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From

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Ignition temperature of hybrid mixtures

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Task description of the thesis for

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Topic: Ignition temperature of hybrid mixtures

Description:

As part of the project, NEX-HYS the MIT of hybrid mixtures has to be determined in detail. Additionally a new GG oven needs to be tested.

Therefore, the following points have to be fulfilled:

- Literature Review on Safety characteristics
- Description of the changes to the standard MIT procedure
- Test with various dust, vapors and gases under variation of a broad range of masses, pressures and concentrations
- Suggestion of a general testing procedure.

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Declaration

I hereby declare that I prepared the work submitted without inadmissible assistance and without the use of any aids other than those indicated. Facts or ideas taken from different sources, either directly or indirectly have been marked as such.

Further I have not made any payments to third parties either directly or indirectly for any work connected with the contents of the submitted thesis.

The work has not so far submitted either in Germany or abroad in same or similar form as Bachelor-/Master thesis and has also not yet been published as a whole.

Magdeburg, 17.09.2020

A handwritten signature in black ink, appearing to read 'Ajinkya Sunil Kunjir', with a stylized flourish at the end.

Ajinkya Sunil Kunjir

Abstract

Accidental ignition of an explosive atmosphere due to hot surfaces is a major concern in the industries. Such explosion hazards involving hybrid mixtures of different states of aggregation are frequently witnessed in industries that process or handle flammable gases/solvents and combustible dust. Hence, to prevent or control the risks associated with such mixtures, deep knowledge of the explosion behaviour of hybrid mixtures is necessary. Investigation of minimum ignition temperature (MIT) of a two-phase hybrid mixture consisting of combustible dust and a flammable solvent was undertaken in this work. MIT of dust and solvent and their mixture were determined. Combustible starch dust and flammable n-heptane solvent were selected as materials for this work. The testing for MIT of dust was done following the European standard EN 50281-2-1 using the Godbert-Greenwald (GG) furnace. For testing the MIT of solvent and dust-solvent mixture, a modified GG furnace was introduced in this work. The new setup is just a slight modification to the standard setup, which includes the introduction of a solvent reservoir, which allows the solvent to vaporize and is then dispersed into the heated furnace by a blast of air.

The results of tests with hybrid mixtures demonstrate that dust can be ignited below its MIT when a small amount of solvent is added, which is either below its individual Lower explosion limit (LEL) or below its individual MIT and vice-versa. For example, MIT of starch in its pure form was obtained as 360°C. It further decreased to 350°C when 0.35 vol% of n-heptane, which is below its LEL, was added to it. Similarly, the MIT of n-heptane was determined as 240°C. It was ignited at 230°C when starch dust as low as 217g/m³, i.e., 1g dust was added to it. The results finally conclude that likelihood of an ignition increases even below the individual MIT of a substance if a second substance is added to it. Hence, the MIT of hybrid mixtures cannot be predicted by over-lapping the MIT of single substances or the effects of single substances.

Keywords: Hybrid mixture explosion, Minimum ignition temperature, Modified GG furnace

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List of Symbols

$(dp/dt)_{\max}$	bar/s	Maximum Rate of Pressure Rise
K_{st}	bar.m/s	Dust Deflagration Index
m	g	Mass of Dust
P_{\max}	bar(g)	Maximum Explosion Overpressure

List of Abbreviations

ASTM	American Society for Testing and Materials
ATEX	Atmospheres Explosibles Directives
BAM	Bundesanstalt für Materialforschung und -prüfung
EN	European Norms
GG Furnace	Godbert-Greenwald Furnace
LEL	Lower Explosion Limit
LOC	Limiting Oxygen Concentration
MEC	Minimum Explosible Concentration
MIE	Minimum Ignition Energy
MIT	Minimum Ignition Temperature
OSHA	Occupational Safety and Health Administration
PID Controller	Proportional–Integral–Derivative Controller
VCE	Vapor Cloud Explosion

1. Introduction

1.1 Background

An explosion is an exothermal reaction that involves rapid chemical oxidation, which results in an increase in pressure or temperature or both together [1]. Explosions involving combustible dusts or flammable gases or a hybrid mixture of both are of major concern in industries that handle or process such flammable materials. A hybrid mixture explosion occurs when two or more combustible substances are mixed in different states of aggregation. Mixtures of such combustible dusts and gases as well as vapors/sprays, are usually encountered in a wide range of industries such as paint factories (pigments and solvents), agricultural industries, mining industry (coal and methane), grain elevators (small grains and fermentation gases), pharmaceutical industries (incipient and solvents) and petrochemical industries that involve handling and processing of both combustible dusts and flammable gases or vapors [2] [3]. Consequently, dust, gas, and hybrid mixture explosions result in property damage, causes casualties, financial losses, and environmental contamination every year worldwide [3].

Electrostatic discharges, electric or frictional sparks, mine fires, naked flames (welding, cutting, matches, etc.) as well as hot surfaces are considered as some of the common sources of ignitions for environments having a combustible dust cloud or any equipment with a dust layer on it [4] [5]. Hot surfaces capable enough of igniting dust clouds can exist in various parts of an industry. E.g., inside furnaces, burners and dryers of various kinds, these constitute a part of the process equipment itself. Many times hot surfaces can also be generated by hot work or electrical heating and by overheating of bearings and other mechanical parts due to friction [6]. Such ignition sources lead to explosions, which results in the development of dynamic pressure and heat energy leading to the loss of lives and property [5].

