieve fraction was used, while different particle size fractions of inert dusts (limestone and silica sand) were used to create mixtures with combustible dusts. This allowed to study the influence of particle size on the inerting efficiency. Size classification was obtained by sieving using a pile of standard sieves operated by a sieving machine.

Extinguishing powders were commercial products named respectively ABC93, ABC2010 and ABC40%. The first two of them were composed mainly by Phosphate Monoammonium (90%), the last is a mixture of Monoammonium Phosphate (40%) and Ammonium Sulphate (55%).

Table 2: PSD of dust samples tested.

Material	d_{10}	d_{50}	d_{90}	Notes
Flourtype "00" (Barilla)	10	34	78	Under sieve ASTM 75 um
Flourtype "00"(Barilla)	12	57	148	
Lactose(Milei)	6	33	101	
Lactose (Milei)	3	22	62	Under sieve ASTM 75 um
Glucose (Ardet)	6	35	83	Under sieve ASTM 75 um
Glucose (Ardet)	42	156	358	
Sucrose (PaneAngeli)	3	14	48	Under sieve ASTM 75 um
Sucrose (PaneAngeli)	4	20	129	
Lycopodium (Aldrich Chemistry)	22	30	41	
Limestone 32/75	6	23	62	Classified by sieving
Limestone 125/250	154	234	351	Classified by sieving
Limestone 250/500	294	446	690	Classified by sieving
Silica sand 32/75	20	56	109	Classified by sieving
Sulphur	3	10	46	
Extinguish powder ABC40 (Gibi SRL)	7	61	196	
Extinguish powder ABC40 (Gibi SRL)	4	25	67	Under sieve ASTM 75 um
Extinguish powder ABC1993 (Gibi SRL)	5	58	199	
Extinguish powder ABC1993 (Gibi SRL)	3	21	63	Under sieve ASTM 75 um
Extinguish powder ABC2010 (Ecoplanservice S.A.S.)	10	72	249	
Extinguish powder ABC2010 (Ecoplanservice S.A.S.)	6	26	72	Under sieve ASTM 75 um

3.5   Minimum ignition temperature of dust clouds

The MIT_C of dust mixtures between flour and different inert is plotted in Figure 1 against mixture mass fraction. Values are measured according to CEI EN 50281-2-1. As expected the MIT_C of mixtures between a combustible and an inert dust increases when the inert dust concentration increases. The behaviour of the mixture depends on the inert characteristics'. As expected the maximum increase of MIT_C is obtained when extinguishing dusts are used. This behaviour is due to the combined endothermic decomposition of the extinguishing dust which subtracts heat to the system and to the chemical decomposition that produce gases so depleting Oxygen concentration in the test chamber.

The use of limestone has much less effect on MIT_C than extinguishing dust. The MITC at 90% inert rises only to 540°C (instead to 940-960°C). This is roughly the MITC showed by a 50% mixture flour-extinguishing dust.

Measurements show almost no effect of inert particle size on MITC, at least as far as the MIT definition given by CEI EN 50281-2-1 is used. Only in case of 60% limestone content, the 125-250 um inert was, as expected due to the lowest surface area and hence to a less efficient heat subtraction, less efficient than the finest 32-75 um.

In order to appreciate the mixture composition effect when dusts with similar ignition temperature are mixed, the SIT of flammable dust mixtures is presented in Figure 2. There, the statistical temperature calculated according to Equation (#) is plotted versus the mixture weight fraction. The Sucrose-Flour mixture behaviour is such that the dependence of the SIT versus %W is almost linear. Mixtures containing Lactose do not exhibit the same trend, since the SIT decrease steeply as the amount of Lactose decreases from 100% to 75%W. In case of Lactose-Flour mixture, at 75% content of Lactose the SIT is almost equal to the SIT of pure Flour. The mixture Lactose – Sucrose show similar trend, whereas at 75% Lactose the SIT is almost halfway between pure Lactose and pure Sucrose SIT. In Figure 2 also the MITC of the mixture Sulphur-Flour is presented. As expected the MITC decreases with increasing Sulphur content, confirming previous data by Dufaud et. Al. (2012).

The same authors proposed the adoption of an harmonic model to predict experimental data on ignition temperature of dust mixtures. This model was then adopted in this part of the work to predict the evolution of the SIT of a mixture made of dusts A and B:

$$\frac{1}{SIT_M} = \frac{(x_{vol})_a}{SIT_a} + \frac{(x_{vol})_b}{SIT_b} \tag{8}$$

The predictions obtained with the harmonic model are presented in Figure 2 as well. In general the harmonic model is no capable to predict the SIT trend.
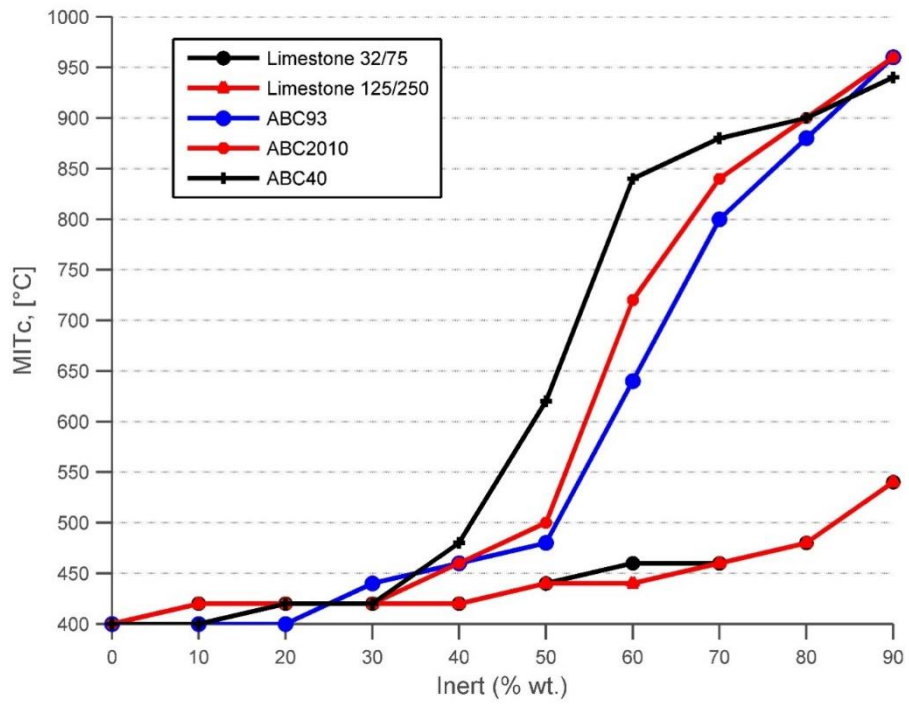
Figure 16: Minimum ignition temperature of dust clouds of flour-inert mistures.
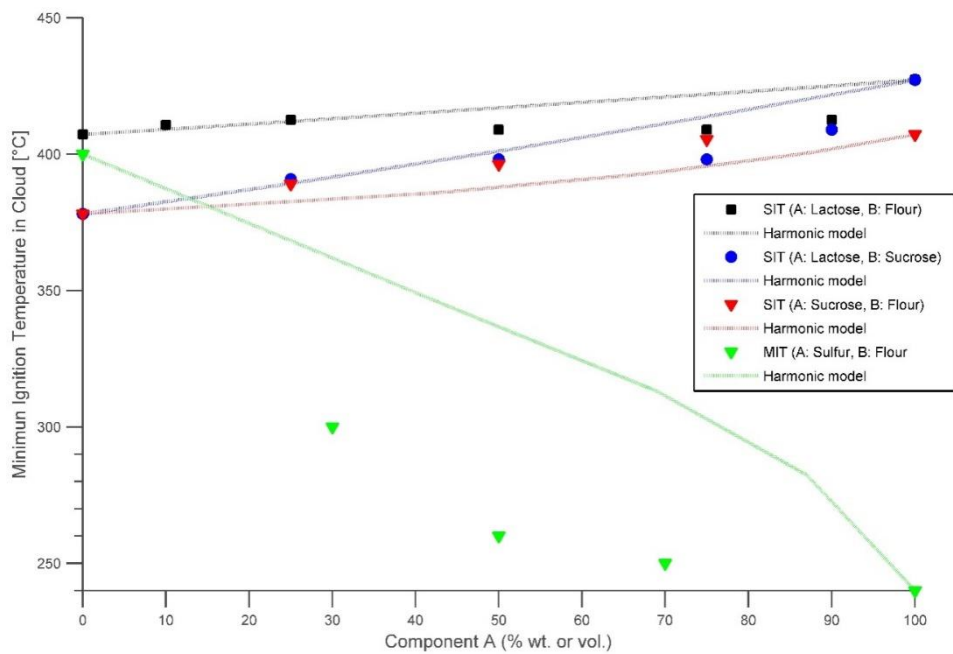

Figure 17: Statistic MIT$_C$ .of various dust mixtures. Harmonic model (Eq. 2) is also represented as dotted line.

3.6   Minimum ignition temperature of dust layers

The minimum ignition temperature of layers of flour mixed with various inert dusts is presented in Figure 3. Pure flour exhibited a MITL of 390 °C (5 mm layer), whereas ignition was difficult. The flour layer curved and fractured a few minutes after run start. This behavior makes ignition difficult, because the heat transfer from the heating plate is reduced to a great extent. Mixtures flour limestone (32-75 µm) do not exhibit such behavior and as a consequence they have a MITL which is lower than pure flour MITL at inert weight fraction up to 40%. Further increase of inert weight fraction make the MITL rise sharply above 400°C.

Mixtures of flour with coarser limestone fractions, although preventing the mixture layer curvature, did not exhibit such a drop of the MITL. The MITL of these mixtures was not measured because exceeded the measuring range of the hot plate used in the experiments (400°C).

Quite surprisingly the finer 32-75 µm limestone dust is the less efficient to prevent layer ignition. On the contrary, it seems to promote layer ignition, since the ignition temperature measured are well below the pure flour MIT_L. This seems mainly a consequence of the fact that these mixtures do not curve during heating. The layer remains well adherent to the plate surface and ignite quite easily. Also in the case of mixtures with coarser inert particles the dust layer remains in contact with the hot plate but no ignition is observed.
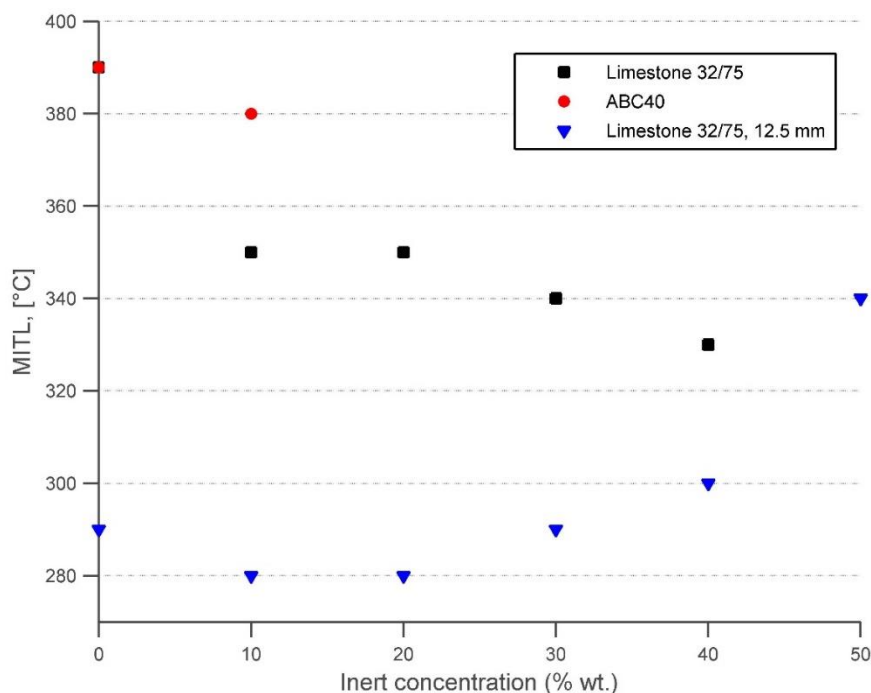
Figure 18: MITL .of mixtures with flour and different inerts.

MITL of the mixture between flour and inert dusts exhibit unexpected behavior and a strong dependence upon the particle size distribution of the inert. Limestone 32-75 µm mixed with flour provoke a 70 °C drop of MITL to 330 °C from 390°C of pure flour.

Also in case of extinguishing powder ABC 40 a slight decrease of MIT_L occurs at 10% inert. In both cases the presence of an inert promote ignition. Such dust mixtures present a higher ignition risk than pure flour. This is mainly due to the behaviour of a 5mm thick flour layer that, as deposed onto the hot plate, cracks and curves. The smaller contact area with the hot plate under these conditions makes ignition difficult.

This phenomenon do not take place in case of mixture.

Again quite surprisingly, in the case of flour-limestone mixture, using a coarser inert do not provoke a decrease of the MIT_L.

3.7　Statistical Ignition Temperature according to Normal law

The methodology base itself on the evolution of the dust ignition probability within a certain well-defined number of attempts. The main objective of the work will be to individuate a new stopping-criterion for the ignition investigation test.

In particular the methodology tried to minimize the attempts number and in the same time to obtain a result with a high accurate reliability range (i.e. within a confidence interval above a defined level, as an example above 95% confidence range).

In the same time the work go into the perspective, cited in Chapter 4, of giving a reliable statistical character to the SET different test procedures, described in the same chapter. The principle is: we have to find the less possible attempts for the more reliable result that will define the sample as safe or not.

The stopping-criterion at which the standard UNI EN 50281 impose to stop the test is the observation of 10 consecutive tests without ignition at a certain temperature. The procedure is costly in terms of time and economic efforts due to the relative high number of tests at different temperatures to be performed in order to determine the minimum ignition temperature.

A stopping-criterion based on a "a priori" defined number of negative attempts that will define the test as negative could allow the definition of a confidence range (expressed in percentage) that grant the result of the test correspond effectively to a negative response (safe condition).

Being the tests "destructive" is not possible to measure on each sample the critical value. It's necessary then to perform a relevant number of tests with different samples, the greater the experimental data the more accurate the estimation of the Statistical Ignition Temperature. For each different degree of stress it could be derived a response curve, i.e. the ignition probability in function of the temperature.

Methodology consist in 10 consecutive attempts of ignition performed at step of 5K from each other. The experimental data are compared with a reference mathematical model, related to the event that is considered. In the present case, the normal distribution (or Gauss) has been adopted to fit the data, assuming that they followed the evolution of a cumulative normal distribution curve. Mean and standard deviation of the curve are derived from tables referred to normal distribution [], where the standard aleatory variable z is defined as:

$$Z = \frac{X-\mu}{\sigma} \tag{9}$$

Where:

X is the value of test temperature, mu and sigma are respectively the mean and the standard deviation of the unknown normal distribution.

Two values of Z (at 2 T) are then compared and associated to the ignition experimental probability found on 10 tests at these temperatures, in order to solve the system and obtain the value of mu and sigma that defines the cumulative distribution desired.

The SIT is defined as the temperature at which the probability of ignition is equal to 5%. This threshold value is chosen according to the work of Bernard et al. (2010), dealing with statistical determination of the MIE of dusts.
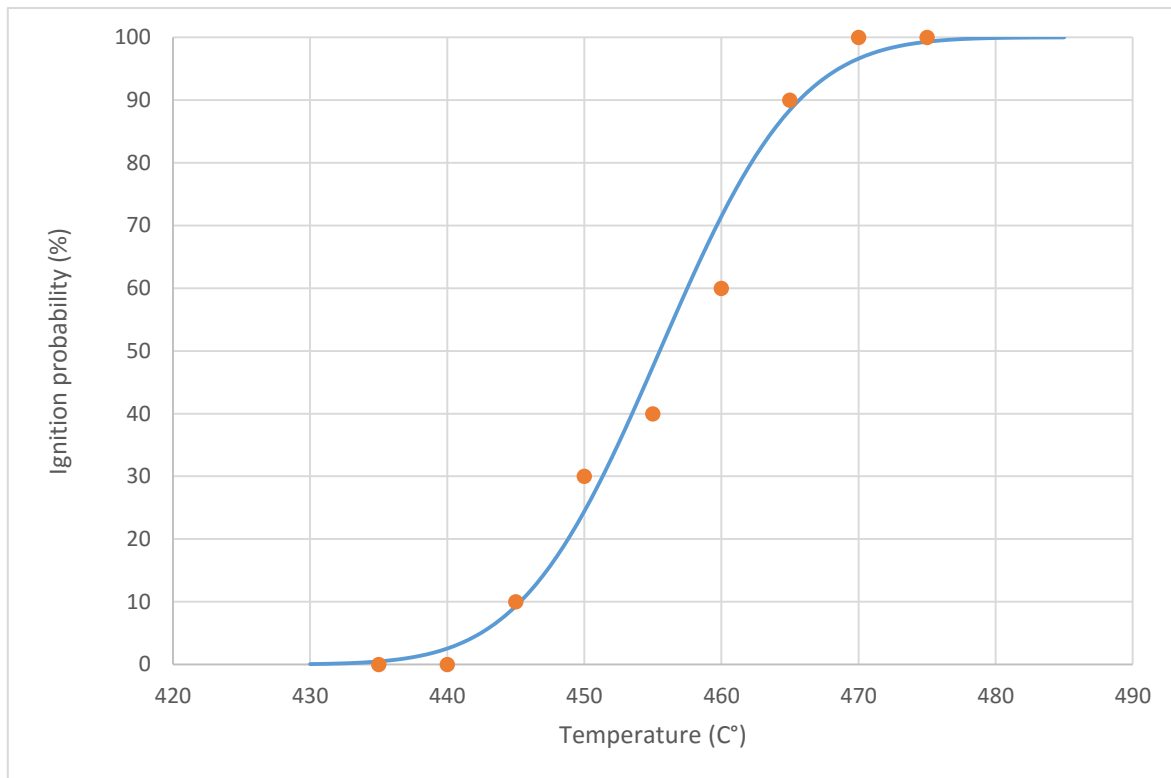
Figure 19: Ignition probability with respect to temperature of pure Lycopodium, experimental data (dots) are plotted with the Normal distributive curve found.

Table 3: Summary of attempts performed with Lycopodium dust, where I. means ignition and N.i. no ignition.

Test n°	440°C	445°C	450°C	455°C	460°C	465°C	475°C
1	N.I.	N.I.	N.I.	N.I.	I.	I.	I.
2	N.I.	I.	I.	I.	N.I.	I.	I.
3	N.I.	N.I.	N.I.	N.I.	I.	I.	I.
4	N.I.	N.I.	N.I.	N.I.	I.	N.I.	I.
5	N.I.	N.I.	N.I.	I.	N.I.	I.	I.
6	N.I.	N.I.	I.	I.	I.	I.	I.
7	N.I.	N.I.	N.I.	N.I.	I.	I.	I.
8	N.I.	N.I.	I.	I.	N.I.	I.	I.
9	N.I.	N.I.	N.I.	N.I.	I.	I.	I.
10	N.I.	N.I.	N.I.	N.I.	N.I.	I.	I.
Ignition	0	1	3	4	6	9	10

Table 3 show the application of the methodology with experimental tests on Lycopodium powder (which characteristic sizes are reported in Table 2). As the table reported, each temperature is associated to an ignition probability, according to the results of the tests, for example at 445°C a probability of 10% is associated (1 ignition on 10 tests performed).