The coal mining industry, particularly in leading coal-producing nations such as China, U.S.A., India, and Germany, have witnessed many such disasters caused due to dust and gas explosions [5]. Largescale vapor cloud explosions (VCEs) have occurred in Buncefield, UK in 2005 and CAPECO, Puerto Rico in 2009 that caused severe damage to human life, property and infrastructure, and the surrounding

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environment [7]. It is, therefore, necessary to study and understand different types of explosions, taking into consideration different explosion parameters and conditions, as a result of which relevant safety characteristics and advanced preventive measures can be designed, developed, and tested to prevent or mitigate such explosions. Table 1 shows the distribution of major losses due to various events in the global chemical industry from 1970-1999 [8].

Table 1: Worldwide major losses in the chemical industry during 1970-1999 (Data from [8]).

Event	Percent (%)
Fire	47
Explosion	30
Vapor cloud fire/explosion	15
Mechanical breakdown	3
Other	5

1.2 State of Art

In 1885, Engler was the first one to observe that a hazardous environment having a potential of a severe explosion with enormous release of heat could be produced if a non-explosible amount of coal dust (i.e., below its minimum explosible concentration (MEC)) is mixed with methane at a concentration below its lower explosion limit (LEL). Since Engler's first experiment in 1885, a lot of research has been carried out on the behaviour of hybrid mixtures to reduce the consequences or either preventing its occurrence [9]. Various researches and reviews on explosion characteristics of combustible dust have been carried out by Sweis [10]; Mishra et al. [5]; Eckhoff [6]; Babrauskas [8]; Polka et al. [11]; Zhao et al. [12]. Similar studies on gas explosions were carried out by Cashdollar et al. [13]; Zipf et al. [14]; Wang et al. 2017a [15], 2017b [16]. In order to prevent or mitigate the risks associated with hybrid mixture explosions, various explosions parameters such as maximum explosion pressure, the maximum rate of pressure rise, deflagration index, lower explosion limit (LEL), minimum ignition energy (MIE), minimum explosible concentration (MEC), minimum ignition temperature (MIT) have to be determined [4].

Hybrid mixture explosions involving a mixture of both flammable dust, gas or solvent continue to occur in industries handling flammable gases or vapors. Some of the

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researches on hybrid mixtures explosions can be found in the literature of Khalili et al. [17]; Addai et al. 2015a [1], 2015b [18], 2016a [3], 2016b [19], 2019 [20]; Dufaud et al. [21], Denkevits [22]. From the analysis of the available literature, it can be concluded that the effects of hybrid mixture explosions cannot be predicted just by overlapping the effects of single substance (only dust and only gas or vapor) explosion. Moreover, the research on hybrid mixture explosions has complexities since there are a large number of complex physical processes occurring during the explosion and various parameters that need to be considered. Hence the data available on explosion characteristics of hybrid mixtures are sparse [1] [2].

The assertions can briefly summarize the main findings of the literature mentioned above on hybrid mixture explosion that both the ignition severity and the ignition sensitivity of dust increase significantly on adding a small amount of combustible gases or vapors, which are below their LEL. It also highlights that the possibility of an explosion in a hybrid mixture increases even when the concentrations of flammable gases or vapors and combustible dust are below their respective LEL and MEC [2].

1.3 Motivation

Industrial process safety is a field where numerous evaluation techniques and measurement methods are used for investigations of various safety characteristics. Prevention and mitigation of dust, gas, or hybrid mixture explosions are one of the sub-fields of process safety research that utilizes various experimental measurement methods for investigating safety-related parameters. One of these safety-related parameters is the minimum hot surface ignition temperature (MIT) of explosible dust clouds or gas/air mixtures [23]. Legally binding regulations on the State Fire Service regarding safe usage of electrical devices states that the surface temperature of power electronic devices installed in an environment where explosive dust is present should be below the $2/3^{\text{rd}}$ value of the minimum ignition temperature of that dust cloud/air mixture. Hence, to minimize the risk of fire/explosion, the key safety parameter, i.e., MIT should be specified from a process safety point of view. The dependence between the explosibility or the probability of explosion and the values of ignition temperatures of a heated surface can be determined. Therefore, these findings can be used to prevent and/or minimize the effects of fires or explosions of dust [11].

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In areas in the industry where explosible dust/gas cloud may occur (inside furnaces, burners, and dryers), it is important to know the minimum temperatures of the hot surfaces present in such environment, at which if the dust/gas cloud comes in contact with such surfaces, can ignite. Once adequate research is done concerning the MIT of single substances and their mixtures, enough measures can be taken to avoid the increase of temperature of such surfaces to these values. To achieve safety goals, advanced safety measures can be designed to prevent or mitigate the effects of dust/gas or hybrid mixture explosion [6].