It's worthy to note that the probability range (and then the PEI) stretches from 0% at 440°C to 100% at 475°C.

With the purpose to validate the statistical approach adopted an experimental campaign investigating on the ignition temperature of Lycopodium dust – inert dust has been performed.

3.7.1 SIT of inert-combustible dust mixtures

Inert dust utilized is the AC93 powder, presented in Table 2and constituted mainly by Phosphate Monoammonium. Physical characteristic of this extinguishing powder are provided by the following Table.

Table 4: Physical properties of extinguishing powder AC93

Chemical composition: Phospate Monoammonium	90%
pH	5.5
Melting point	170-190°C
Density	0.9 g/cm^3
Not soluble in water	-
Relative humidity	0.85%

Mixture preparation adopted the same procedure described previously in this Chapter. The weight ratio of Lycopodium-AC93 was 3:2. The standard procedure to evaluate the MIT (UNI EN 50281) has been adopted to compare its results with those expected with the SIT method based on the Normal law, as in the case of the pure Lycopodium dust sample.
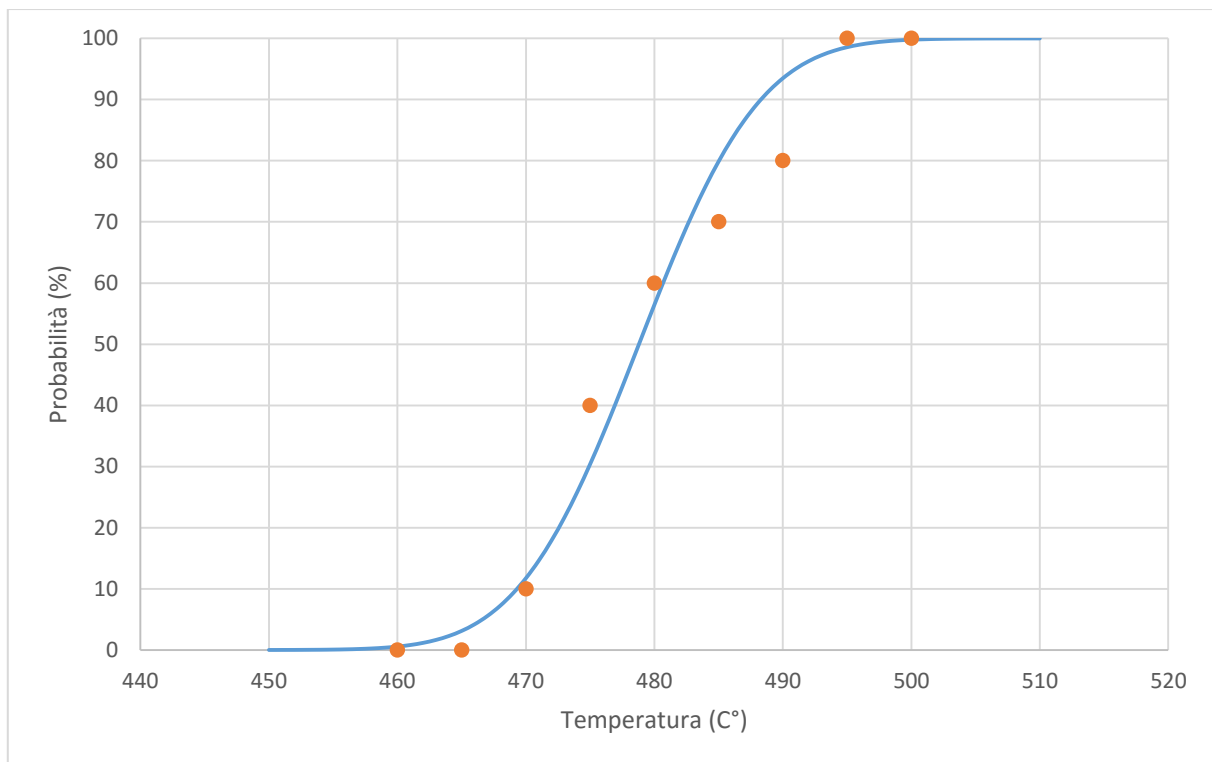

Figure 20: Ignition probability with respect to temperature of Lycopodium in mixture with AC93 (experimental data with Normal distribution curve).

First ignitions here are found at 470°C, while PEI stretches up to 495°C, 10°C greater than the interval defined with the pure Lycopodium dust.

3.7.2 Influence of PSD of inert dust on ignition sensitivity of mixture

As to evaluate the influence of PSD of inert dust on the ignition sensitivity of the mixture the same procedure has been applied for a mixture of Lycopodium dust and AC93 powder, that has been sieved to 75 um through mechanical sieving operator (the sieve standard number is ASTM 200).

The curve relative to this third mixture shifts towards higher temperatures, while the PEI stretches between 475°C and 500°C. This demonstrates the efficacy of a finer particle distribution on the inertial capacity of the extinguishing powder.
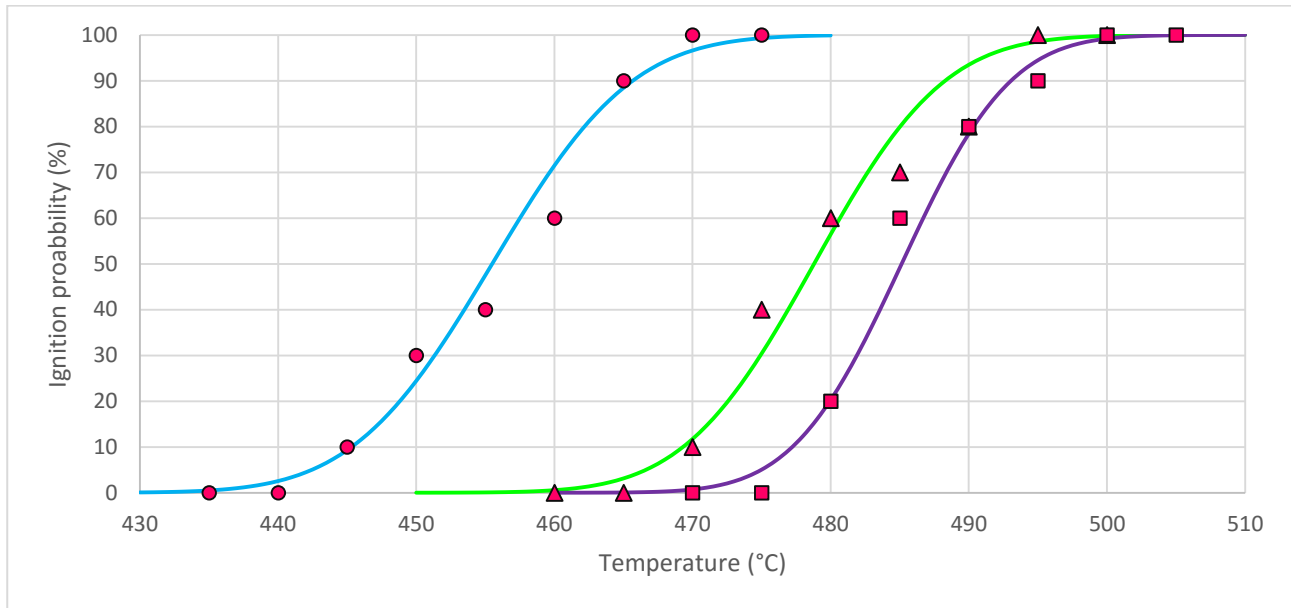

Figure 21: Ignition probability vs Temperature of: Lycopodium dust, pure (blue line, red dots); Mixture with AC93 (green line, triangles); Mixture with AC93, sieved at 75 um (purple line, squares).

3.8 Statistical Ignition Temperature according to Generalized Linear Models

This section deals with a third more comprehensive approach that has been adopted for the definition of the ignition temperature of a pure combustible dust sample, comparing experimental with Generalized Linear Models (described below).

The autoignition temperature (AIT) of a dust cloud is defined as the temperature of a hot surface that can provoke the spontaneous ignition of a dust cloud that comes into contact with it and is measured according to CEI EN 50821 (whose procedure is described above in chapter ###) or ASTM E1491.

In order to assume that the ignition probability at MIT, p_{acc}(AIT) is smaller than p^*_{acc}, measurement procedures defined by technical standard (CEI EN 50821) imply to make multiple ignition attempts at decreasing T, spanning 20°C at each attempt (10 °C if below 300°C). The test ends after 10 attempts without ignition at a given T, say T_X, which is also defined as MIT.

Conceptually, this procedure poses two serious problems. The first is that AIT assumes discrete values. Under a purely scientific point of view, the accuracy could be non-adequate to study certain behaviors, such as the effect of admixing two dusts or the effect of a change of moisture, PSD, etc.

The second kind of problem is that there is no theory to assess which is the ignition probability at T_X. As a consequence, it is impossible to state which is the confidence of the statement that 10 negative attempts at T_X mean no ignition at T_X.

A significant improvement would come from the knowledge of the dependence between the ignition probability and T over PEI, where p(T) varies from 0 to 1. Further improvement will come from defining a suitable probability threshold for the "safe" condition. Recently, Bernard et al. (2010) have assumed this threshold as 0.05 (5%), which is a figure much higher than what usually accepted for undesired events. This value has been adopted by the authors according to pyrotechnics industrial requirements, being 5% the probability of ignition corresponding to the higher energy which provide adequate safety in storage of those materials

The aim of this work is to define the dependence of p(T) vs T, such to better define the AIT and its measurement procedure. To this purpose, following common assumptions on undesired root events, p_{acc} will be assumed as 0,001.

3.8.1 A new definition of MIT

As we mentioned in the Introduction, the PEI is the range of temperatures where the autoignition of a dust cloud is not a deterministic event: even when the experiment is performed under the same experimental conditions, repeated trials yield different outcomes.

It is therefore appropriate to formulate a mathematical model for the ignition probability. A few natural requirements for such model are listed below.

- The outcome of any single trial at a given temperature T_i is a random variable Y_i whose value is either 1 (ignition) or 0 (no ignition) and the ignition probability is a function $p(T)$ of the temperature T such that $E(Y_i) = p(T_i)$.
- An ignition never happens if the temperature is small enough while if t is sufficiently high we always get ignition. That means that $\lim_{T \to 0} p(T) = 0$ and $\lim_{T \to \infty} p(T) = 1$. It is fairly reasonable to assume that the function $p(T)$ is increasing and continuous.

From the statistical viewpoint this situation is well described in the framework of Generalized Linear Models (GLMs), cf. McCullagh, P. and J. A. Nelder. (1989) on which we base all the discussion below. In the classical univariate Linear Model (LM) the mean of the response variables Y_i (independent and with the same variance, often assumed to follow the Gaussian distribution) is a linear function of the independent variable, $E(Y_i) = \beta_0 + \beta_1 T_i$.

The more flexible univariate GLM extend the LM in the following sense:

- The response variables Y_i are assumed to be independent random variables, each with a distribution from a specified exponential family, in our case Bernoulli random variables with mean $E(Y_i) = p(T_i)$.
- The linear predictors $\eta_i = \beta_0 + \beta_1 T_i$, sometimes called Systematic Component of the model, are linked to the expectation of the responses $E(Y_i) = p(T_i)$ by a function g, usually called the link function, such that $g(p(T_i)) = \eta_i$.

For models with dichotomous outcomes the link functions that are most commonly used in practice are:

1. the logit link g(p)= $\log \frac{p}{1-p}$ whose inverse function is the logistic function, that, composed to the predictor, gives $p(T_i) = \frac{e^{\beta_0 + \beta_1 T_i}}{1 - e^{\beta_0 + \beta_1 T_i}}$.
2. theprobit link $g(p) = \phi^{-1}(p)$ whose inverse is the Normal Cumulative Distribution Function ϕ that, composed to the predictor, gives $p(T_i) = \phi(\beta_0 + \beta_1 T_i)$.
3. the complementary log-log function (Cloglog), $g(p) = \log[-\log(1-p)]$ whose inverse, composed to the predictor, gives $p(T_i) = 1 - \exp[-\exp(\beta_0 + \beta_1 T_i)]$.

Logit and probit are symmetric link functions and they just differ in how fast they decrease to 0 (and increase to 1). Probit is faster. Cloglogis mainly used when there is evidence of the asymmetric behavior between the right and left tail.

Whatever link function is chosen, from a set of experimental data Maximum Likelihood estimators $\hat{\beta}_0$ and $\hat{\beta}_1$ for the parameters (and the relative standard errors) are computable. An estimated ignition probability curve is given by $\hat{p}(T) = \frac{e^{\hat{\beta}_0 + \hat{\beta}_1 T}}{1 - e^{\hat{\beta}_0 + \hat{\beta}_1 T}}$ in the case of logit link and by the analogous expressions with other links. To such estimated probability curve we can also associate confidence intervals and in particular in the next section we are interested in the upper bound $\hat{p}_{U,\alpha}(T)$ of the one-sided confidence interval $\left(0, \hat{p}_{U,\alpha}(T)\right)$. This value is such that the true value $p(T)$ of the ignition probability is smaller than $\hat{p}_{U,\alpha}(T)$ with probability $1 - \alpha$.

Useful statistics for assessing the fit of the model are the Pearson χ^2 and the Deviance G^2. Under the hypothesis that the model is true, their asymptotic distribution should be approximated by a chi-square distribution with n-2 degrees of freedom (where n is the number of different values of the covariate at which the tests are performed and 2 is the number of parameters we are estimating). The approximation, however, is known in many relevant circumstances to be quite poor so that p-values of the tests based on such statistics are not reliable. Still, smaller values of such statistics provide indication for a better fit.

The definition of autoignition temperature given in the European standard CEI EN 50821 is an operative definition, based on the results of an experiment. The number of trials requested for such experiment is small and there is no scientifically sound assessment of which is the probability that an ignition occurs at such temperature.

Based on the models above it is possible to formulate an abstract definition of the autoignition temperature probability that is independent of the experiment outcome. According to our model (whatever the link we use) the ignition probability $p(T)$ is strictly positive for any T, so that it is not possible for any temperature to guarantee that no ignition can occur. However if we agree to fix a small enough ignition probability level k that is considered a tolerable risk, then temperature T_k at which $p(T_k) = k$ could be considered as a new definition of the autoignition temperature.

Operatively, after an experimental campaign we can give an estimate of such a temperature in at least two ways. The naïve way is it to define the estimator $\widehat{T_k}$ as such value of the temperature for

which the estimated ignition probability curve $\hat{p}(\widehat{T}_k) = k$. Such a value is still random and it would be safer to take into account its variability. A more prudential estimate would be given by the value of the temperature such that the upper confidence bound curve $\hat{p}_{U,\alpha}(t)$ gives $\hat{p}_{U,\alpha}(\widehat{T}_k) = k$. At such temperature, we can affirm that the probability that the true value of $p(\widehat{T}_k)$ is smaller than k is $1 - \alpha$ (95% for example).

3.8.2 Experiments

Lycopodium was used as test dust, due to its stable characteristics and narrow PSD. All the tests were done using a single 1kg batch of dust from Sigma-Aldrich so to avoid any influence on the results. Size characteristic of Lycopodium samples are those provided by Table 2

Autoignition tests were done in a GG furnace (Chilwort technology, SN J303161B). A given amount of dust, weighted on a laboratory balance, is placed in the injection system and blown into the test room by an air pulse. The pulse is obtained by releasing the air contained in a reservoir previously pressurized to a given pressure. Compressed air with a dew point of -4°C is used to blow the sample into the test room. Test conditions are the furnace temperature (T), the dust mass (m) and the blowing pressure (P). Optimal mass m and P conditions were defined as those that produced the most evident explosion in a series of test at a range of temperature well above the AIT. Same conditions for P and m were maintained through the whole campaign, i.e. 0.25 bar and 0.1 grams.

The autoignition probability was measured in the temperature range 434 to 473 °C, which is wide enough to let $p(T)$ to span from 0 to 1. The trials were done at 3°C interval, such to cover all the expected PEI. The number of trials at each temperature was set to 40 at the tails of the distribution and to 30 at the center.

3.8.3 Data analysis

The results of our experimental campaign are summarized in Table 5. The PEI for Lycopodium seems to span around 30°C (440-470°C). As expected the ignition probability increases with T. A plateau seems to appear at about the PEI middle (452-458). It is still unclear whether this is the real behavior of the system or the consequence of an uncontrolled variable. A discussion about the AIT that would be obtained according to CEI EN 50821 is given at the end of the next section.

Table 5: Summary of experimental campaign, PEI is highlighted in green.

T (C°)	434	437	440	443	446	449	452	455	458	461	464	467	470	473
Trials	40	40	40	30	30	30	30	30	30	30	30	30	40	40
Ignitions	0	0	0	6	5	10	14	15	15	20	27	27	40	40

We estimated the parameters of GLM models with different link functions and the results are summarized in Table 6. Comments on the quality of the fit and on diagnostic tools are given in the discussion of next Section.

Table 6: Regression analysis. The estimated values $\hat{\beta}_0$ and $\hat{\beta}_1$ are reported with relative standard errors. The value of the fit statistics χ^2 and G^2 are also reported with their degrees of freedom.

Link	$\hat{\beta}_0$	$\hat{\beta}_0$ s.e.	$\hat{\beta}_1$	$\hat{\beta}_1$ s.e.	Dev. G^2	Pears. χ^2	d.f.
Logit	-91.7	7.7	0.20	0.02	25.2	20.5	12
Cloglog	-62.6	4.9	0.14	0.01	23.9	18.5	12
Probit	-53.57	0.01	0.12	0.01	20.9	17.8	12

The data and the estimated models are compared in Figure 1, where two-sided 95% confidence interval for the estimated ignition probability curves obtained in the different models are plotted.