A general review of the previously mentioned literature concludes that hybrid mixtures are more complex than they appear. The effects of hybrid mixture explosions cannot be simply examined by overlapping the effects of single substance explosion (only dust, only gas or vapor). During risk assessment or explosion hazard assessment, particularly during the applications of ATEX Directives, the possible presence of such hybrid mixtures is not frequently appreciated to its full value. Generally, only the materials of more interest are considered even though there might be another side product or reactant present in the atmosphere, which could make it explosible [9]. Hence, it becomes significant to study the explosion characteristics of a hybrid mixture explosion.

1.4 Problem statement

As a result of the reasons mentioned above, research on various explosion parameters of explosive materials in their pure form and especially a mixture of two or more substances becomes of greater interest. The present thesis seeks to investigate the Minimum Ignition Temperature (MIT) of a two-phase hybrid mixture of starch dust and n-heptane solvent vapor.

Therefore, to understand the fundamentals of an explosion a literature review should be carried out which focuses on the safety characteristics and properties of explosive materials. Brief insight should be sought to study the MIT of dusts, gases and solvent vapors and their role in hybrid mixture explosion. A detailed description of the standard procedure used to investigate MIT of dusts using the Godbert-Greenwald furnace (GG furnace) followed by the modifications made in the standard procedure and the experimental apparatus to allow the investigation of MIT of solvent vapor and dust/vapor/air hybrid mixture should be done.

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However, the MIT of a cloud of dust in air or a vapor cloud is not a physical constant of that particular dust or gas/vapor but largely depends both on the concentration of dust or gas/vapor and state of cloud movement (turbulence, pressure and directional flow), as well as size and shape of the hot surface used as ignition [6]. Hence, tests with dust, vapor, and their hybrid mixture should be carried out under the variation of a broad range of masses, pressures, and concentrations. MIT values can be used to ensure safe operations in industries having areas/surfaces that can be an effective ignition source if heated above the MIT values. Consequently, a suggestion of a general testing procedure and if necessary, to the testing apparatus should be made.

The experimental work presented in this thesis was undertaken to extend the knowledge of MIT of a two-phase hybrid mixture of combustible dust and flammable solvent. Also, to investigate the effect on the MIT of dust and solvent after addition of another solvent and dust, respectively, to make a hybrid mixture.

2. Basic Literature: An Overview

2.1 Explosion

A baffling variety of accidents occur in the process industries during various operations, i.e., during storage, manufacture, and transportation of goods. These accidents range from small innocuous leakages to catastrophic releases (like the ones in Seveso and Bhopal), from hardly notices tiny sparks to whole-consuming fires, and from the plop of a bubble to earth-shattering explosions witnessed at Feyzin [24], Mexico City [24], Jaipur [25], Buncefield [7]. The impact can also have a bewildering range from a temporary malfunction of a breakdown of a certain small component of equipment to the demolition of the entire factory [26].

On 4th August 2020, a catastrophic disaster that led to a massive explosion of 2750 tonnes of ammonium nitrate stored in a warehouse (shown in figure 1), sending a huge mushroom cloud into the air and a supersonic blast wave throughout the city preceded by a large fire was witnessed at the port of Beirut, Lebanon. According to a US report, the maintenance work which was being carried in the warehouse set fire to unspecified materials in the warehouse, which further increased and initiated the ignition of the ammonium nitrate stockpile [27]. Figure 1 shows the epicentre of the explosion and damaged surroundings.