Figure 22 shows quite many points outside the confidence bounds in all three models. This is an indication for a lack of fit (less apparent in the probit case), which is also confirmed by the high values of the Deviance and of the Pearson statistics. A chi-square test at 95% based on the Deviance values rejects the null hypothesis that the data follows a GLM with logit and cloglog link. In the case of probit link the p-value is 0.052 which does not lead to rejection at 95% confidence level but that is very close to the critical value. The same test for the Pearson statistics would be more tolerant. Let us however remember that in our case the chi-square approximation on which such tests are based might be rough and it is much better not to take such values too seriously. In any case both deviance and Pearson statistics suggest that the probit model is preferable to the other candidates.
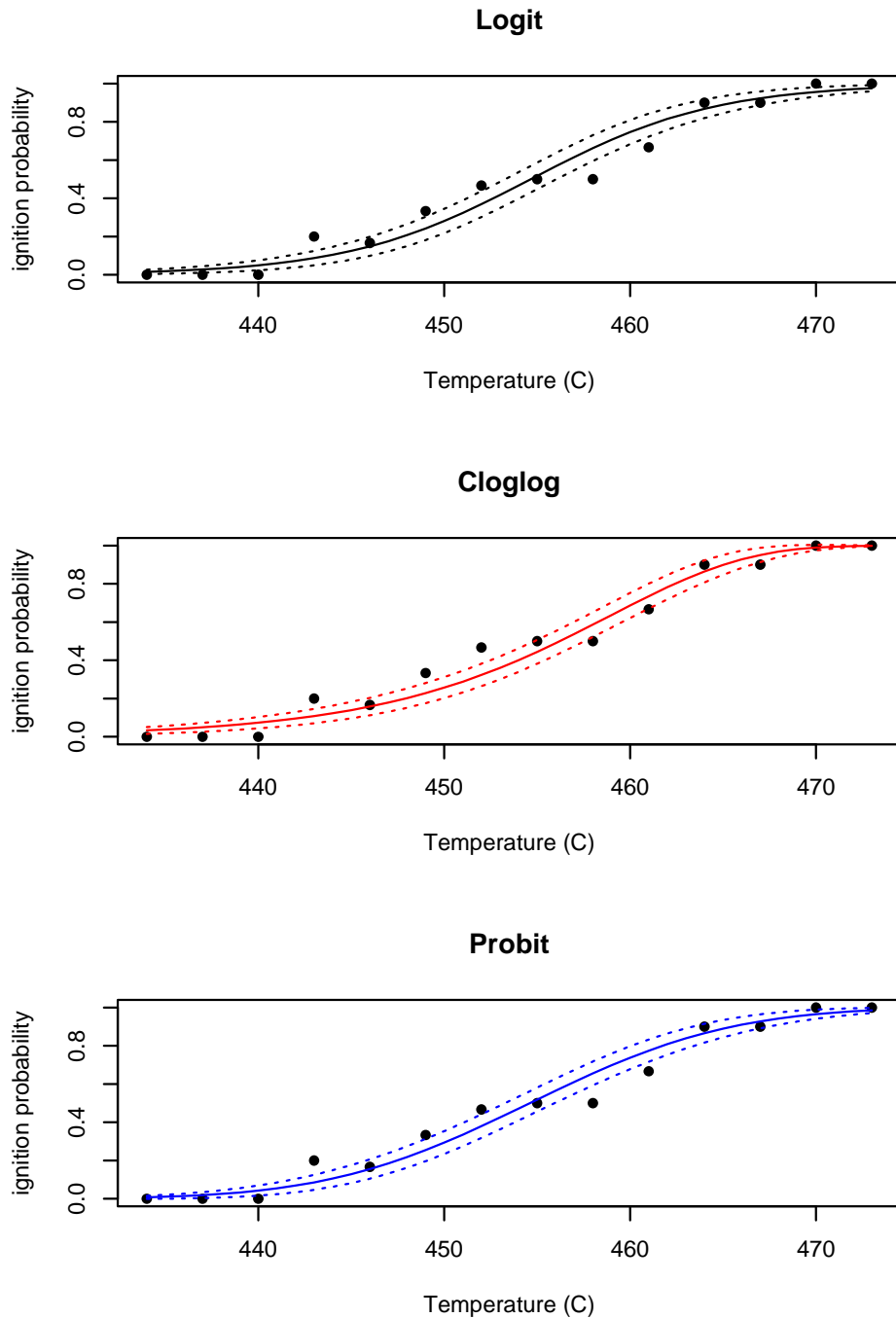
Figure 22: Plots of the data compared with the fitted models.

As a further diagnostic check, we consider the value of the overdispersion parameter. In the literature it is reported that due to different causes, for example in the case of a missing covariate, data very often display too much variability with respect to that predicted by the model. This is apparent also in our case as it can be seen from Figure 22. A measure of the overdispersion is the ratio between χ^2 and the number of degrees of freedom. If it is greater than 1, overdispersion is present to some extent. The overdispersion parameters for the logit / cloglog / probit links are displayed in Table7 and they again suggest that the probit link guarantees the smallest overdispersion among the three considered.

Table7: Overdispersion parameter

Overdispersion		
Logit	Cloglog	Probit
1.71	1.54	1.48

Finally we can affirm that among the three model we have considered the GLM with probit link gives the best fit, even if some overdispersion is still present. The reason for this overdispersion deserves to be investigated as could be due to some environmental factors that could have affected the data acquisition process. Apparently, all the process variables were properly controlled except than ambient temperature, pressure and moisture content. Since the data set was acquired through many days, a change in the weather conditions could have influenced to some extent the data set. In any case these variables should have minor effects on data dispersion, since they do not affect the test environment, except for ambient pressure.

A GLM with random effect might deserve to be considered as an alternative model that could account for some unmeasured source of variability that is not considered in our analysis. This topic will be subject of further studies together with a more controlled experimental setup and air compressors system.

A prudential estimate of the autoignition temperature can be given as such value of the temperature at which the upper confidence bound curve $\hat{p}_{U,\alpha}(T)$ gives $\hat{p}_{U,\alpha}(\widehat{T_k}) = k$ (cf. Section 3.8.1 for more details). As ignition can be considered as a root event of a chain leading to an accident (e.g. in case mitigation/protection devices fail to protect the system), it seems reasonable to assume as ignition tolerable probability a value $k = 0.001$ (any different value could be considered as well on the basis of a risk analysis)

The estimates of the autoignition temperature according to the three models are displayed in Table 8.

Table 8: Estimates of the autoignition temperature.

Autoignition temperature (C)		
Logit	Cloglog	Probit
427.8	417.8	434.1

What about CEI EN 50821 norm? It prescribes to start at 500 C and to decrease each time by 20 C unless no explosion is seen in 10 trials. In Table 5 we report the probability that a trial conducted according to the norm would stop at given temperature assuming that the true model is a probit with the parameters we have estimated.

Table 9: Probability that the CEI EN 50821 norm identifies a given temperature as the AIT given the probit model.

Autoignition temperature according to CEI EN 50821 (C)				
Temperature	460	440	420	400
Probability	1.5e-06	0.65	0.35	8e-05

The resulting value of the AIT is highly volatile. The probability of stopping the trial at 500 C, 480 C and 460 C is negligible, since the ignition probability is rather high at such temperatures while at 440 C the probit model gives an ignition probability of 0.043 that corresponds to a probability of getting zero ignitions in 10 trials of 0.65. That means that in the 65% of the cases (if the probit model were true), after a campaign designed according to the norm once would evaluate the AIT to 440 C. This is a clearly too high value that corresponds to an ignition probability of 4.3%. In the remaining 35% of the cases, the experimenter would have recorded the AIT as 420 C that is 14°C less than the already conservative value obtained with our method.

We believe that such a result is quite relevant and that the use of sophisticated statistical methodology such as those introduced in this paper would foster the development of safer procedure based on more solid theoretical ground.

The development of a explosibility test for dusts

Does this dust will explode? This is one of the most common questions that employers or HSE specialist asks if approaching a new explosion risk assessment or updating an existing one after a change in the manufacturing process.

Nowadays several technical standards (ASTM 2012, UNI-EN 1999, 2004) can be used as guide for dust testing, in order to measure the typical parameters that describe the behavior of an explosible dust (Minimum Explosible Concentration, MEC, maximum pressure rise P_{max}, the deflagration index k_{st}, minimum ignition energy MIE, autoignition temperature AIT).

As underlined in chapter ## laboratory tests necessary to measure the above parameters are generally expensive, especially for SME with small budget and often many different dusts whose behavior is a priori unknown. It may happen that, in face of a possible negative result, employers avoid to submit dusts to expensive tests so underestimating in fact the risk due to all the explosible samples which are not tested.

Work by Rodgers and Ural (2011) present the issues related to marginally explosible dusts.

They reported that combustible dusts could be:

- Non explosible;
- Marginally explosible;
- Severely explosible.

This classification do not come from any standard or any threshold value dependent on explosible dust parameters. The issue posed by this work is then the definition of the marginal explosibility criterion and how standard test method lose their accuracy when "marginally explosible" dusts are concerned.

In effect, same legal restriction are to be satisfied when dealing with marginal or severely explosible dusts. For these reason standard tests for explosibility definition has to be the more universal and accurate and not have any dependence on the nature of the analyzed dust sample.

The key factors for the authors are the quenching limit and the flammability limit. The definition of explosibility (in this context associated to the term flammability with the same meaning) is referred to the ability of a dust cloud to be ignited by a sufficiently high energy source and to auto-sustain a deflagration when the energy provided is dismissed (or far from the igniton source position).

To demonstrate the self-propagating nature of the flame generated from the dust tested, the key point is the dimension of the test apparatus: flame thickness has to be larger than the enclosure space to avoid relevant heat losses at the walls that could prevent the flame to propagate. The quenching limit is the condition in which heat generation from the combustion reaction exceeds the heat loss and let a self-sustained flame to propagate. Flammability refers as the condition in which an adequate concentration of dust generate sufficient energy to auto-sustain its flame and it is not limited by the quenching or by weak ignition energy.

The problematics with small test apparatus rises from the fact that in such enclosures, combustion products remained close to the flame and add to it their energy, letting the propagation to occur. This is defined as the overdriving of an explosion. In a larger test apparatus, with the same ignition source, a smaller volume fraction is affected by the ignition energy and thus result in easier prediction of a false positive.

For this reason, standard test for the determination of the explosibility of dusts are more likely to be performed in larger test equipment, like the 1 m3 chamber or the 20–L Siwek sphere.

Hartmann tube was used for flammability test (see Table 10), but less used recently because of the relative low ignition source that could be used and for the quenching limitation, as Rodgers reported.

ASTM E1226 proposed the explosibility test with the 20-L spherical vessel with 5 or 10 KJ chemical igniters. Actually, if an explosion occur with these "high" energetic igniters, and no measurable explosion is achieved with the 2.5 KJ igniters, then the system is supposed to have overdriven the reaction.

The ASTM recommend in this case the repetition of the test in a $1m^3$ chamber to assess the actual nature of the dust. In the same study, the authors made a comparison between dust explosible properties, tested in the 20-L sphere and in the 1 m^3 chamber. They concluded that dust that exhibit a k_{st} value lower than 45 bar-m/s in the sphere would likely be non-explosible in the larger test apparatus.

4.1   Yes/No screening test, state of art

The need of a lab-scale test that could straightforward establish, with a relative high grade of reproducibility and that gives statistically safe results in defining a dust sample "capable of giving an explosion or not" is desired.

Since earlier studies on dust explosion, this task was taken into account by scientific worldwide research, the amount of dust sample to test (concentration in air) and the energy of the ignition source being the two more relevant variables involved.

These two variables are chosen in order to grant the main characteristic of the test, i.e. to create the "worst conditions as possible" that is the best conditions for an ignition to occur.

The size of the dust cloud to be tested has to be adequate with respect to the experimental setup in which is dispersed, as to maintain a certain level of turbulence, but not lower than the minimum explosible concentration of dust in air (MEC). The optimal solution should be the stoichiometric concentration of the examined dust in air, factor that depends on the sample chemical nature. A reasonable attempt is a dust cloud of 500-1000 g/m^3, which is the range in which most combustible dust are found to be explosible (according to Amyotte, 2013).

An adequate powerful ignition source is an ignition that has the energy to start the combustion process and the self-sustained flame propagation in the dust cloud. More discussions are nowadays still opened on the optimal ignition sources, thus depending on factors non-inherent with the specific samples to test, but to external factors also (dispersion system, industrial equipment involved).

The first information on the utility and practicing of a screening test, investigating dust explosibility is dated back to seventies and developed by Palmer (Palmer, 1973).

More recently some other test procedures, also with different laboratory setups, has been proposed. summarizes tests together with setup, testing conditions and criteria for ignition (where specified).

Table 10: Review of dust explosibility tests.

Development	Setup	Ignition source	Test conditions	Ignition criteria
Palmer, 1973	Vertical Perspex tube	Hot coil or electric sparks	As received, then sieved down to 1400 um (dried and further sieving if no ignition, up to 25 um)	Visual observation of flame detaching from ignition source
Modified from original Lutolf, 1971 (then Bartknecht, 1978)	Modified Hartmann tube	Electric spark, hot coil (up to 1000 °C)	NS	Visual observation of flame
Kuhner explosibility test	20 l Siwek sphere	2 kJ chemical igniters	Concentration range	Overpressure greater than 0.3 bar
ASTM E 1226:2012	20 l Siwek sphere	5-10 kJ chemical igniters	Concentration range	Overpressure greater than threshold value
Norway	Open vertical mild steel tube	Acetylene welding torch	The amount of powder and the dispersion air pressure are varied to produce optimal conditions for ignition	Visual observation on flame length, color and appearance violence
Classification test Group A/B (UK, Explosion Hazard Testing Ltd), Commercial test	Hartmann tube, G-G furnace, 20 l Siwek sphere	Electric spark, hot coil, hot ambiance, low energy chemical igniters.	Concentration range variation according to results	Visual observation of flame propagation and overpass of overpressure threshold
ADINEX screening test, Commercial test	Hartmann modified tube	Temporary discharge spark(10 J energy), confirmation with 2 kJ igniters in 20 l sphere	Concentration range from 30 g/m3 to 2500 g/m3.	Visual observation of flame propagation and overpass of overpressure threshold
Chilworth, Commercial test	Hartmann modified tube, G-G furnace	Electric continuos arc (10 kV), hot environment (1000°C)	Concentration range variation according to results, with further sieving and drying if no ignition occur.	Visual observation of flame propagation

4.2 The Speditive Explosibility Test

4.2.1 Testing philosophy

The aim of this work is to define a speditive, low cost and reliable esplosibility test for dusts, whose results are aligned with the standard tests described in the technical standards.

The philosophy that stands behind such a test is related to the concept of ignition probability, described in Chapter 3. The test have to be speditive, but its results should lay on a strong statistical basis that grant the reliability of them. Indeed a dust sample that is classified as Non explosible by the test must have passed a sufficient number of attempts in which no ignition has occurred, being this number defined a priori on statistical considerations.

Furthermore the test conditions must be "as worst as possible", thus meaning that each procedure has to maximize the strength of the ignition source. This is required not to have false negative in the explosibility classification.

To this purpose, an ad hoc test procedure defined "Speditive Esplosibility Test (SET) composed of four different tests was developed. The SET procedure comprehend two tests in a modified Hartmann tube, a test in a Goodbert Greenwald Furnace and a test on a hot plate.

All these tests are done at conditions that maximize the ignition probability. Continuous arc and glowing wire are used in the Hartmann tube, temperature high as 800 °C and 400°C are used, respectively, in the GG furnace and in the hot plate test.

Several dusts having different origin but all from the process industry were submitted to SET and to traditional tests in the 20L sphere to compare the results. In this case the sample reactivity and eventually the k_{st} indexes of the violence of explosion were measured according to UNI EN 14034 standard.

The k_{st} measurement is not included in the SET, because the actual aim of the test is not to evaluate some single parameters among those considered when the explosibility of a dust sample is regarded, but to assess its ability to provoke an explosion hazard.

Nevertheless, all of the dust samples analyzed were tested also in the sphere, both using 2*5kJ igniters and 2*1kJ igniters (Simex manufacturer) to compare the SET results with those from standard (UNI EN 14034) tests and to check for explosion overdriving.

4.2.2 Experiments

Twenty-two dust samples supplied by different process industries were collected and submitted to SET and to 20-L sphere tests. Pretreatment and test methods are described below. As a rule only the fraction passing through a 500 um opening sieve was submitted to the test.

Each sample, as collected from industry, was characterized by the measure of the particle size distribution (PSD) and the moisture content. Test procedures are applied according to standards or to laboratory internal methodologies (Table 11).

Table 11: Characterization tests.

Parameter	Standard/Methodology	Unit of measure
Moisture	ISO 562:2010	Weight %
PSD by Sieving	Internal procedure 08/2014	Weight %
PSD by Laser granulometry	ISO 13320:2009	Weight percentiles
BZ flammability class	VDI 2263-1	-

PSD by laser granulometry was measured with a Malvern laser Particle Size Analyzer (Mastersizer 2000E), with a dry powder disperser unit (Scirocco 2000M). The measurement range of the instrument is 0.01-1000 um. The measurement procedures were carefully calibrated, as the feed rate and air pressure dispersion are significant operative variables when this kind of disperser is used. As a general rule each sample was first sieved and the fraction passing the 500 ▯m was used for Laser PSD measurement.

PSD by Sieving was obtained using a pile of standard sieves: 2000 um (ASTM n°10), 1000 um (ASTM n° 18) and 500 um (ASTM n°35), 250 um (ASTM n° 60), 200 um, 125 um (ASTM ° 120), 75 um (ASTM n°), 63 um (ASTM n° 230) and 32 um.

An amount of about 10÷15 g of sample is placed on the larger openings sieve and piled with the others below. A sieving machine (IG3/WET by Giuliani, Torino) operates the pile of sieves for 10 minutes. PSD was obtained by weighting under-sieve and over-sieve amounts recovered from the pile of sieves:

In case of "non traditional" dusts (as defined by Amyotte, 2012) no dry sieving (as described above) or laser PSD were possible. In those cases, wet sieving was necessary. In this case an adequate amount of dust sample is placed on the top of the sieve pile. Sample is flushed with water and gently moved. The sample fractions are collected from the sieve pile and dried in a laboratory stove at 65° C for at least 12 hours. The PSD is obtained by weighting. Samples treated by wet sieving are number 14 to 17.

Any sample having a moisture content higher than 15%, it was submitted to SET both "as received" and conditioned for moisture content. To this purpose, samples were dried in a laboratory stove at 65°C for 5 hours.

SET is conceptually divided in two parts, named "room temperature" and "high temperature" tests, as described in Table 12. SET starts with test N° 1, and stops at the first ignition. In case of no ignition in test 1, SET is continued to test 2 and so on.

Table 12:SET test structure.

Test N°	Test device	Ignition method	Classification
1	Hartmann Tube, open	Continuous arc	Room temperature
2	Hartmann Tube, open	Glowing wire	Room temperature
3	GG furnace	Autoignition, 800°C	High temperature
4	Hot plate	Autoignition, 400°C	High temperature

4.2.3   Room Temperature Test

Hartmann 1.2-L tube is used for room temperature tests (1 and 2). Test conditions and procedure are, as close as possible, those described by the UNI EN 13821 standard to measure the Minimum Ignition Energy (MIE). The aim of this test is to evaluate the ignition sensitivity of the dust sample to ignition source with relative high energy (as worst as possible conditions). Dust cloud is generated at room temperature.

Each sample is first submitted to continuous arc ignition attempt (test N° 1). After UNI EN 13821, the arc is generated between two Tungsten electrodes having a 6 mm gap, placed at 1/3 of the tube height from the bottom. The sample is placed at the tube bottom close to the mushroom air disperser. An air blast delivered from a 50 ml reservoir (7 ATE) is used to create the dust cloud. The arc characteristics are 29 kV (maximum at dielectric breakage) and 0.75 mA.

Each dust sample is tested at different concentrations: 0.3, 0.5 and 1 grams are dispersed into the Hartmann tube. The test is repeated 10 times at each concentration unless ignition is observed. As per UNI EN 13821, no more than three attempt are made with the same dust amount.