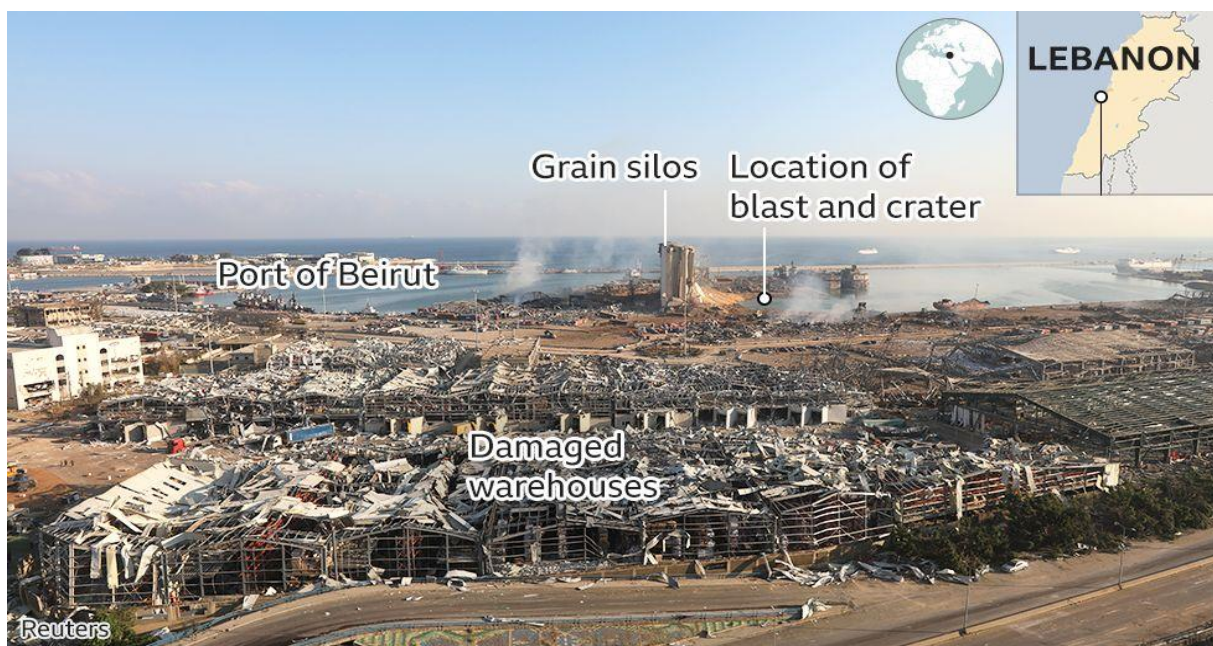


Figure 1: Epicentre of the explosion at the port of Beirut and damaged surroundings. (Source: BBC News [28])

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About 200 people died in the accident, and almost 6000 people were injured, leaving almost 300,000 people homeless due to the destruction caused by the shock wave in the surrounding areas up to a distance of 9km. The blast was heard as far away as 200km. The total collective financial losses are being estimated to reach up to \$10-15bn [28]. Figure 2 shows the extent of damage caused to the surroundings in the radius of 500m.

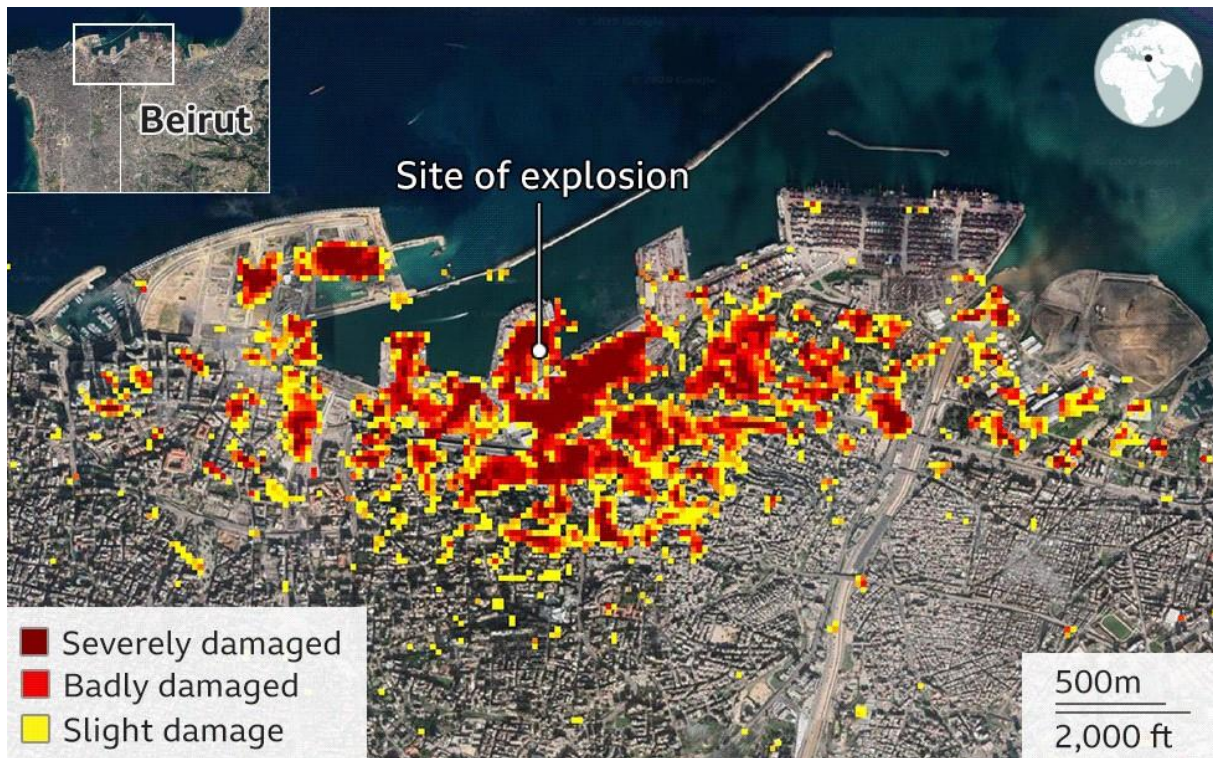


Figure 2: The extent of damage caused to the surroundings in the radius of 500m. (Source: BBC News [28])

An accidental explosion frequently occurs in places where combustible gas or dust is processed or handled. Such mixtures of combustible dusts and gases, as well as vapors, can be encountered in paint factories (pigments and solvents), mining (coal and methane), grain elevators (grain dusts, and fermentation gases), or pharmaceutical industries (incipient and solvents) [3]. After an explosion incident has occurred, the damage quickly spread to wide areas in a very short time, which often results in serious consequences. In simple terms, explosion means rapid formation and expansion of gases. If such a reaction happens in an open space, it creates a pressure wave, also called a blast wave. But If such a reaction happens in a confined/closed space, the pressure increases exponentially until the enclosure is broken by high pressure to generate a blast wave propagating outward [29].

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In more technical language, the phenomenon of an explosion can be defined in terms of an event's ability to generate massive overpressure [26]. According to F. Lee [24], an explosion is a sudden and violent release of energy, where the extent of violence depends on the rate of the energy released. If the energy is released suddenly, a violent explosion could happen. On the other hand, if the same energy is released slowly, it would be dissipated slowly.

For a brief understanding, an explosion may be distinguished by the following characteristics:

- A sudden release of physically or chemically generated and stored energy.
- A blast wave/shock wave moving out rapidly from the explosion source.
- Debris/flying fragments may from the explosion source, also cratering of the soil underneath may lead to projectiles.

The general physical mechanism of an explosion can be broadly summarized as follows:

- When in a confined space, the pressure built-up exceeds the ability of the vessel or container to withstand that pressure, an explosion may occur.
- When in an unconfined space, if the rate of pressure rise due to the energy conversion process gradually exceeds the ability of the surroundings to diffuse the pressure rise, an explosion may occur.

2.2 Types of explosion

Accidents in the process industries can be classified into three categories: fire, explosion, and toxic release. Numerous subcategories which differ from each other in subtle ways fall under these three main categories. To carry hazard assessment and predict likely accidents and their consequences, it is important to classify the different subcategories based on their different attributes. In the case of explosions, only three kinds of energy are significant, i.e., physical, chemical and nuclear energies. Out of these, only physical and chemical energy are encountered in process industries. Figure 3 shows the proposed scheme for the classification of an explosion in chemical process industries by Abbasi et al. [26].

2. Basic Literature: An Overview

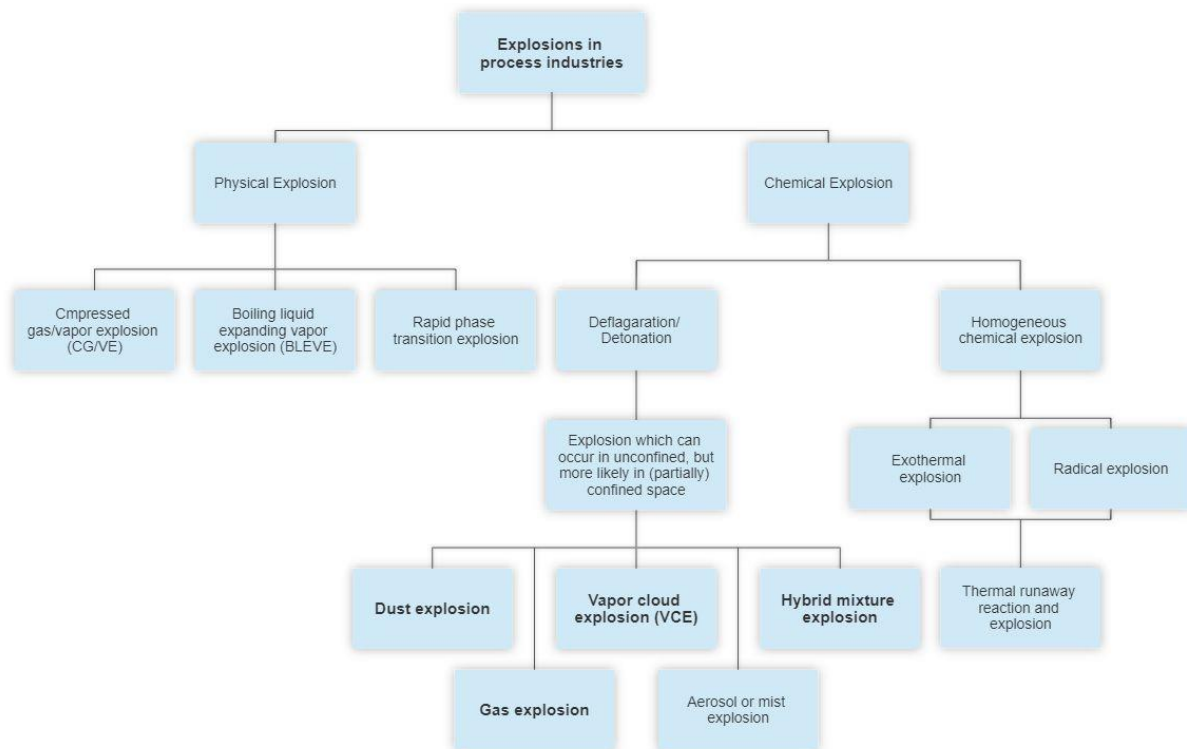


Figure 3: Classification of explosions in chemical process industries. ([26])

Explosions in process industries are primarily divided into two categories; physical explosion and chemical explosion. A physical explosion takes place when the energy accumulated is released suddenly in a physical change, such as the expansion of a compressed gas or flash vaporization of a superheated liquid. The cause of all physical explosions is purely mechanical energy. A chemical explosion is initiated due to a chemical reaction that has the potential to generate large quantities of gas in a very short period that leads to a higher rate of pressure rise that is fast enough to cause an explosion even in an unconfined space. A chemical explosion is divided into two types viz homogeneous chemical explosion and deflagration or detonation. Homogeneous explosion means the reactions are taking place homogeneously in the whole material all at once. It may be due to a sharp rise in the temperature, called an exothermal chemical reaction, or due to the formation of surplus radicals. In case the reaction is happening only in the propagating reaction zone, it can be divided into two well-defined types with different intensities; deflagration and detonation [26].