The evidence of the ignition is the presence of a flame propagating from the ignition point (space between the electrode) to the top of the Hartmann tube. According to EN 13821, the ignition has occurred if a flame greater than 5 cm is seen in the test.

In case of no ignition in all the electric arc tests, the sample is submitted to the same procedure using a glowing wire as ignition source. The glowing wire is located at the same height as the electrodes. A continuous current of 11.8 V and 13.1 A is used to make the wire glow.

When one ignition is observed the test is interrupted as the sample is classified as "explosible at ambient temperature (EA)".
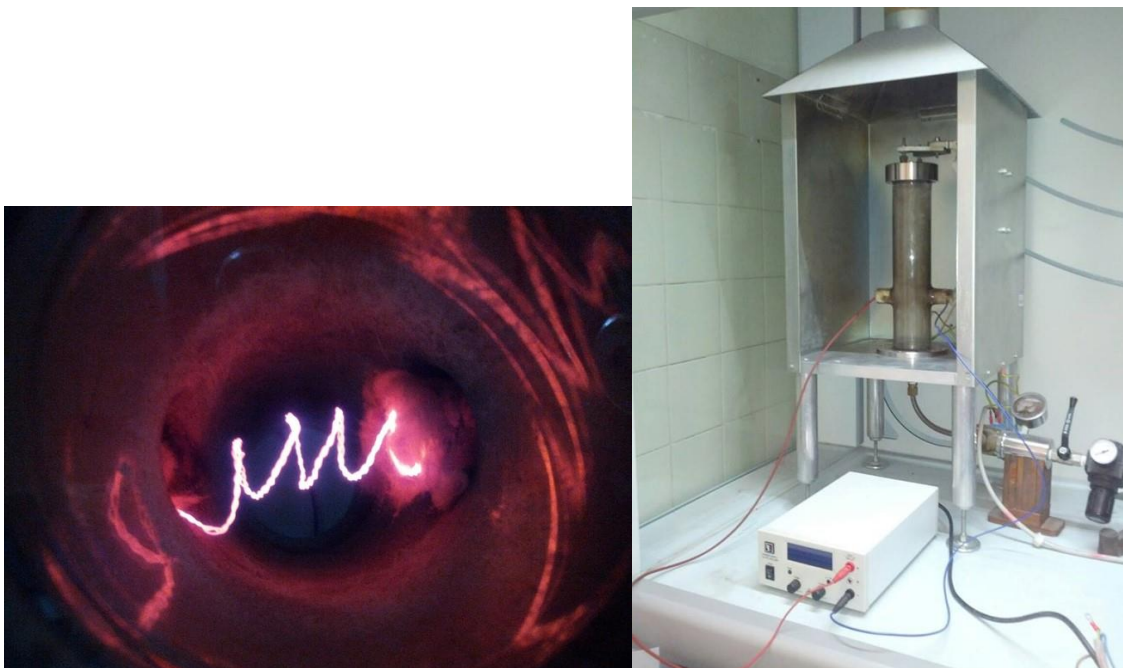

Figure 23: Glowing wire detail (left-side) and Hartmann modified tube (right-side) for the ambient temperature tests.

4.2.4 High temperature Test

In case of no ignition in room temperature tests, samples are submitted to "High temperature tests". These are derived from the cloud Autoignition Temperature test and the layer Autoignition Temperature tests described in UNI EN 50281:1999 standard. The apparatus used are the Godbert-Greenwald furnace, and the hot plate device, both normalized by UNI EN 50281:1999 standard.

Again the "as worst as possible" ignition conditions are assumed: the temperature in the GG furnace is 800°C and the temperature of the hot plate is 400 °C (both are maximum operating set up of the apparatuses).

The first test examine the hazards related to dust cloud dispersed in air. Each dust sample is tested at different concentrations obtained by injecting different amounts of dust in the furnace (0.1, 0.3 and 0.5 grams). The air pressure in the blowing reservoir is set at 0.25 bar.

The test is continued up to ignition or at minimum 10 attempts are made at each sample amount. Again, ignition is assessed when flame propagates outside the furnace bottom. Sparks is not considered as proof of explosion.

In case of no ignition, the sample is tested for layer autoignition on the hot plate device to assess the sensitivity of dust sample when placed in a layer. An adequate amount of dust is weighed in order to fill the metal ring used to form the layer on the plate. The ring height is 5 mm, according to UNI EN 50281:1999 standard for the MIT layer measurement.

The evidence of an ignition is set as the same described in the ASTM E2021-2006 (4.3), considered more conservative with respect to the UNI EN standard.

Unless any ignition is observed, the test is repeated three times.

In case of ignition during a "High temperature test" the sample is defined as "explosible at high temperature" (EH).
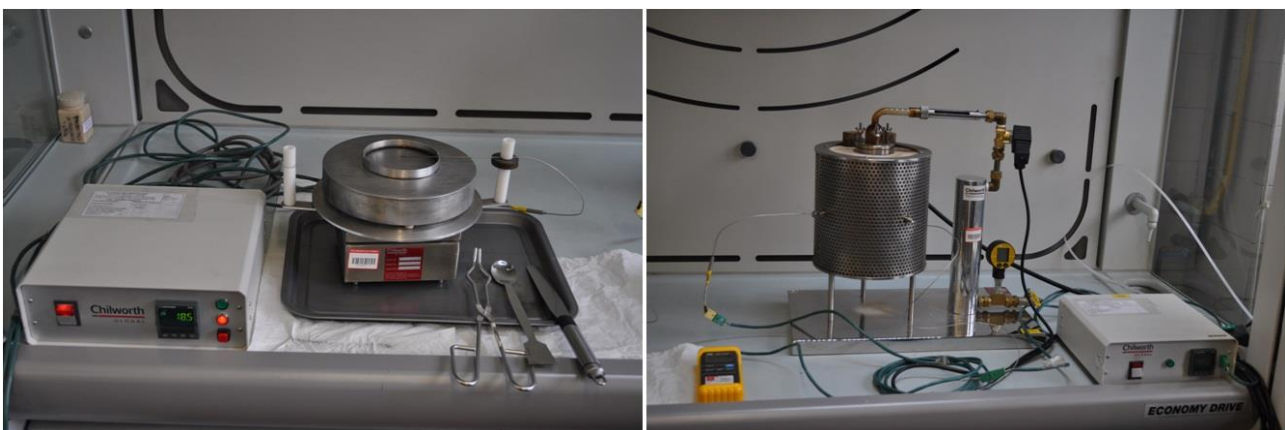

Figure 24: Hot plate device and G-G furnace for the high temperature tests.

4.2.5 Tests in 20 l Siwek Sphere

All the samples were submitted to explosion tests in a 20l sphere with the aim to measure the kst. A wide range of concentrations were tested (generally starting from 250 g/m3, the maximum

depending on the sample nature) for each sample. Both 1 kJ and 5 kJ chemical igniters were used (manufactured by Simex control) to verify possible overdriving effects on the "weakest" samples.

The test procedure is the same as the standard (UNI EN 14034), unless non-traditional dusts are handled. In this case, the samples were not dispersible through the injection valve of the sphere, resulting in poor reproducibility of the measures.

For this reason, the total amount of the sample was directly introduced into the sphere, paying careful attention to the fact that the dust distributes all around the rebound nozzle as to obtain a homogeneous dispersion by the air blast.

The 1 kJ tests have the precise aim to assess if a "false positive" as consequence of explosion overdriving has occurred during tests with the 5kJ igniters.

When using 1kJ igniters, the threshold for an ignition is the same adopted in the Kuhner explosibility test, i.e. when values of overpressure pm and P_{ex} are respectively greater than 0.2 bar and 0.5 bar.
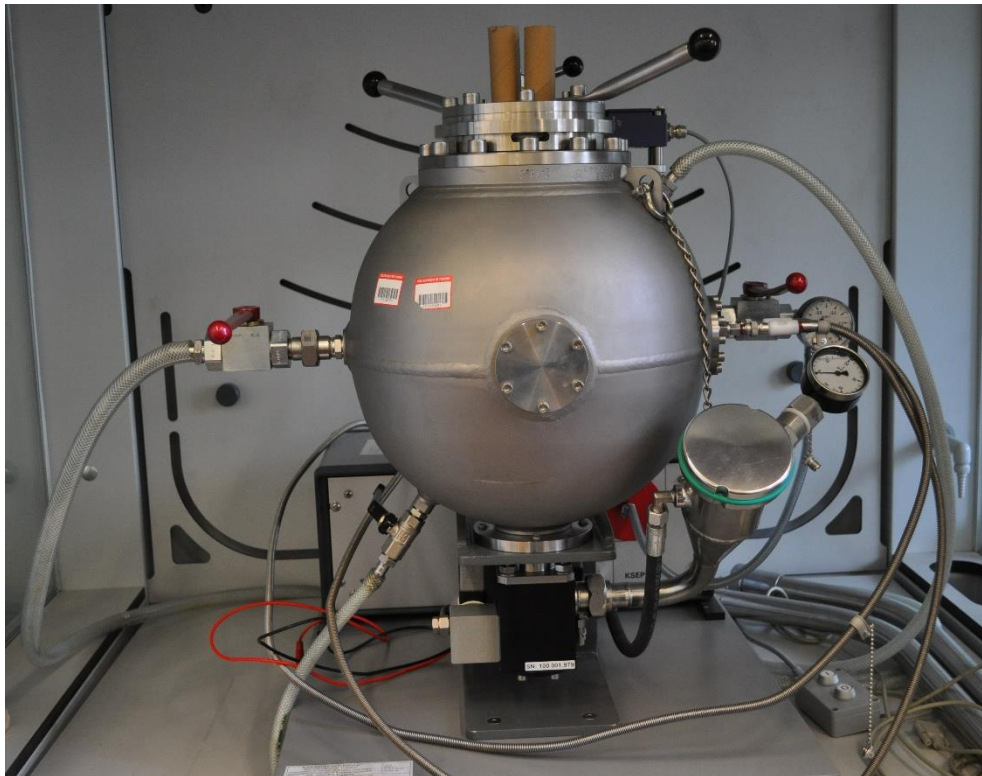
Figure 25: Siwek 20 L sphere operating an explosion parameters measurement.

Figure 26: Detail of the couple of 5 kJ chemical igniters.

4.2.6 Materials

Dust samples in industrial context are rarely pure elements: the great majority consists of mixture of different materials or chemicals, depending on the type of manufacturing process from which materials derive. In many situations, the sample could be a mixture of different dusts, often inert – combustible dust mixtures can be generated by processes including some thermal treatment such as laser cutting of metallic artifacts.

This study deals mainly with these types of samples, that are the more representative of the process industry, but also the more challenging to examine, as scientific fundaments on dust mixture behavior are not yet well understood and several works are in progress with this aim in the academic and research context all-around the world. The screening test purpose is also to try to fill this lack of knowledge of actual industrial dust samples behavior to ignition.

The samples studied here are representative of different Italian industrial realities. Their nature is reported in Fig.27, the Table 13 resume sample characterization (including industrial source and type of process).
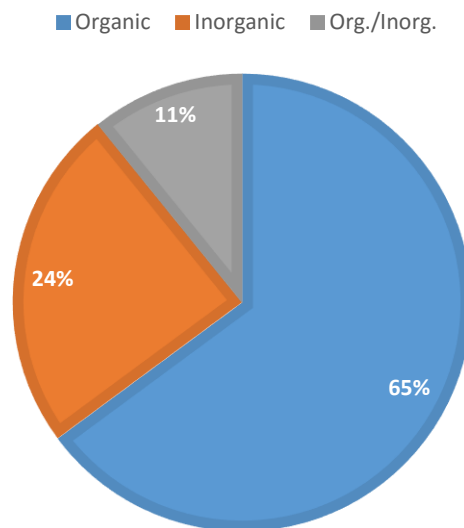

Fig.27: *Classification of dust samples.*

Most of the samples came from engineering industry, other from the textile industry. Almost all the samples are waste materials collected from abatement devices or from the workplace. One sample (N° 1) was a mixture of Cellulose Acetate (1b in Table 14) and Bentonite (1a in Table 14) at different weight ratios. Four samples derive from textile processes and have a strong fibrous nature.

Table 13:Features of the samples submitted to SET.

Sample	Source	Aspect	Peculiarity
1	Dust mixture cellulose-ether and bentonite, at different concentrations, processed for packing production	Pure elements, cellulose is white, while bentonite grey-like	
2	Waste dust from processing of steel screw and joints	Mainly constituted by calamine, gray aspect.	High dustability and tendency to stain
3	Waste dust from an industrial silo, from plastic laminates production. Organic nature of sample.	Ochre colored, with persistent smell. Little plastic shaving present in it	Low grade of adherence to surfaces and easy to disperse
4	Waste dust from mechanical workshop. Sample collected in proximity of machineries processing wood artifacts. Organic sample.	Light yellow colored, similar to sawdust.	Homogenous in particle sizes
5	Wood dust used for feeding of a furnace in a concrete production site.	Sample constituted by a finer fraction and a rough fraction with shavings up to 1 mm length.	
6	Sample collected from waste of a milling machinery in a polymeric resins model production site (organic)	Ochre colored. Wide particle size ranged, persistent smell.	High tendency to cumulate electrostatic charge and adherence to container walls, difficult to collect.
7	Waste dust from remaining from a laser cutting machine of metallic elements.	Burgundy-like color, poor smell, similar to varnish..	High persistence on objects and tissues, tendency to stain
8	Metallic nature dust sample from remaining from grinding machines	Grey color, poor smell	Very fine dust, scarce tendency to agglomerate
9	Metallic nature dust sample from remaining from welding process	Dark brown color, persistent and acre smell	
10	Waste dust from blasting process of aluminum artifacts	Dark color, mainly constituted by finer than 500 um particles.	
11	Sample dust from suction filter operating on grinding - sandblasting machineries	Dark color, grey like, high fine fraction.	
12	Dust sample processed for artifacts production form recycled material (PET)	Light blue color, very fine particles.	
13	Metallic dust sample from traction tools production unit (bearings, brakes)	Dark color, with acre smell	High persistency on objects surfaces, mainly fine particles with a little fraction of shavings up to 1 mm length.
14	Dust sample from waste of industrial textile processing	Fibrous, high fraction of fine particles, grey.	
15	Dust sample from waste of industrial textile processing	Fibrous, high fraction of fine particles, black	
16	Dust sample from waste of industrial textile processing	Fibrous constituted by finer particles and greater fibers of ca. 2 cm length, grey	

17	Dust sample from waste of industrial textile processing	Fibrous, scarce presence of fine particles, tendency to agglomerate in flocks, black
18	Sample dust from bag filter of metallurgic industrial site	Silver color
19	Sample from waste of foundry operations – sanding machine	Dark color, high percentage of fines
20	Sample from waste of foundry operation- smearing machine	Dark color, high percentage of fines

Samples PSD and moisture are presented in Table 14. They are characterized by the percentiles of the PSD in case of laser diffraction measurement, otherwise the weight fraction is adopted. It is evident as the majority of the samples contain a significant amount of small particles. All the samples present a wide PSD, samples 14, 15, 16 and 17 have a fibrous nature.

Table 14: PSD by laser diffraction and sieving for all the samples tested in SET.

| Sample	PSD, – Laser diffraction (um)			PSD-Sieving (%w)				Moisture (%w)
	d(0.1)	d(0.5)	d(0.9)	<2000	1000<d<2000	500<d<1000	<500	
1a	3.54	13.10	37.98					
1b	106.15	271.77	523.89					
2	10.42	65.23	102.80	0.54	0.62	1.36	97.5	<0.05
3	54,62	193.99	523,56					
4	36,97	159.08	407,39					
5	NE	NE	NE	3.5	9.5	23.4	63.6	5.2
6	36,50	170.76	489,85					
7	1,32	4.84	87,17	0.02	0.16	0.28	99.53	0.25
8	2,21	15.61	96,65	0.43	0.08	0.16	99.33	0.82
9	1,03	5.04	150,60	8.77	6.65	4.39	80.19	1.7
10	7.83	45.56	231.32	<0.01	0.12	0.39	99.49	0.08
11	4.69	34.20	117.22	0.26	0.09	0.32	99.33	0.17
12	18.10	51.50	128.99	0.8	0.8	0.4	98.4	0.81
13	7,17	81.75	320,53	0.3	0.5	0.7	98.5	0.83
14	3.06	13.71	261.49	34.7	0.48	1.38	63.5	7.1
15	NE	NE	NE	44.4	0.9	5.0	49.6	4.76
16	4.88	39.80	350.49	69.3	0.63	1.67	28.4	5.0
17	NE	NE	NE	<0.1	<0.1	<0.1	>99.9	5.0
18	7.59	102.49	342.54	0.19	1.04	8.16	90.1	6.7
19	26.76	207.34	549.34	<0.1	0.13	5.95	93.8	1.04
20	11.44	140.54	434.34	0.59	0.08	1.00	98.3	0.06
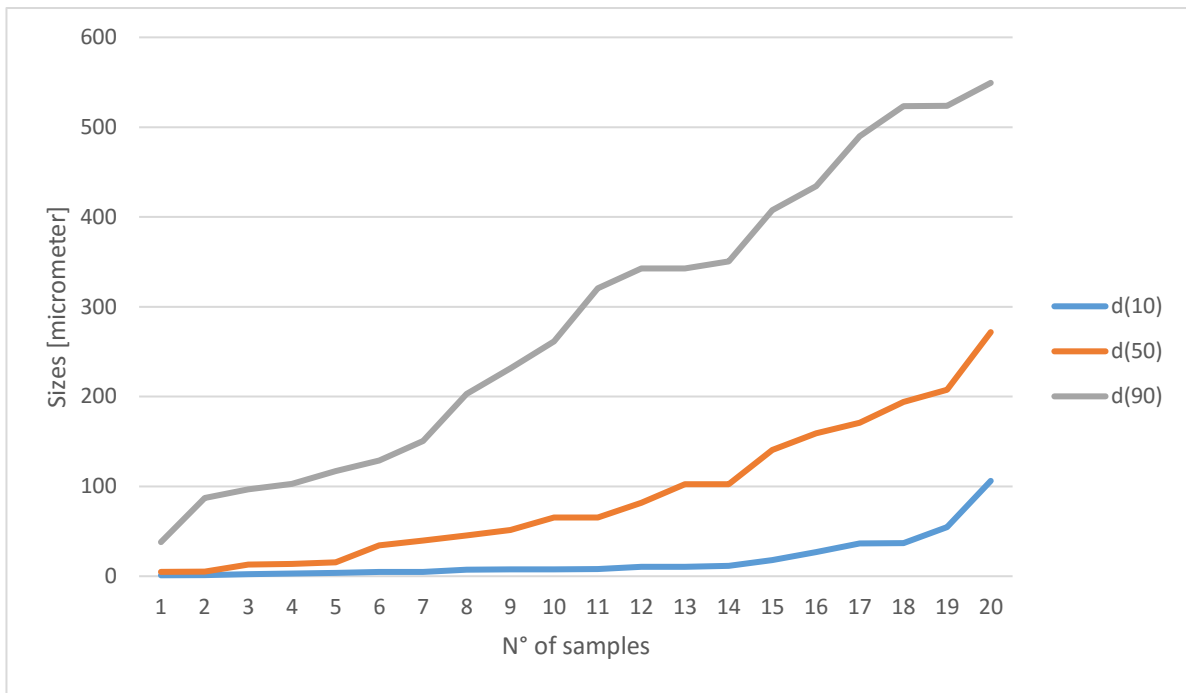
Figure 28: Quantiles distribution versus the amount of samples.

The majority of the dust samples examined by Laser Diffraction were constituted by fine particles: all the samples have a d(10) smaller than 100 um. The exceptions are counted among the samples deriving from textile industrial wastes, that have a fraction of particles bigger than 2000 um greater than 35% in weight (samples 14, 15 and 16 in Table 14).

Dust morphology was observed through microscopy: the majority of samples' particles could be defined to be sphere-like, while textile dust samples are constituted by fiber-like particles, as is shown in the microscopic image of Figure 45

No sample is found to have a moisture content greater than 15 %, being the limit at which internal laboratory procedures adopted suggest to dry the samples in stove.
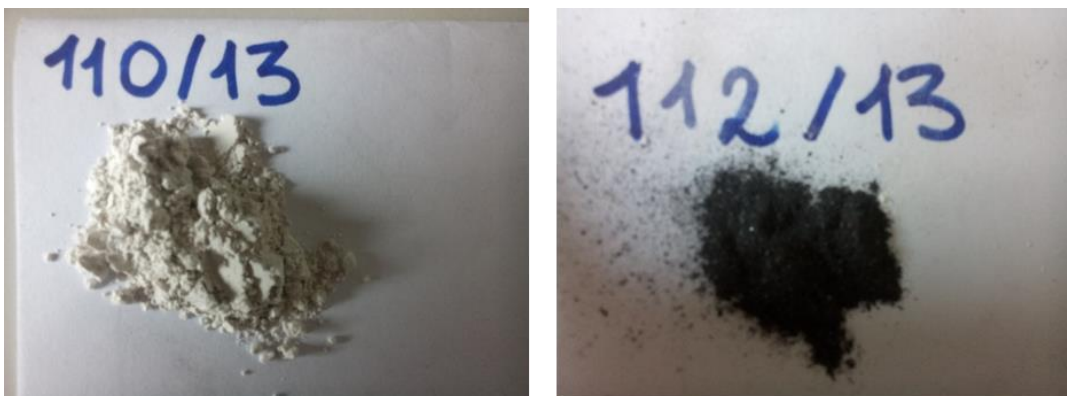
Figure 29: Left side: Sample #1, Right side: sample #2
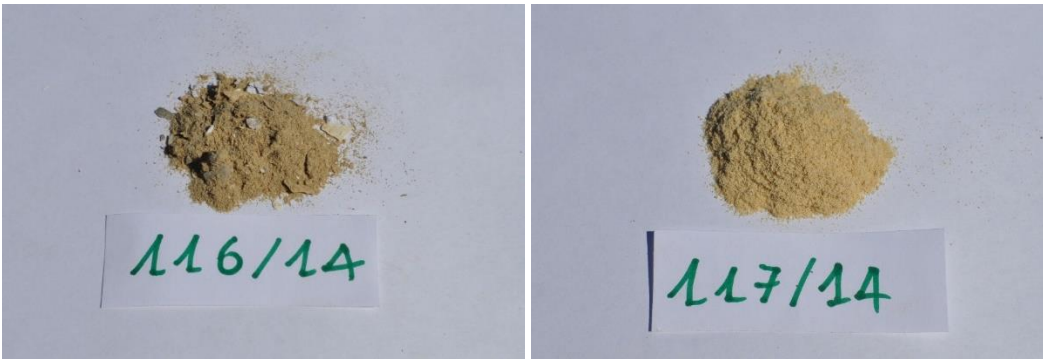
Figure 30: Left side: Sample #3, Right side: sample #4
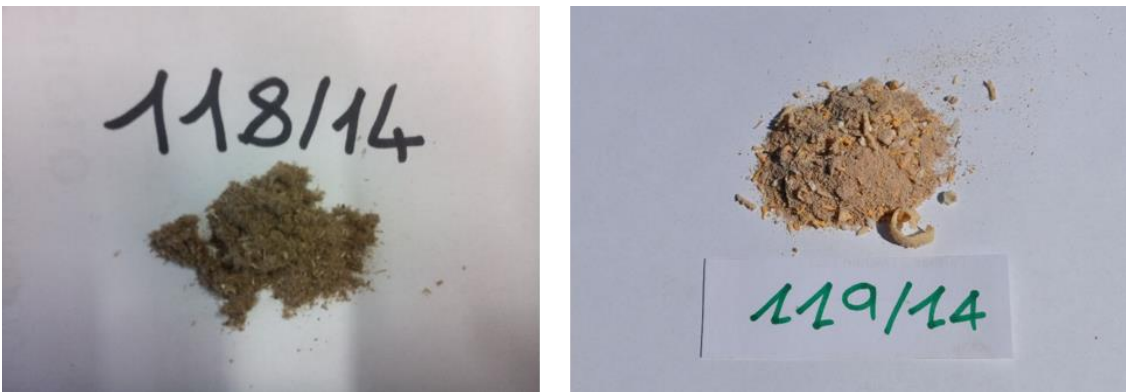
Figure 31: Left side: Sample #5, Right side: sample #6
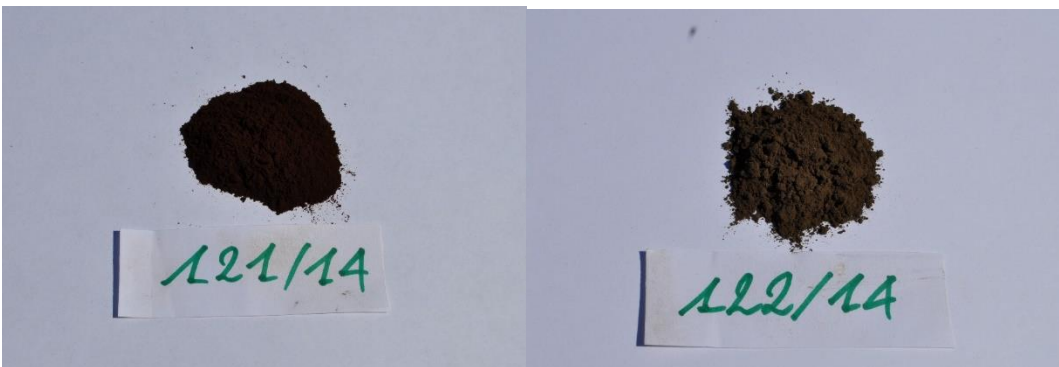
Figure 32: Upper Left: Sample #7, Upper Right: sample #8

Figure 33: Upper Left: Sample #9, Upper Right: sample #10
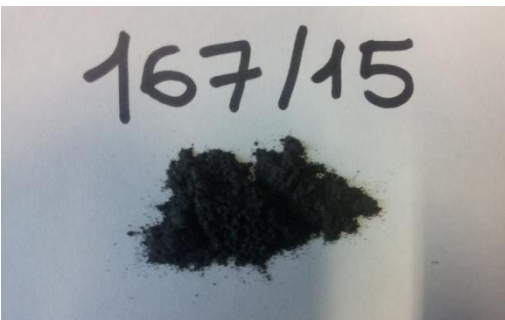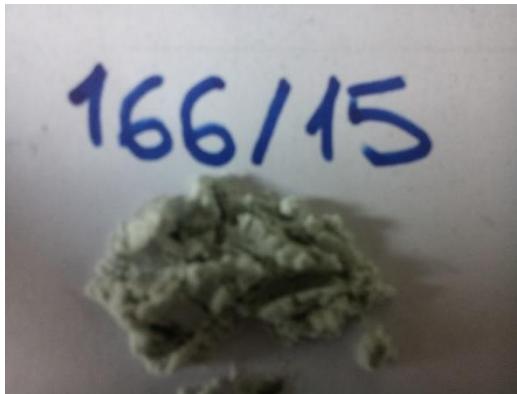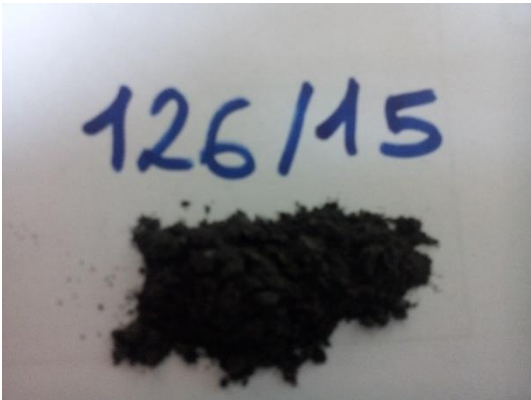
Figure 34: Upper Left: Sample #11, Upper Right: sample #12, Bottom left: sample #13

Figure 35: Left: Sample #14, Right: sample #15

Figure 36: Left: Sample #16, Upper Right: sample #17

Figure 37: Left: Sample #18, Upper Right: sample #19

Figure 38: Sample #20.

Table 15: Summary results of SET, where O: organic sample, N: inorganic sample, M: mixture of both. Y: positive result, N: negative result. NE: test non executed.

Sample	Nature	AT Test	HT Test	SET class	10 KJ	2 KJ
1b	O	Y	NE	EA	Y	Y
1c	M	Y	NE	EA	N	N
1d	M	N	Y	EH	N	NE
2	N	N	Y	EH	N	NE
3	O	Y	NE	EA	Y	NE
4	O	Y	NE	EA	Y	NE
5	O	Y	NE	EA	Y	NE
6	O	Y	NE	EA	Y	NE
7	N	N	N	Nexp	N	N
8	N	N	Y	EH	Y	N
9	N	Y	NE	EA	Y	NE
10	N	Y	NE	EA	Y	NE
11	N	Y	NE	EA	Y	NE
12	O	Y	NE	EA	Y	NE
13	N	N	Y	EH	Y	N
14	O	Y	NE	EA	Y	NE
15	O	Y	NE	EA	Y	NE
16	O	Y	NE	EA	Y	NE
17	O	N	ND	Nexp	Y	NE
18	N	N	Y	EH	N	N
19	N	N	Y	EH	-	Y
20	N	N	Y	EH	-	Y
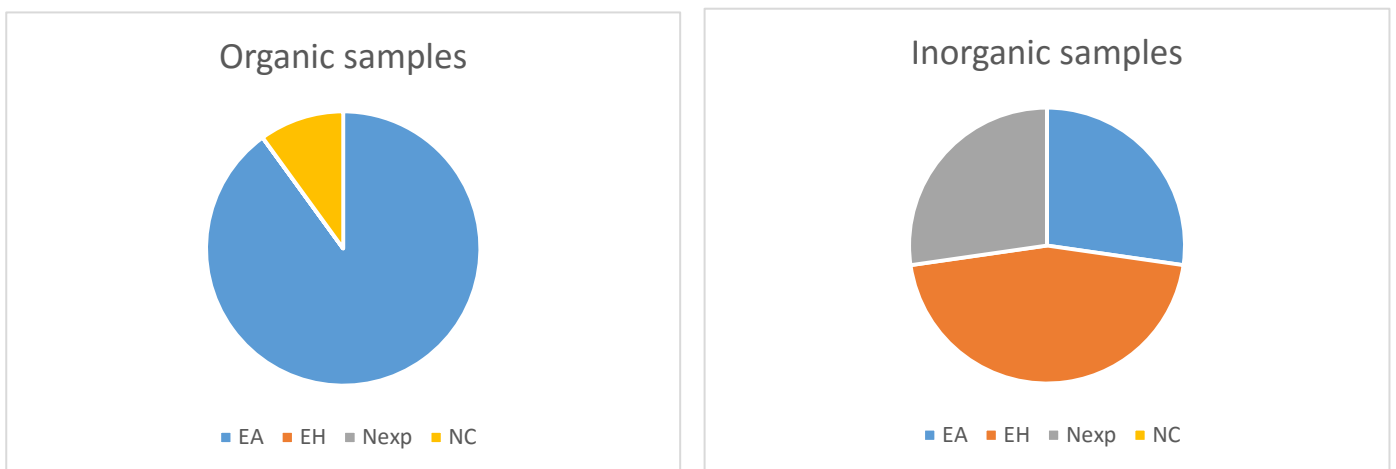
Figure 39: Summary of results according to nature of dust samples.

Table 16: Detailed results of SET. Where: N: no ignition, P: ignition. NE: test non executed, ND: sample non dispersible. (I/0.1): n° of attempt and sample weight at which ignition occur.

Sample	Room temperature Tests		High temperature tests		SET class	20l Siwekspheretests			
	Continuou sarc	Glowin gwire	G-G furnace	Hot plate	-	Pmax (bar)	$(dP/dt)_{max}$ (bar*s^{-1})	kst (bar·m·s^{-1})	St Class
1b	N	P (I/0,5)	NE	NE	EA	6.3	253	69	1
1c	N	P (II/0,5)	NE	NE	EA	0	0	0	0
1d	N	N	P (I/0,3)	NE	EH	0	0	0	0
2	N	N	P (I/0,3)	P	EH	0	0	0	0
3	N	P (III/1,0)	NE	NE	EA	6.8	314	85	1
4	N	P (I/0,3)	NE	NE	EA	7,4	365	99	1
5	N	P (I/0,3)	NE	NE	EA	7,6	284	114	1
6	P (II/0,5)	NE	NE	NE	EA	7,0	250	67	1
7	N	N	N	N	NE	0	0	0	0
8	N	N	P (I/0,3)	NE	EH	5,7	197	53	1
9	P (I/1,0)	NE	NE	NE	EA	5,9	349	94	1
10	N	P (III/0,3)	NE	NE	EA	3,6	450	122	1
11	N	P (I/1,0)	NE	NE	EA	2,5	75	20	1
12	P (I/0,3)	NE	NE	NE	EA	6,3	526	142	1
13	N	N	N	P (II)	EH	3,8	49	13	1
14	P (II/0,3)	NE	NE	NE	EA	7,0	390	105	1
15	P (IV/0,3)	NE	NE	NE	EA	6,1	164	44	1
16	P (II/0,3)	NE	NE	NE	EA	6,3	183	49	1
17	N	N	ND	NE	NE	6,1	93	25	1
18	N	N	P (I/0,3)	NE	EH	0	0	0	0
19	N	N	P (III/1.0)	NE	-	-	-	-	-
20	N	N	P (III/1.0)	NE	-	-	-	-	-

3.4.1 Validation test in the 20-L Siwek sphere

Results of the SET are presented in Table 16. The majority of the samples were classified as "explosible at ambient temperature". Among these, all but sample 1c exhibited a measurable k_{st} in the 20 L sphere operated with a couple of 5kJ igniters. The same samples exhibited a measurable explosion in the 20 L sphere operated with a couple of 1 kJ igniters.

Sample 1c did not exhibit any explosion in the 20 l sphere nor with 5kJ or 1kJ igniters. In both cases P_m and P_{ex}, although being measurable, did not meet the criteria to assess the explosion. The reason for this behavior is still not clear. The evident difference of PSD between Bentonite and Cellulose Acetate may suggest a certain mixture classification most probably in the Hartmann Tube tests which could lead to create a cloud richer than expected in the flammable component, so leading to ignition.

Two samples were classified as "Not Explosible" at the SET. One of these, sample #7, was a dust from laser cutting of metallic artifacts. It is reasonable to suppose that the material was inert as it was oxidized by the high temperature of the laser beam.

The other "Not Explosible", Sample 17, yielded a measurable kst of 25 bar/ms-1, and resulted explosible as well in the test conducted using the 1kJ couple of igniters. This discrepancy can be explained with considering the highly fibrous nature of the sample, which demonstrated a scarce dustability. This behavior is evident in the Hartmann tube, where it was not properly dispersed by the air blast. The same in the GG furnace. In this case, the sample stuck into the injection system, such that it was not possible to test the dust. It is reasonable to assume that the most powerful air blow in the 20 l sphere overcome these problems creating an explosible cloud.

Samples 1d, 2, 8 13 and 18 were classified as "Flammable at High temperature" after the SET test. Among these samples 1d, 2 and 18 did not exhibit a measurable k_{st} with the 5kJ igniters, such to result not explosible with traditional tests.

The other way around, samples 8 and 13 tested in the 20 L sphere with two 5kJ igniters resulted in a weak, but measurable k_{st} equal to 53 and 13 bar/ms^{-1} respectively. The same samples submitted to the 20 L sphere test with a couple of 1kJ igniters did not match the criteria for explosion assessment. Therefore, according to standard tests, these samples would be characterized by a k_{st}but it would not be possible to measure their Lower Explosible concentration. This result could drive the conclusion that samples 8 and 13 did undergo to explosion overdriving in standard 20Ltest for k_{st} measurement.

Samples 19 and 20 also showed a different behavior with respect to other samples of the some nature (i.e. inorganic)

Both were classified as Explosible at High temperature with the SET procedure. The validation was done with 2X1 kJ igniters in the Siwek sphere. An evidence of explosion (overpressure greater than 0.5 bar, a) occurred with both the samples with a relative high concentration of 3000 g/m3. No tests were made for these two samples with 5kJ igniters, consequently they are not St classified.

This concentration was close to the injection valve-working limit, in fact values of p_d (expansion pressure) lower than expected were measured by the pressure sensors.

In order to assess the real behavior of those two dust samples a test in the 1 m^3 chamber of explosion is suggested, here not performed due to absence of adequate equipment).

Testing "Non traditional" dusts

The results of the SET, shown in the previous sections of this work, have underlined the problematics related to materials that could not be dispersed efficiently through the traditional injection systems for dust explosion testing, also present in the SET setup.

In particular, the dispersion units on board of the Hartmann tube and the Godbert-Greenwald furnace tend to fail in their attempt of dust dispersion, when non-spherical particles dust are to be tested. The problem is related to the air blow generated by the reservoir on board of the machine (G-G furnace) and to the injection solenoid valve that opens to convey the air on the dust-charging chamber.

This problem of dipersibility in testing equipment is related to the characteristic size of the dust particles: increasing the diameter decreases the attitude of the fibers to be dispersed. The fibers demonstrated a strong attitude to coagulate into spherical aggregates with greater diameter. The longer the fibers, the stronger the attitude

This part of the Thesis' work is an application of a explosion hazard evaluation on non-traditional dusts. Since few database exist up to now in literature, the present one could constitute a starting pint for increasing the awareness on the hazards related to those type of materials.

5.1   The concept of dustiness

The concept of dustiness, the tendency of a dust to be dispersed and stay airborne (according to VDI 2263, part 9) has been only in recent times included in those safety parameters that characterize a dust susceptible of ignition.

Actually explosive dust-air mixtures occur due to dispersion of dust layer, conveying of dust or filling up of containers. Dust clouds generated in these processes are usually not homogeneous. Since dustiness is not a physical value of the dust, but it is influenced by several variables, to develop a standard measurement method is not an easy task. The standard method presented by VDI 2263-9 intend to assess the likelihood of dust clouds generation of a certain dust sample after free falling or chute.