Deflagration consists of a moving exothermic reaction zone, which is sustained by heat flow from hot reaction gases to the unreacted material through radiation, convection, and conduction. Propagation in deflagration can be extremely fast, i.e., up

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to a few hundred meters per second, but still w.r.t the velocity of sound in the material, it is always subsonic. While in case of detonation, the energy required to initiate the reaction is transferred by a shock wave; hence it is a supersonic propagation. The detonation velocity can reach up to a few kilometres per second [24] [26].

As explained above, most of the chemical explosions occur in the mixture of fuel and an oxidizer. A chemical explosion is likely to be initiated if appropriate quantities of the fuel and air oxygen as an oxidizer come together at the required minimum ignition temperature in the presence of a potential or an effective ignition source. Hence, it is necessary to understand the types of chemical explosions in order to help prevent the ignition or minimize the consequences of a chemical explosion. Based on the phase or the state of aggregation of the fuels, chemical explosions are distinguished as follows [26]:

- Combustible dust fuelled – dust explosion
- Flammable gas-fuelled – gas explosion
- Flammable solvent vapor fuelled – vapor cloud explosion
- Aerosol (in the form of liquid droplets) fuelled – aerosol/mist explosion
- A mixture of two or more fuels (mentioned above) – hybrid mixture explosion

2.2.1 Dust Explosion

A dust explosion is initiated by the rapid combustion of combustible particles suspended in the air. Higher the degree of subdivision, i.e., the smaller the particle size, the more catastrophic and rapid explosive burning [30]. According to the Occupational Safety and Health Administration of the US (OSHA), combustible dust could be considered as explosive if present in finely divided form. Such dusts can be found as by-products in coal mining, wood industry, food industry [31]. If the dust cloud ignition occurs in an unconfined space, only a flash fire could be caused. But if the dust cloud is ignited in a fully or partially confined space, the heat of combustion in such a reaction may result in massive pressure built-up along with a flame propagation across the dust cloud and emission of a large quantity of heat. Besides high temperature and overpressure caused by dust explosion, emission of toxic gases is also possible in such violent chemical reactions [30][31]. Figure 4 shows the dust explosion pentagon that illustrates five major elements required for a dust explosion to occur.

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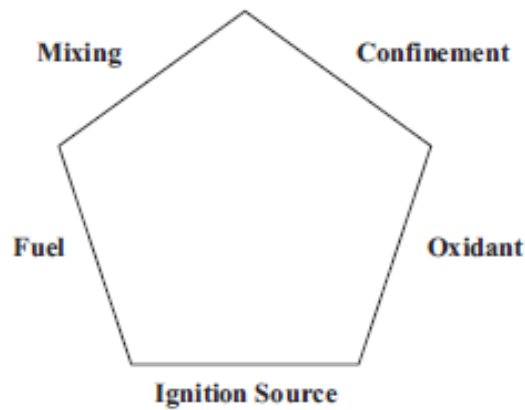


Figure 4: Dust explosion pentagon [32]

The dust explosion pentagon shown in the above figure is similar to the fire triangle. While the fire is triggered by three major factors, i.e., fuel, oxidant, and an ignition source. Dust explosion demands two more factors: mixing (of air and dust) and confinement (of dust cloud). The dust explosion pentagon is formed when these five elements occur together [30]:

- (i) Presence of combustible dust in finely divided form
- (ii) Availability of oxygen
- (iii) Presence of an ignition source
- (iv) Degree of confinement
- (v) State of mixed reactants

Dust explosions may have a domino effect, i.e., a series of explosions could be initiated after the first explosion if it has insufficient pressure release devices or venting devices or if it's design pressure too low. Hence, dust explosions can be divided into two subparts: primary explosion and secondary explosion.

The primary dust explosion is the first explosion that occurs when sufficient quantities of dust concentration and oxygen are available. Such conditions rarely build-up outside of process vessels. Hence mostly primary explosions occur within equipment (E.g., silos, dryers, filters, ducts, etc.). In reality, almost all dust explosions events occur after an initiating accident or mishandling of materials. The noticeable difference between dust and gas explosions is that a gas/vapor explosion rarely occurs inside the equipment vessel due to lack of air, however dust cloud is generally suspended in the

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air inside the equipment, which can initiate a dust explosion. A secondary dust explosion can be initiated by the disturbance of dust layers in the surrounding by the blast wave raised from the primary explosion. Such dust layers occupy very little space, but once disturbed, can form highly explosive clouds in the air. Higher-order explosions are caused by primary explosions because pressure-piling is likely to occur in the secondary explosions.