There is no standard procedure to assess the attitude of a dust to generate a cloud by lifting of dust layers through airflows, whereas this is the mechanism of secondary explosions. The concept of dustiness or dispersibility of dust is not new but recently new research development was provided by some authors (Klippel et al., 2013 and 2015

The major unknown dealing with secondary explosion is related to the easiness (or difficulty) of dispersion in air of the combustible dust, a detailed study in perspective will help to develop new regulations or guidelines to measure the dustiness of dust layers and to assess the risk of secondary explosions.
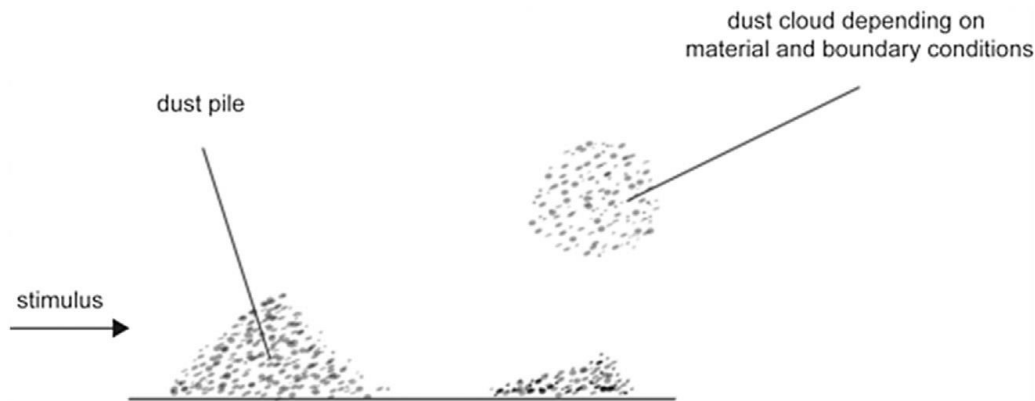
Figure 40: Mechanism of dust pile or layer lifting by an air blast, from Echkoff (2003).

At present, our research has focused on the behavior of non-traditional dusts and on their low tendency to be dispersed to form clouds, once settled to the ground. The dynamics of explosion involving those type of combustible are relatively unknown and very scarce literature exists nowadays on this problematics; nevertheless, hazards related to non-traditional dusts are real and this research could increase the knowledge in this field and promote the buildup of a more conscious safety culture on the matter.

Few studies had been performed on the explosibility of fibers, as textile dusts were recently studied (Amyotte, 2012, Marmo & Cavallero, 2008 and Iarossi et al., 2013) but no literature is present on the dustiness of fibrous textile dust all around the world.

Such fibers can be composed of a multitude of materials, including cotton, acrylic, and polyester, and have a multitude of uses such as upholstery, carpeting, toys, paper, noise reduction, and insulation materials. Flocculent materials would not be considered as dusts according to the NFPA definition as "*finely divided solid with a diameter of less than 420 µm (0.017 in.). A dust will pass through a US No. 40 standard sieve.*"

However, flocculent materials may be considered dusts, though "nontraditional", given the NFPA definition of a combustible dust. Under this definition, a combustible dust is any combustible particulate solid that when suspended in air poses a risk of fire or deflagration. This definition gives no restriction on the shape or size of the particle.

Recently NFPA published a standard document that include under the definition of combustible dust "*Flat platelet-shaped particles, flakes, or fibers with lengths that are large compared to their diameter...usually do not pass through a 500 µm sieve*" if they actually represent a deflagration hazard. NFPA 652 conclude that a combustible dust is "*a finely divided combustible solid particulate that present a flash fire hazard or explosion hazard when suspended in air or the process specific oxidizing medium over a range of concentration*".

In this, combustible solid particulate is defined as "any solid material composed of different particles or pieces, regardless of size, shape and chemical composition, that when processed, stored or handled in the facility has the potential to produce a combustible dust".

5.2   Textile industry dusts

Whereas Textile industry is generally not considered to be at high risk of dust explosions, it has encountered in the past some episodes worldwide. Marmoet. Al. (2010) described a flock explosion occurred in a facility that produced nylon-based threads. Piccinini (2008) and Salatino et.al. (2012) described the same episode, a powerful explosion occurred in a wool manufacturing.

The detailed accident list provided by Abbasi (2007) includes three episodes of dust explosions involving textile samples worldwide, among these one with relevant consequences (57 deaths and 177 injured in China, 1987). The percentage of textile-related accidents is anyway relative low with respect to other involved industrial realities (like food and feed chain or coal handling). This could be an explanation on the very low degree of consciousness of the risk of explosion due to fibers in, for example, those industrial comparts where they are commonly found, as is the case of the textile industry. Consequently, regulations on explosive atmosphere do not explicitly comply with the presence of combustible fibers.

Moreover the episodes reported by Abbasi (2007) deal with cotton or linen, but not with wool.

Wool is generally regarded as a flame-resistant material and adopted for fire fighters equipment due to its ignition temperature ($\sim$ 570 °C). Furthermore, once ignited, wool fibers display charring phenomena and behave as flame retardant.

This could constitute another reason for disregarding the risk associated to wool dust explosions and fires.

Recently many authors (among them Amyotte et.al. 2012) underlined that non-traditional dusts, such as fibers, are a potential hazard factor that affects the textile industry. Textile processes handle fibers, which are materials having specific morphology with one dimension being at least two orders of magnitude bigger than the others. Generally speaking, textile fibers are long cylinders having diameter in the range 10-1000 µm. Characterization of such type of dusts is often based upon the length-to-diameter ratio rather than on the diameter value alone.

Explosion likelihood of this type of dusts also increases with a diminishing particle size, as for spherical traditional dusts.

Minimum explosible concentration, minimum ignition energy, and minimum ignition temperature have all been shown to decrease with reduced particle size.

Same authors have found that the MIE of flocculent materials is generally higher when compared to values of spherical-shaped particle dusts. They explained this difference focusing on the length dimension of the fiber: when subjected to thermal operation fibers shorten and aggregate into a spherical shape, with greater diameter, decreasing in the meantime the degree of dispersion of the sample and consequently their tendency to ignite (higher MIE).

Flocculent materials therefore present explosion hazards similar to spherical dusts and should be considered as combustible dusts. Nylon flock ignition has been determined as the cause of at least one explosion in flock manufacturing plants in recent years. Marmo (2010) described an accident occurred in a flocking manufacturing plant in North-west of Italy, where cumulated flock layers in ducts and inside operation machines ignited and generated a secondary explosion, once entrained by the primary pressure shock wave.

Salatino et al. (2012) reported an incident in a wool factory, involving wool dust, occurred in 2001 in Italy. In this case the combustible materials was constituted by wool processing byproduct,

namely, noils and burr. Wool noils are short fibers removed in the processing of textile fibers, and burr consists of lumps or flakes of wool together with the vegetable fragments present on the sheep's fleece.

The explosion was triggered by the smouldering combustion of a cumulated layer of dusts that evolved in a flaming combustion and then caused the propagating explosion of the finer airborne dust particle dispersed along ducts and inside operation machines.
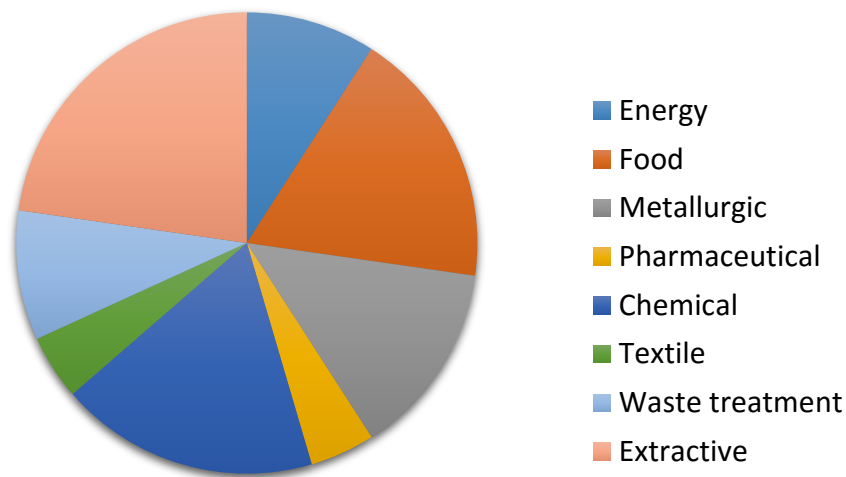

Figure 41: Statistical data on dust explosions according to industrial facilities, from 1986 to 2004, by Abbasi, 2007. Textile industry is reported as 4.5% of the total accidents episodes.

5.2.1 State of the art of non-traditional dusts explosion investigation

Flocculent materials are not being extensively studied and only recently authors seem to interested in this type of non traditional dusts.

A review by Amyotte et al. (2012) presented some aggregate results of investigation both on ignition sensitivity and explosion parameters of fibrous materials.

As for traditional dusts, the explosion severity increase with the decreasing of particle size. In particular, PSD exhibit greater influence on pressure rise rate than on maximum pressure value. Moreover Amyotte et al. (2011) revealed that P_{max} and k_{st} values are generally achieved for lower concentrations and with smaller particles.

The same authors have measured k_{st} and P_{max} values for polyethylene fibrous dusts and wood fibrous dusts; both the samples resulted classified as St 1 and present values of Pmax between 5 and 8 bar/ms^{-1}.

The non traditional dusts could be defined both for diameter or length values (or by length to diameter ratio). Amyotte et al. (2010) underlined the influence of both those characteristic sizes on the MIE determination, while the diameter is the controlling parameter (relatively smaller than the length).

The influence of this latter is anyway accounted in the MIE determination, mainly because during melting process, fibers tend to shorten and form spherical particle, with increased diameter and

because longer fibers contribute to increase the aggregation of the samples and reduce its dispersibility.

Nylon fibers were found to have similar explosion propertiesto organic dusts, with explosion severity generally falling into the St1 dust explosion class.

Marmo and Cavallero (2002) tested nylon flocks coming from an Italian manufacturer (diameter between 10 and 27.4 mm, length between 600 and 1800mm) in the modified Hartmann tube to obtain data on MIE of those kind of dusts. They found that the explosive characteristics of the nylon fibers (flock) were very similar to those of an explosible organic traditional dust. The MIE was as low as 145mJ. Both the diameter and the length of the fibers influence the MIE, as it increases as both dimensions decrease. As expected, the influence of the diameter is more obvious, since it is the controlling variable of the heat and mass transfer phenomena, as reported above by Amyotte's review (2011).

To date, the explosion risk associated to non traditional dusts, in the case of textile waste materials, is being investigated by some authors, but still scarce data on explosion parameters of those type of dusts exist. Frank (2004) have illustrated the importance on focusing on the explosibility of flocculent materials, while describing the Malden Mills explosion occurred in 1995. Van Pidoll has underlined the risks associated to ignition of flocculent textiles by electrical discharge originated in the flocking process itself (2001, 2002).

Furthermore, Piccinini (2008) and Salatino (2012) described a severe accident occurred in a wool factory in Italy.

A further need of data is required with the objective to build up standard equipment to measure explosion parameters of those specific kind of dusts that are not easily tested with the equipment for so defined traditional dusts. The work of this thesis could also be an attempt to raise the awareness on the hazard related to non-traditional dusts and to orient the research on the development of more specific laboratory test, adequate and reliable for fibrous materials.

5.3   Experiments

The work presented in this section and supported by the Unione Industriale Biellese has 2 main purposes, which are connected:

- To define the particle size distribution of dust samples collected at textile industrial reality of the North-west of Italy;
- To assess the actual explosion hazard of those samples (investigation carried out on 15 samples considered more hazardous)
- To trace potential correlations between the size of samples and the specific machinery dusts are produced by, with respect to their explosion likelihood.

During the present work 100 dust samples were sampled from different companies in a textile district in the north of Italy. The samples have been examined, as expected all could be included in the non traditional dust category. All the samples were subjected to particle size distribution analysis and 15 of them to the Screening Explosibility Test (SET) developed at the Politecnico di Torino, described in previous Chapter.

The conclusions and assumptions that could be made on the results of this work could be relevant in the direction of increasing the knowledge of nontraditional dusts behavior and sensitivity to different ignition sources.

5.3.1 Methodology

Samplings were performed in situ and the collection included "ambient samples" and "machinery samples". The first category includes samples collected from workplace, from air treatment system or in general from the environment of the sites examined, while machinery samples were collected nearby process machinery or from on board suction unit.

Each sample, as collected from field, was characterized by the particle size distribution (PSD) and the moisture content. A selection of 15 samples were submitted to the SpeditiveExplosibility Test and among them 4 were subjected to explosion trials in the 20 l sphere.

The characterization tests were performed with the same procedures described before for the sample submitted to the SET test (Chapter 4).

Table 17:Characterization tests.

Parameter	Standard/Methodology	Unit of measure
Moisture	ISO 562:2010	Weight %
PSD by Sieving	Internal 08/2014	Weight %
PSD by Laser diffractometry	ISO 13320:2009	Weight percentiles

The problematic related to fibrous dust here raised relevantly. An instrument, like the Laser granulometer used here, working on the light scattering principle, could fail to distinguish between different particle shapes assuming all signals are generated from spherical particles. The result is that, with flat-like or flock-like particles as the ones analyzed in this work, the laser diffractometry analysis could be less precise than the sieving procedure. To overcome these difficulties, the samples were subjected to laser PSD and sieving measurements and the results were compared for internal check. In case the results were comparable, the laser diffractometry was used as it is faster.

The laser diffractometry was used to measure 56 samples. The 44 remaining samples were treated with the wet sieving procedure.

In this case an adequate amount of dust sample is placed on the top of the sieve pile. Sample is flushed with water and gently moved. The sample fractions are collected from the sieve pile and dried in a laboratory stove at 65 °C for at least 12 hours. The PSD is obtained by weighting.

5.4   Materials

Dust samples are divided per textile process section and per sampling position.
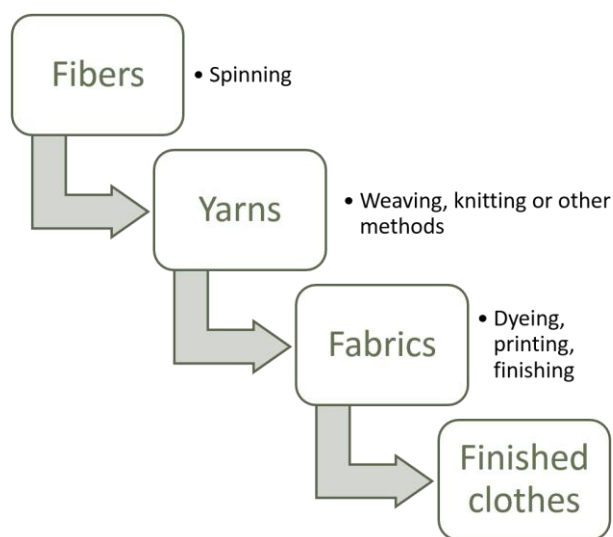

Figure 42: Textile process.

The basic scheme of a textile process plant is shown in the figure above.

Spinning is a series of operations by means of which a mass of textile fibers (staple), initially in disorder, can be arranged into one very long unit (yarn). There are multitudes of spinning processes which vary according to the characteristics of the fibers to process and to those of the yarns one wants to obtain. The different phases of the process are complex and aimed at achieving a high quality, regular, and resistant yarn with minimum hairiness.

Carding is a mechanical process that disentangles, cleans and intermixes fibers to produce a continuous web or sliver suitable for subsequent processing

Winding is the last phase of spinning and consist in winding the yarn on a tube that can be either cylindrical or conical in shape, unwinding is the opposite process.

Weaving is a method of textile production in which two distinct sets of yarns or threads are interlaced at right angles to form a fabric or cloth.

Reeling is the process that convert yarn in commercial portable form. It's normally done after twisting/winding to wrap yarn carefully in hank form and unwinding it later.

Figure 43 shows the different source of the samples, collected in several textile industrial sites. Samples are composed mainly by wool, natural fibers and synthetic fibers, depending on the original site of processing.
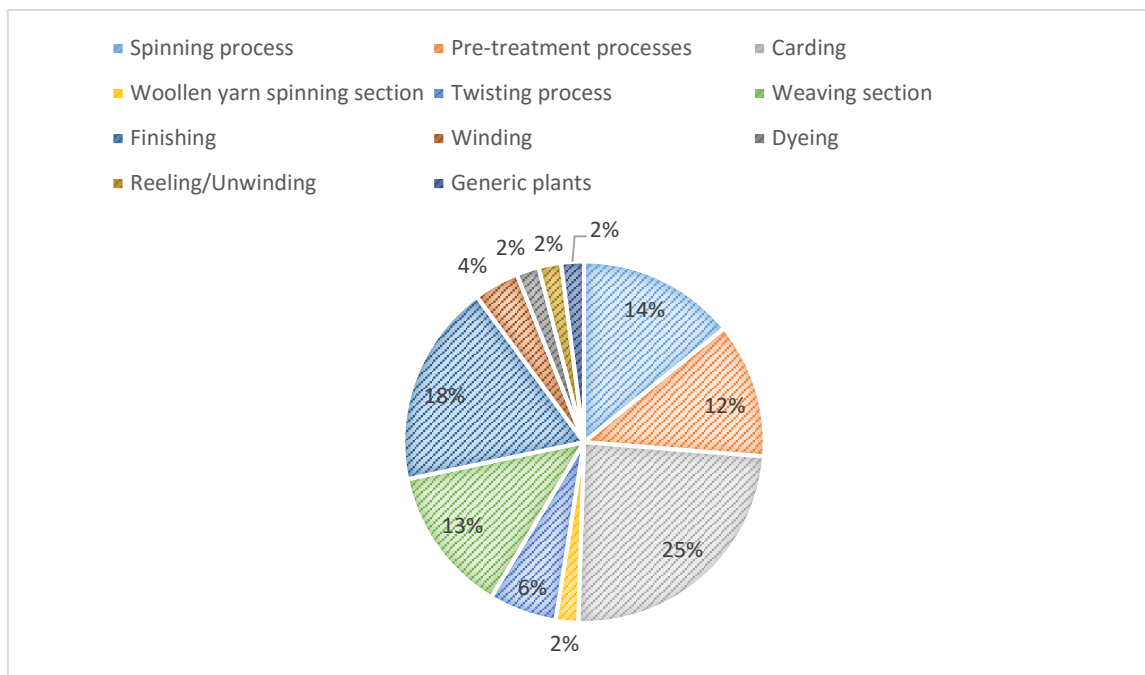
Figure 43: Samples origin, based upon the textile processing sections.

Table 18: Samples classification by sampling sites, divided per process sections.

Process section (n° of samples)	Ambient samples	Machinery samples
Spinning (14)	10	4 (3 spinning, 1 open-end spinning)
Pre-treatment processes(12)	7	5
Woollen yarn spinning (2)	2	-
Carding(24)	5	19 (Carding, open-end, mixed, threshing)
Twisting (6)	5	1
Weaving (13)	8	5
Finishing (18)	-	7 (clipping), 11 (other machineries)
Winding (4)	3	1
Dyeing (2)	-	2
Reeling-Unwinding (2)	2	-
Generic plants (3)	-	3

5.5 PSD characterization

In this section the PSD of the samples is presented according to the process section from where they were collected. Both PSD results obtained by laser diffraction and by sieving are reported (where present). Laser diffractometry was used to characterize, when possible, the PSD of the fraction smaller than 500 µm obtained by sieving.

Morphological characterization through photographs and microscopic observations, along with tendency to disperse in air are also described for each group of samples.

5.5.1 Spinning section

The PSD by laser diffraction was satisfactory for 9 of the total amount of 14 samples. The samples are characterized by a high content of fibrous elements and present scarce dustability. Only one sample is composed by a relevant fraction of fine particles and could be considered easy to disperse in air (132/15 in Figure 44).

Table 19: PSD results, Spinning section samples.

Sample characterization		Undersieve (<500) PSD	
PSD by sieving($\square$m)	Mean of 14 samples (%w)	PSD by laser diffraction	Mean of 9 samples (µm)
>2000	54.19	d10	8.05
1000<d<2000	2.01	d50	59.98
500<d<1000	3.42	d90	380.49
<500	40.40		
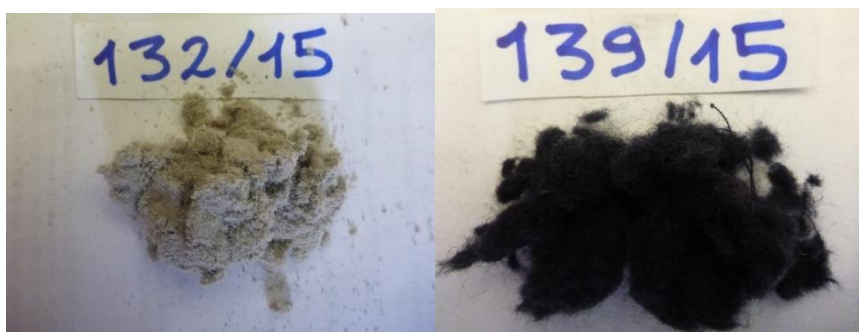
Figure 44: Dust from ambiance sampling (132/15) and machinery sampling (139/15). Origin: spinning section.
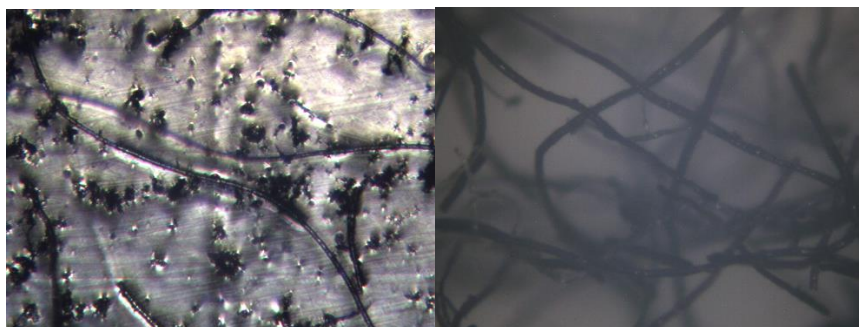
Figure 45: Optical microscopy image (5X) of the two samples above, 132/15 contains fine particles, while only fibers are present in 139/15

5.5.2 Preparation processes

As what concerns machinery samples only one of five could not be processed by laser diffraction. The PSD obtained from 3 of the other 4 samples present a single peak, one was binomial.

All the environmental samples were submitted to laser diffractometry PSD analysis.

Table 20: PSD results, Preparation section, machinery sampling.

Sample characterization		Undersieve (<500) PSD	
PSD by sieving (µm)	**Mean (%w)**	**PSD by laser diffraction**	**Mean (µm)**
>2000	58.18	d10	10.05
1000<d<2000	0.44	d50	34.51
500<d<1000	1.27	d90	253.06
<500	40.12		

Figure 46: *Preparation section samples, from machinery (137/15) and ambiance sampling (181/15).*

Table 21: PSD results, Preparation section, ambiance sampling

Sample characterization		Undersieve (<500) PSD	
PSD by sieving (m)	**Mean (%w)**	**PSD by laser diffraction**	**Mean (m)**
>2000	27.58	d10	868
1000<d<2000	2.96	d50	38.76
500<d<1000	5.96	d90	302.39
<500	63.80		

5.5.3 Woollenyarn spinning section

One over two samples could not be processed by laser diffractometry. The samples are both characterized by fibers and not appear to have a significant attitude to be dispersed in form of dust cloud.

Table 22: PSD results, Woollen yarn spinning

Sample characterization		Undersieve (<500) PSD	
PSD by sieving (m)	Mean (%w)	PSD by laser diffraction	Mean (m)
>2000	67.40	d10	3.58
1000<d<2000	1.61	d50	49.20
500<d<1000	5.62	d90	375.17
<500	23.35		

***Figure 47:** Woollen yarn spinning samples.*

5.5.4 Carding section

The samples collected from the carding section splits up in two groups with similar characteristics: the PSD of 12 samples could be measured by laser diffractometry. A relevant fine particles fraction was detected. The second group was composed of samples that could not be processed by laser diffraction, characterized by a relevant amount of particles greater than 2000 um (mean value greater than 50%).

Table 23: PSD results, Carding section.

Sample characterization		Undersieve (<500) PSD	
PSD by sieving (m)	Mean of 24 (%w)	PSD by laser diffraction	Mean of 12 (m)
>2000	55.51	d10	14.42
1000<d<2000	6.13	d50	76.14
500<d<1000	4.60	d90	373.91
<500	33.42		

Figure 48: *Carding section samples, from ambiance (203/15) and machinery sampling (205/15)*

5.5.5   Twisting process

The samples collected in the section devoted to the twisting process were quite homogeneous in shape and size. Only 2 of 6 could not be processed by laser diffraction being composed by fiber-like particles.

Table 24: PSD results, Twisting process.

Sample characterization		Undersieve(<500) PSD	
PSD by sieving (μm)	Mean of 6 (%w)	PSD by laser diffraction	Mean of 4 (μm)
>2000	31.16	d10	2.69
1000<d<2000	1.18	d50	10.28
500<d<1000	3.36	d90	90.31
<500	64.28		

Figure 49: *Ambiance (141/15) and machinery sampling (224/15) from twisting process.*

5.5.6   Weaving

Four of the 13 weaving dust samples could not be processed by laser diffraction; the other nine presented a PSD curve with one single peak. These samples had a relevant fine particles fraction and with a homogeneous distribution of particle sizes.

Table 25: PSD results, weaving section.

Sample characterization		Undersieve (<500) PSD	
PSD by sieving	Mean of 13 (%w)	PSD by laser diffraction	Mean of 9 (μm)
>2000 um	38.05	d10	7.42
1000<d<2000	3.18	d50	32.84
500<d<1000	3.55	d90	155.36
<500	54.74		

Figure 50: *Ambiance (171/15) and machinery sampling (169/15) from weavingsection.*

5.5.7   Finishing

Samples from finishing departments present a relevant fraction of particles greater than 2000 μm (mean value greater than 50%) and are typically samples composed by fibers and relevant agglomerates of particles. Their tendency to disperse in air is extremely low. Only three samples of 18could be dispersed such that their PSD was measured by laser diffraction.

The PSD of these samples presented as much as three maximum, as a confirmation of their inhomogeneity and to underline the difficulty to analyze efficiently those kind of samples with diffraction methods.

Table 26: PSD results, Finishing section.

Sample characterization		Undersieve(<500) PSD	
PSD by sieving (μm)	Mean of 18 (%w)	PSD by laser diffraction	Mean of 3 (μm)
>2000	52.14	d10	18.76
1000<d<2000	12.29	d50	112.53
500<d<1000	7.63	d90	435.94
<500	28.31		
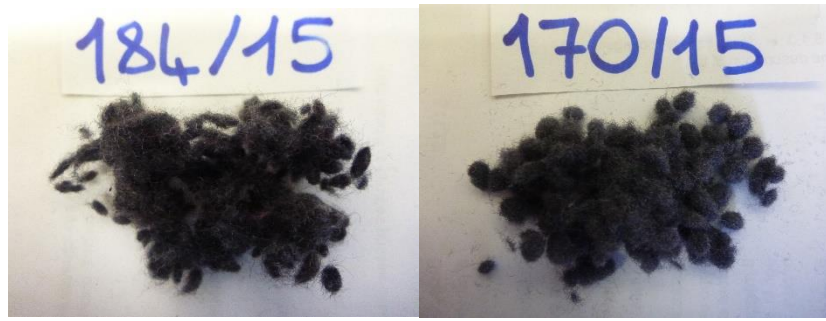
Figure 51: Ambiance samples from finishing sections.

5.5.8 Winding

The PSD of the samples collected from ambiance sampling in the winding department was measured by laser diffraction. On the contrary the samples collected from machinery wastes were composed entirely by fibers and could not be analyzed by laser diffractometry.

Table 27: PSD results, Winding section.

Sample characterization		Undersieve (<500) PSD	
PSD by sieving (μm)	Mean of 4 (%w)	PSD by laser diffraction	Mean of 3 (μm)
>2000	42.74	d10	3.36
1000<d<2000	1.86	d50	18.05
500<d<1000	4.77	d90	155.67
<500	50.56		

Figure 52: Ambiance (215/15) and machinery sampling (231/15) from winding section.

5.5.9 Dyeing

Samples from dyeing departments could not be processed by laser diffractometry. These samples were collected from the machinery which are in the final steps of the textile production, thus explained the flock shape of the samples collected here and its low tendency to disperse finer particles in air.

Table 28: PSD results, Dyeing.

Sample characterization		Undersieve(<500) PSD	
PSD by sieving (m)	Mean of 2 (%w)	PSD by laser diffraction	Mean (m)
>2000	84.15	d10	NE
1000<d<2000	0.50	d50	NE
500<d<1000	0.80	d90	NE
<500	14.55		
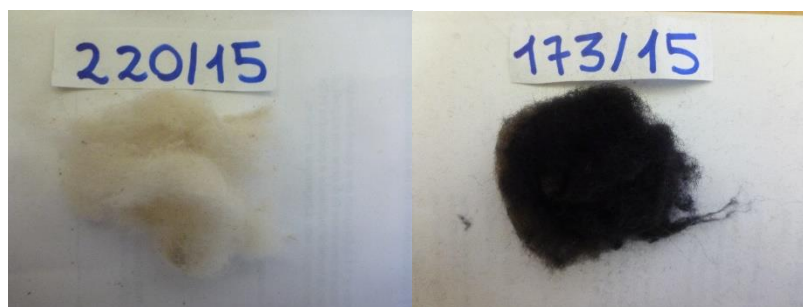
Figure 53: *Dyeing samples, from machinery*

5.5.10 Reeling – Unwinding section

The PSD of the two environmental samples collected at unwinding department were measured by laser diffraction and resulted similar.

Table 29: PSD results, Reeling- unwinding section

Sample characterization		Undersieve(<500) PSD	
PSD by sieving (m)	Mean of 2 (%w)	PSD by laser diffraction	Mean of 2 (m)
>2000	6.78	d10	7.62
1000<d<2000	2.49	d50	32.86
500<d<1000	6.66	d90	184.06
<500	84.05		

5.5.11 Generic plants

These samples are characterized by a briquette-like agglomerates of particles. Laser diffraction was possible for all, although they had to be dispersed manually before being injected to the particle size analyzer.

Table 30: PSD results, Generic plants.

Sample characterization		Undersieve (<500) PSD	
PSD by sieving (m)	Mean of 3 (%w)	PSD by laser diffraction	Mean of 3 (m)
>2000	25.97	d10	16.66
1000<d<2000	18.18	d50	95.73
500<d<1000	8.05	d90	415.13
<500	47.83		

Figure 54: *Generic plants (carding and spinning machineries) sample.*

5.6   PSD investigation results

The first part of this work intended to define the PSD of dust samples derived from different department and machinery of a huge number of typical textile processes. Different PSD measurement techniques were applied to all the samples depending on their characteristics.

Figure Figure **55**, Figure **56** and Figure **57** underlined the followings considerations:

- A relative high fraction of the samples (44%) contains a fine particle fraction (<500 um) greater than 50% in weight (see Figure 55);
- 56 samples could be examined by laser diffraction and presented a d90 smaller than 600 um (Figure 57);
- 32 samples are examined by wet sieving and among these 50% contains a fraction smaller than 75 um greater than 10.5% (Figure 56);
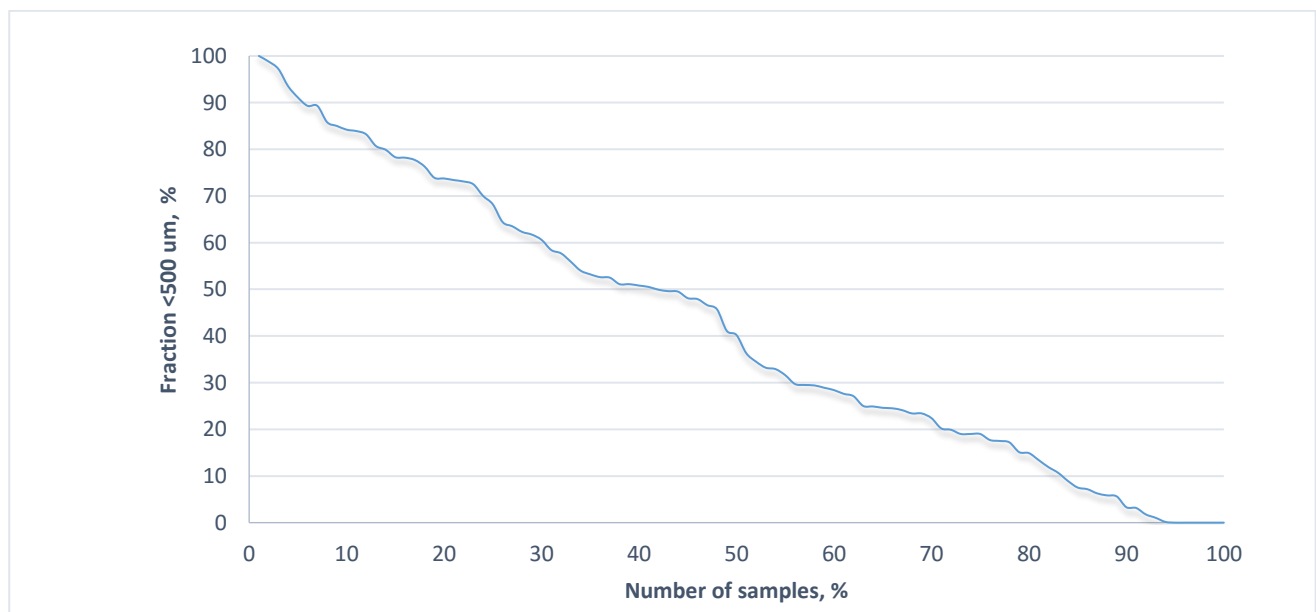
Figure 55: Content of particles <500 µm (weight base) with respect to sample number.
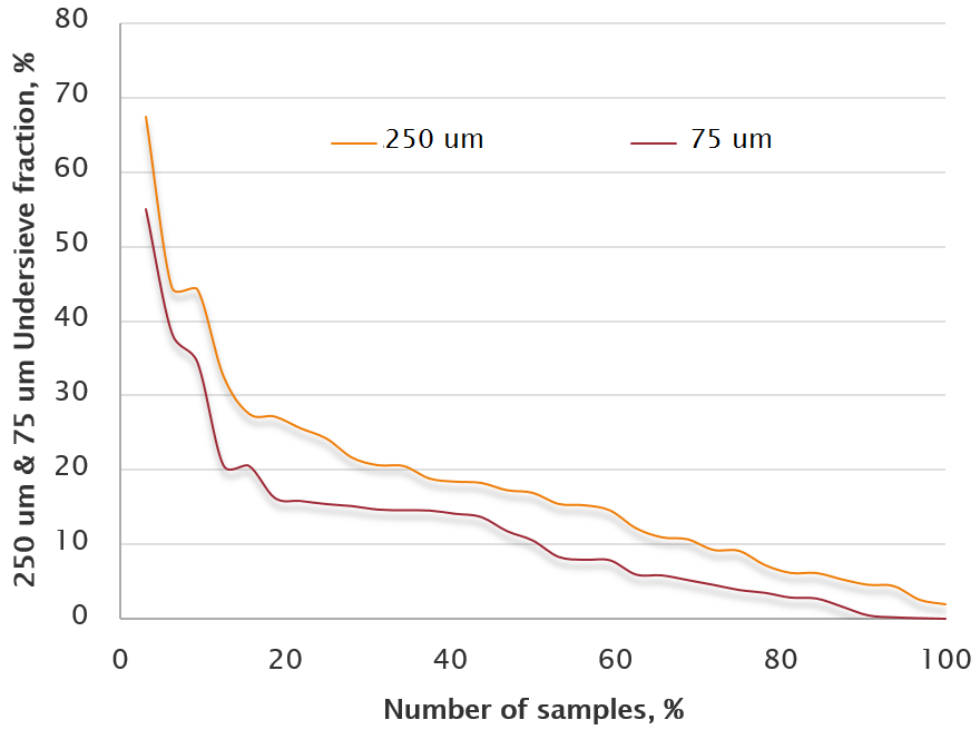
Figure 56: Content of particles <250 μm and 125 μm(weight base) with respect to sample number.
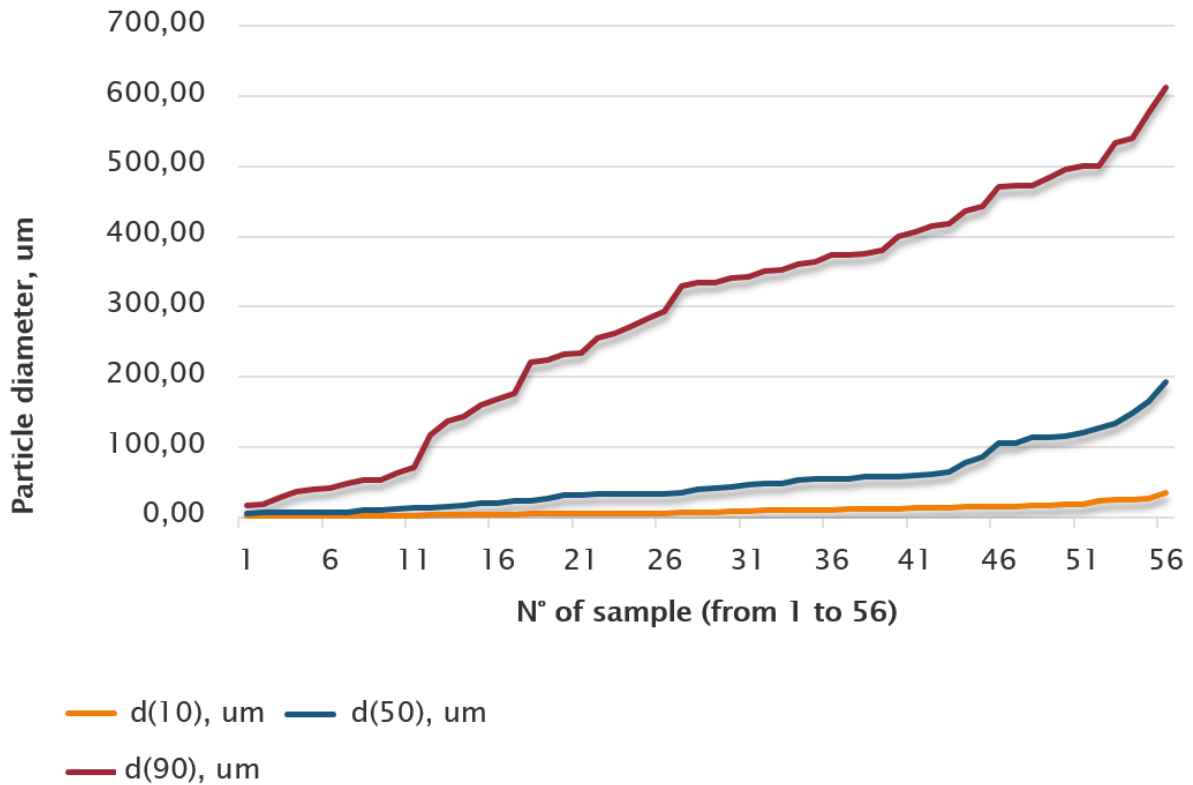
Figure 57: Quantiles distribution versus the amount of samples (56) processed by Laser Granulometer.