The necessary conditions that must prevail for a dust explosion to occur should be identified and summed up under the dust explosion pentagon. Efficient strategies should be applied not to allow the dust explosion pentagon to be closed. A dust explosion can be prevented by modifying the process to reduce dust handling hazards, preventing suspension of flammable dusts, complete removal or minimization of ignition sources, and inerting [30].

2.2.2 Gas/Vapor Explosion

Gas/vapor explosion can be defined as a process resulting in a rapid increase of pressure due to the combustion of premixed gas clouds i.e., air-fuel mixture or fuel-oxidizer. Gas/vapor explosions can occur inside process equipments or pipes, in buildings or offshore modules, in open process areas, or unconfined spaces. The consequences of gas explosion depend on the area in which the flammable gas cloud is present. Hence, a gas explosion is commonly classified into three types: (i) Confined gas explosions (within vessels, pipes, tunnels or channels), (ii) Partly confined gas explosions (in compartments, buildings, offshore modules), (iii) Unconfined gas explosions (in process plants or unconfined areas) [33].

A confined gas explosion can be defined as an explosion that is caused due to accidental/uncontrolled leakage of gas into closed equipment (tanks, pipes, process equipments) mixing with air. Such an explosion results in a loss of containment. Subsequent events could be strong blast waves, fires, or toxic release. The pressure built-up in such explosions can be estimated by the size of the gas cloud formed inside the enclosure. A partly confined gas explosion could be defined as explosions initiated in a partially closed environment such as compressor rooms and offshore modules, due to the mixing of partially leaked gas with air or an oxidizer. The consequences of such an explosion depend on the type of fuel, size, and concentration of the gas cloud. An unconfined gas explosion is one that occurs in an open, unobstructed area such as

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process plants. Largescale experiments have shown that an unconfined gas cloud ignited by a weak ignition source produces small overpressure resulting in flash fires. In an unconfined environment, a vapor cloud burns as a cloud fire at a steady velocity of 10-30 m/s with no noticeable overpressure. However, if an unconfined cloud detonates, high explosion pressure of the order of 20bar g is generated [7] [33]. The blast wave is the main consequence of gas explosion, as well as breaking of buildings, scattering of materials is encountered after a gas explosion [29].

Once a flammable gas or vapor has been accidentally leaked/released to the environment, for it to lead to a gas/vapor cloud explosion, the following requirements have to be met [26]:

- The released material must be flammable.
- The vapor cloud/gas cloud must mix with air to produce a sufficient mass in the flammable/explosive range of material released depending on the fuel's reactivity.
- The surrounding environment of the cloud should offer sufficient confinement for the generation of turbulence in the flow driven by expanding hot combustion gases to initiate a feedback flame acceleration process and pressure wave reflections.

Pure gases or vapors can explode during processing or handling in mining, agriculture, and pharmaceutical industry. Commonly used gases for heating or cooking such as natural gas, methane, butane, or propane, are responsible for household accidents. Gas/vapor explosion could be prevented or mitigated by avoiding flammable gas leakages, prevention of formation of explosive atmosphere, or eliminating ignition sources. Installation of external control devices like venting, blast fire barrier, flame arrestors can also help to decrease the risk of gas or vapor explosion [33].

2.2.3 Hybrid Mixture Explosion

Hybrid mixtures consist of two or more volatile components, i.e., combustible dusts, gases, and/or vapors. Such mixtures can be encountered in various industries that process or handle combustible dusts, gases, or vapors [9]. During the applications of ATEX directives or during the hazard assessment or risk assessment, such mixtures are not taken into consideration to their fullest significance. Only the materials of

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interest are considered even though there could be another side products or reactants. On the contrary, when a gas or vapor concentration is low, i.e., below their LEL, their influences are often neglected, similarly when the dust concentration is below its MEC, the analysis is done only for gas explosion while the dust presence is neglected [17].

As mentioned in section 1.2, many tests have been done concerning hybrid mixtures to study the explosion characteristics of hybrid mixtures. Hybrid mixtures are complicated because many complex physical processes happen simultaneously during the explosion and various other factors need to be considered. Hence, the effects of hybrid mixtures cannot be predicted just by overlapping the effects of single substances, i.e., only dusts, only gases, or only vapors.

Hybrid mixtures consist of flammable gas and combustible dust, both of which are present in amounts less than their respective LEL and MEC and still have a high probability of igniting if they come in contact with an ignition source. The focus when studying hybrid mixture is on the fact that in the admixture of flammable gas, the gas itself is present below its LEL. If this amount increases above the LEL, the chances for a pure gas explosion as a primary explosion increase, which increases the threat of initiating secondary explosions [34]. It is, therefore, important to study the effects of such secondary components that are present in very low quantities but still have the potential to create an explosive atmosphere. Studies have been done to investigate lower explosion limit (LEL), minimum explosible concentration (MEC), maximum explosion pressure (P_{\max}), the maximum rate of pressure rise, and few studies on minimum ignition energy (MIE) of hybrid mixtures. However, few studies have been carried out for investigation of the minimum ignition temperature (MIT) of hybrid mixtures with a hot surface as the ignition source. These investigations could help in preventing an explosive hybrid mixture atmosphere from coming into contact with a hot surface in the surrounding, which can act as an ignition source.