5.7   SET Results

Speditive Explosibility Test (SET) was used to determine the explosibility of 15 of the 100 samples studied here, to assess their ignition sensitivity and explosion likelihood. Results are shown in Table 31. The SET combines different type of ignition sources at ambient temperature and in a hot environment as described in details in Chapter 4.

On the total amount of 15 samples:

- 10 resulted as "Explosible at Ambient Temperature"
- 4 resulted as "Explosible at High Temperature";
- 1 resulted as "Non Explosible"

Generally speaking, all the samples have scarce tendency to create dust clouds when subjected to air blows. One sample of 15 is almost non dispersible and could not be tested in any of the equipment used in the SET procedure.

The dispersion unit used to feed the G-G furnace, in particular, seem to be inefficient with such nontraditional dust samples, thus compromising the results of the SET when dealing with those type of samples.

Also the dispersion system used in the Hartmann tube is only partly efficient to disperse flocculent dusts such as those studied here. Hence the reproducibility of tests conducted in the Hartmann tube is quite low. Despite these aspects should decrease the apparent reactivity of the dust examined at SET, it is worth to note that 14 samples were classified somehow flammable.

Table 31: SET results. N: No ignition, P(I,0,5): Ignition (attempt N°/sample mass), NE: not executed. EA: explosible at ambient temperature, EH:esplosible at high temperature, NE: not explosible

| Sample | Section of sampling | Room temperature Tests | | High temperature tests | | SET class |
		Continuous arc	Glowing wire	G-G furnace	Hot plate	-
133/15	Spinning, ambient	N	P (I/0,5)	NE	NE	EA
135/15	Carding, ambient	N	P (II/0,5)	NE	NE	EA
142/15	Weaving, ambient	N	N	P (I/0,3)	NE	EH
144/15	Finishing, machinery	N	N	P (I/0,3)	P	EH
150/15	Weaving, ambient	N	P (III/1,0)	NE	NE	EA
154/15	Weaving, ambient	N	P (I/0,3)	NE	NE	EA
158/15	Winding, ambient	N	P (I/0,3)	NE	NE	EA
160/15	Finishing, machinery	P (II/0,5)	NE	NE	NE	EA
165/15	Carding, machinery	N	N	N	N	NE
168/15	Finishing, machinery	N	N	P (I/0,3)	NE	EH
177/15	Twisting, ambient	P (I/1,0)	NE	NE	NE	EA
181/15	Preparation, ambient	N	P (III/0,3)	NE	NE	EA
186/15	Carding, machinery	N	P (I/1,0)	NE	NE	EA
213/15	Spinning, ambient	P (I/0,3)	NE	NE	NE	EA
227/15	Generic plants, machinery	N	N	N	P (II)	EH

Table 32: Siwek 20 L sphere tests performed on four of the textile dusts.

| Sample | SET class | 20l Siwek Sphere tests | | | |
	-	Pmax (bar)	$(dP/dt)_{max}$ (bar*s^{-1})	kst (bar·m·s^{-1})	St Class
133/15	EA	7,0	390	105	1
135/15	EA	6,1	164	44	1
158/15	EA	6,3	183	49	1
160/15	NE	6,1	93	25	1

Conclusions

This Thesis has concurred in the investigation and definition of the explosibility of solid materials, in form of dusts. The term "explosible" is strictly connected to the attitude of the dust examined to be ignited, then to its sensitivity to different kind of ignition sources determined by the place and conditions in which the dust is generated.

The probability with which a dust will ignite when submitted to external stress (like hot environments or electric discharges or arcs) is found to grow monotonically with the intensity of the applied stress.

The individuation of an interval in which the ignition probability, so the explosion hazard, grow from the 0% to the 100% with respect to a defined variable is a relevant result coming from this work. In the case, the variable examined was the temperature, but this strategy will apply also to energy of external ignition sources.

The first implication of this consideration contribute to the determination of a temperature safety threshold value, corresponding to sufficiently low ignition probability, then to the determination of a new concept of Minimum Ignition Temperature with respect to the standard one.

The second implication deal with the development of a explosibility test (SET) that could define the explosibility or not of a dust sample on a strong statistical basis, derived from the PEI theory.

Consequently, this work should suggest a reexamination of the international standards applied nowadays for Minimum ignition Temperature determination and also on the possibility of redefined explosibility test when dealing with non traditional dusts. At this purpose the experimental application of SET and PSD investigation performed on several textile dust samples show relevant results both regarding the ignition sensitivity and the explosion severity of this type of materials.

The findings are reported under separate headings according to the different phases of the work.

Speditive Explosibility Test

The outcomes of the SET are:

- EA: Explosible at Ambient temperature;
- EH: Explosible at High temperature;
- Nexp: Non explosible

Dust sample that are classified as NExp are reasonably regarded as dusts that, in normal conditions, both in form of clouds or layers, should not imply an explosion hazard in the workplace in which they are located.

Dusts classified as EA have to be regarded carefully, because should reasonably imply an explosion hazard, even at ambient conditions. More measurements on their explosion parameters are suggested in order to implement adequate protection measures for equipment in which these samples are manufactured or handled.

Dusts classified as EH are considered hazardous as they could explode also at ambient conditionns. For those type of dusts the SET suggest a verification of their explosion strength with a test in the 20 L Siwek-Sphere with more energetic ignition sources (couple of 5 kJ and couple of 2 kJ

chemical igniters). The test with the 2 kJ is required for those samples, in order to eliminate the possibility that an overdriving has occurred with the more energetic sources (10 kJ).

In the present study, seven of 22 dust samples were classified as Explosible at High temperature and a verification procedure has been performed with the tests in the Siwek sphere.

Among those, two samples measured values of k_{st} equal to 0 when tested with the couple of 5 kJ igniters, confirming their non-hazardous nature at ambient conditions.

Other two samples, although showing evidence of explosion with the 10 kJ igniters, have measured values of k_{st} equal to 0 when tested with 2 kJ igniters; therefore it could be demonstrated that an overdriving occurred with the more powerful ignition source.

Two samples are classified EH and they exhibit a measurable kst with the weaker igniters. An evidence of explosion (overpressure greater than 0.5 bar, a) occurred with both the samples with a relative high concentration of 3000 g/m3

In order to assess the real behavior of those two dust samples a test in the 1 m^3 chamber of explosion is suggested, here not performed due to absence of adequate equipment).

Finally, a sample does exhibit a measurable k_{st}, even if it was classified as Nexp in the SET procedure, due to its fibrous nature and impossibility to be dispersed through the dispersion system of the SET equipment.

In conclusion, SET results were verified by these "high energy" standard tests and the procedure adopted here seemed to reveal the occurrence of false positive (i.e. overdriving phenomena) for some samples. In addition, test classification criteria seem to agree with criteria proposed in the Kuhner explosibility test, present in the 20 L sphere and with the standard procedure to determine dust explosion parameters UNI EN 14034.

Nevertheless, these procedures seemed not to work well with fibrous samples and mixture of different dusts. A more comprehensive procedure has to be develop to include those kind of dusts in the SET classification and verification.

The followings approaches could be adopted in future works to align results of the SET to update literature findings on overdriving effect.

Alternative approaches to overdriving phenomena evaluation

Many authors have deal with the under and over driving phenomena that occur when dust samples are tested in different vessel tests and with igniters of different energy.

The above-cited work by Rodgers and Ural (2011) presented the OSHA NEP implementation to the definition of a combustible dust. The test apparatus for this procedure is again the 20L sphere. A range of different dust cloud concentrations is tested with one single 2.5 kJigniter.

The maximum pressure ratio PR and thenormalizedrateof pressure rise K are recorded foreach test.

$$PR = \frac{(P_{ex,a} - \Delta P_{ignitor})}{P_{ignition}} \tag{10}$$

Where:

$P_{ex,a}$ is the maximum explosion pressure reached during a single test; $\Delta_{p,ignitor}$is the maximum pressure rise in the chamber due to the ignitor by itself; $P_{ignition}$ is the absolute pressure at the time of igniton.

And K is defined as

$$K = \left[V^{1/3} (dP/dt)_{max} \right] \tag{11}$$

OSHA consider an explosion to occur and consequently a dust sample explosible, when PR is greater than 2 and K is greater than 1.5 bar/ms^{-1} in anyone of the tests

As said before, they suggest paying attention to marginally explosive dusts and their classification with tests performed in the 20L sphere. Indeed, they reported results of k_{st} tests for the same dust sample with three different methods: the OSHA with the 20L sphere and two commercial tests in the 20L sphere and in the 1 m^3 chamber respectively.

Values of PR and K of the tests performed in the sphere revealed the dust to be explosible, while in the 1 m^3 chamber the explosion threshold imposed by OSHA is not overpassed. They concluded that when OSHA test or Standard 20L-Explosibility test results indicate a marginal degree of explosibility for a dust sample it is cost effective to perform the definitive screening in a 1 m^3 chamber or larger enclosures.

Proust et al. (2007) made a comparison between explosibility test performed in the 20L sphere and in the ISO 1m^3 vessel.

They reported some dust samples that show significant explosion severity in the 20L sphere are not even explosible in the larger vessel. They suggest the ignition energy is the dominating force in the reaction, inducing a strong preheating on the samples. Experimental results indicate that a significant percentage of dusts (5 of 21 tested) explode in the 20L sphere, even weakly, and not at all in the ISO 1 m^3 chamber.

They stated from their analysis that dust samples that exhibit a k_{st} equal to or smaller than 45 bar/ms^{-1} would not explode in the 1 m3 chamber, defining this value as the overdriving threshold in the 20L sphere.

Our experimental data could be compared to Proust assumptions. In particular, we have found sample 11, 13, 15 and 17 with a value of k_{st} smaller than 45 bar/ms^{-1}.

Result associated to sample 13 agreed with Proust threshold, since we have found this sample not explosible with both 2X5 kJ and 2X1 kJ igniters. However, samples 11 and 15 resulted Explosible at Ambient Temperature in the SET procedure and have not been subjected to the verification with the 2X1 kJ igniters.

As what concerns sample 17, as said before, due to its fibrous nature, problematics related to the dispersion efficacy of SET equipment are influencing results and a revision of the tests has to be done before drawing any conclusions on its explosibility.

In addition we could state that also sample 8 could be considered by Proust approach, even its k_{st} value overpassed the threshold found by this author (53 bar/ms^{-1}); this little discrepancy between tis value and the 45 bar/ms^{-1} individuated by the experimental campaign of Proust has to be verified, preferably with tests in the 1 m^3 vessel.

More recently Kuai et al. (2013) proposed another two approaches to evaluate whether an over driving has occurred. The first approach is based on the determination of the flame thickness. This value is estimated as the distance traveled by the rear of combustion in the last phase of the explosion, assuming the peak pressure is reached in the instant when rear of combustion reaches the wall of the vessel.

The authors study the evolution of the flame thickness with the energy of the igniters. They found that the curve shows a steep decrease in the initial phase, then a minimum is attained and the flame thickness almost increase linearly. The rate of pressure rise with ignitor energy is also reported. Authors have found a critical value of energy (E_{cr}) corresponding to an inflection in the

dP/dt versus E_{ign} curve, from steep increase to almost asymptotic behavior. This value of energy is the same as the one that minimize the flame thickness and authors have adopted it as overdriving threshold.

All igniters that generate an energy greater than E_{cr} could cause overdriving phenomena and alter the results of the explosion parameters measurement.

The other approached they proposed is based on the evaluation of the induction time t_i, in comparison with the time to reach ignition, t_{ign}.

The occurrence of $t_i < t_{ign}$ means that energy from the ignitors overpassed the sufficient energy for ignition and a significant part of it is devoted to heat propagation and that overdriving has occurred.

From our experimental tests data are not sufficient to verify the consistence of this method. This could be an interesting perspective in order to validate our procedure in agreement with these literature findings.

Ignition temperatures of dust mixtures

The Minimum ignition temperature of dust clouds obtained by mixing different dusts was measured.

In the case of mixtures obtained mixing a combustible (flour) and an inert dust, the MIT_C decreases as the mass fraction of inert rises. Inert dusts, which undergo endothermic decomposition, are, as expected, much prone to increase the mixture MIT_C. Limestone effect on MIT_C is limited: when mixed with 10% flour the temperature rises to only 540°C. The size of the inert dust has little influence on the MIT_C. Limestone 32-75 µm and 125-250 um mixed with flour exhibited the same MIT_C except at 60% (weight basis) inert. At that composition the mixture obtained with finer 32-75 um limestone exhibited a 10 °C lower MIT_C than the one obtained with the coarser 125-250 um.

It is also clear that MIT_C measured according to CEI EN 50281-2-1 is not precise enough to study the behavior of mixtures when the influence of the variables is small. To this purpose a more defined representation can be obtained using the Statistical Ignition Temperature (SIT).

The use of SIT allowed to describe the behavior of mixtures between combustible dusts having similar MIT_C. It was evident that some mixtures do not follow the harmonic model, in the case of lactose a small amount of a different component (flour, sucrose), cause a steep decrease of MIT_C. Sulphur as well promote a steep decrease of MIT_C inducing a trend that is not represented by the harmonic law.

The Statistical definition of Ignition Temperature

The second approach to the definition of a Statistical Ignition Temperature was made with the adoption of a Normal distribution curve fitted over the experimental data of Lycopodium and Lycopodium-AC93 powder mixture tests.

The model proposed succeed to represent the effect of the inert powder to increase the MIT of the mixture and the shift of the Probability Escalation Interval towards higher temperatures.

The table below reported the results obtained. A difference of 23.2 °C exist between the MIT defined through standard procedures and the Normal law definition.

The statistical approach well represented the effect of the decreasing of inert particle sizes in increase the inertial capacity of the powder, a SIT greater than 8°C was found in this case, with respect to the SIT of mixture with inert powder not sieved.

	M.I.T (°C)	S.I.T. (°C)	ΔT (°C)
Lycopodium	420	443,2	23,2
Lycopodium/AC.93	440	467.3	27.3
Lycopodium/AC.93 (<75µm)	-	475.5	-

The third statistical approach not only moved towards a refinement of the statistical value of the ignition probability, but also gave birth to a new definition of the Minimum Ignition Temperature with respect to the standard.

This approach was applied to Lycopdium pure dust sample.

The Autoignition probability of Lycopodium at a temperature range just above the Autoignition Temperature (AIT) is a monotonically increasing function with respect to temperature. This range of temperatures was called the "Probability Escalation Interval", PEI. The set of data was analyzed by a GLM with different link functions and the probit link provided the best fit.

The AIT can be estimated by assuming a residual ignition probability small enough to fit a risk analysis requirement. In the present case, by assuming $k = 0.001$, AIT was 434,1°C. According to our results, CEI EN 50821 measurement procedure applied to the same data would have a 65% probability to yield a result of 440°C and 35% probability to yield a result of 420°C.

It is evident that the statistical approach proposed here is much more suitable to treat experimental data when an accurate value is needed or if small differences have to be appreciated, e.g. for scientific purposes. On the contrary, the CEI EN 50821 method would bring to estimate an AIT of 440°C with a 65% probability, or an AIT of 420°C with the 35% probability. No intermediate values could be obtained.

Beside these more scientific arguments, it is interesting to observe that, if the probability escalation interval (PEI) would be wider than 25-30°C as in the case of Lycopodium, using the method proposed by the norm would have a greater probability to produce poor estimate of the AIT, and, the most relevant, the probability to overestimate AIT would be not negligible.

The literature data on the probability escalation interval are scarce, as the data on the probability dependence respect to T (or to energy, in case of spark ignition). It seems quite important to further explore this matter in order to improve the awareness on the confidence of AIT or MIE measurement methods, as well to improve the measurement accuracy needed to study some physical phenomena that could affect AIT or MIE, such as PSD variation, mixture behaviors etc

Testing non traditional dusts

The PSD of 100 textile waste dusts sample was investigated. The samples were collected in different textile industrial realities in the North-west of Italy and they are characterized depending on the different section of the textile process they derived.

As expected, all the samples could be defined as non-traditional dusts, having fibrous nature and with a relative scarce tendency to disperse in clouds.

Samples from Winding, Twisting and Weaving sections are smaller than samples from other sections;

At each department, it was observed that samples representative of ambiance dust have smaller PSD than samples collected at machinery.

According to PSD the dust samples more prone to explode are those coming from Winding, Twisting and Weaving departments

In general, environmental dust samples had a smaller PSD than "machinery" dust:

Eight over 9 environmental samples were defined as "Explodible at Ambient temperature" according to SET, among these 2 were from Weaving section and 1 from Winding and Twisting respectively.

Dust dispersion provided by the GG furnace is not adequate for flocculent materials, in particular GG furnace dispersion system should be improved to test these kind of dust.

The four dusts submitted to Siwek 20 L sphere tests all exhibit a measurable k_{st}. and should be classified as St1. Among them sample 133/15 show the most severe explosion with a k_{st} equal to 105 bar/ms^{-1}.

This value is in agreement with literature k_{st} values found for textile dusts: Piccinini (2008) found a

St1 classifcation (with k_{st} equal to 110 bar/ms^{-1}) for samples composed mainly by wool dusts and vegetable particles.

The samples named 135/15 and 158/15, despite being classified as Explosible at Ambient Temperature, exhibit a less severe k_{st} values, below the threshold defined by Proust (2007) to eliminate the possibility of an overdriving occurrence. For these samples an alternative procedure to estimate their actual explosion strength is suggested.

The last sample 160/16 (that is reported as sample 17 in Chapter 4) exhibit a measurable k_{st}, even if SET declared it Non explosible. The poor dispersion of this sample explained here the poor efficacy of SET equipment, while 20 L sphere dispersion mechanism could succeed in generating the dust cloud and explode it with the 10 kJ igniters. Again further tests are suggested for the explosibility definition of this sample.

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