2.3 Minimum Ignition Temperature (MIT): Safety Characteristic

The design of safety measures in the industry and the research laboratories can be done effectively with the adequate knowledge of different safety characteristics that are directly or indirectly responsible for predicting the explosivity of combustible substances or a mixture of two or more substances. These characteristics are not physical constants, but they largely depend on the concentration of substances in the

2. Basic Literature: An Overview

dust/gas cloud. They also depend on the size and efficiency of the ignition sources. Such parameters/characteristics can help develop safe operation measures as well as explosion protection measures [34]. Critical parameters that play a vital role in the explosion of dust, gas, or vapor are explained in Table 2.

Table 2: Safety characteristics and their applications in explosion protection (Data from [35]).

Safety Characteristic	Definition	Unit	Application
K_{st}	Dust Deflagration index	bar.m/s	Measures the relative explosion severity compared to other dusts.
P_{max}	Maximum explosion overpressure generated in the test chamber	bar(g)	Used to design enclosures and predict the severity of the consequence.
$(dp/dt)_{max}$	Maximum rate of pressure rise	bar/s	Predicts the violence of an explosion. Used to calculate K_{St} .
MIE	Minimum Ignition energy	mJ	Predicts the ease and likelihood of ignition of a dispersed dust/gas cloud.
MIT	Minimum ignition temperature	°C	Predicts the probability of ignition due to hot surfaces.
MEC	Minimum explosible concentration	g/m ³	Measures the minimum amount of dust, dispersed in air, required to spread an explosion.
LEL	Lower explosion limit	Vol %	Measures the minimum amount of gas/vapor, dispersed in air, required to spread an explosion.
LOC	Limiting oxygen concentration	Vol %	Determines the least amount of oxygen required for explosion propagation through the dust cloud.

2. Basic Literature: An Overview

The explosion characteristic 'Minimum Ignition Temperature (MIT)' of a flammable dust/gas cloud can be defined as the minimum temperature necessary for igniting a dust/gas cloud in a self-propagating manner. It is applicable for dust-air mixtures, gas-air mixtures, as well as for mixtures of vapors of flammable solvent with air [5][9]. MIT of a given dust/gas cloud is not a physical constant but depends on the concentration of the dust/gas in the cloud and also on the cloud movement, i.e., turbulence and directional flow, as well as on the shape and size of the hot surface [6]. If an explosible dust/gas cloud is generated in an accidental uncontrolled manner in the vicinity of a hot surface having a temperature greater than the MIT of that cloud, it will explode and rapidly propagate the explosion throughout the cloud. Hence, it is very important to have the knowledge of MIT of commonly used dusts and gases/vapors that pose an explosible atmosphere in various industries. It can help to take adequate measures to prevent the temperatures of hot surfaces below the MIT of possible dust/gas cloud [5].

2.3.1 Factors affecting MIT

MIT is used to evaluate the ignition sensitivity, severity, and probability of a dust explosion. It is critical for the risk and hazard assessment and safety management of explosions. Therefore, accurate testing and calculation is important in the evaluation of MIT. The following are some major factors that influence the values of MIT [36]:

- Fuel/Volatile Content: The MIT values do not have intermediate fuel concentration values, rather it decreases with an increase in the fuel concentration or increasing volatile content [8].
- Moisture Content: MIT values increase with an increase in the moisture content as it becomes difficult for the dust to disperse and ignite. Also, the dust agglomerates together to form larger particles which need high temperature for ignition.
- Dust Concentration: As the dust concentration values increase within a certain range, the MIT values decrease accordingly. Measurement results are more accurate with a low concentration gradient.
- Experimental Apparatus: The MIT values obtained in the BAM oven are lower than those in the GG furnace. This is because in the BAM oven during heating, dust samples generate fumes that are more likely to get ignited than a dust cloud generated in GG furnace.

2. Basic Literature: An Overview

- Particle Size: MIT values decrease with decreasing particle size. As the total area of dust that comes in contact with the oxidizer increases in small particles, the chemical activity, and explosion severity increases.
- Oxygen Concentration: MIT can decrease sharply as the O₂ mole fraction increases, consequently increasing the explosion risk significantly. The higher the oxygen concentration, the greater becomes the probability of a destructive explosion.
- Inertant: Increase in the concentration of inerting materials increases the values of MIT until a certain threshold when no further ignition is obtained. Inerting suppression efficiency depends largely upon the types of inertants, concentration, and particle size of the inertants and the heat capacity of the inertants.

3. Experimental Setup and Procedure

The standard test procedure for determining the MIT of dust-air mixtures is defined in the standards ASTM E1491-06, 2012 [37] and EN 50281-2-1, 2007 [38]. ASTM E1491 is a particular standard that does not describe a specific test apparatus, but rather allows the user to select any one from a list of four entirely different apparatuses: (1) The Godbert-Greenwald (GG) furnace; (2) The BAM oven; (3) The heated version of the Bureau of Mines 1.2L Hartmann apparatus; and (4) Bureau of Mines 6.8L furnace. The standard test procedure for the determination of MIT of gases or vapors is described in EN 14522, 2005 [39]. A modified GG furnace is introduced in the present thesis with the aim of calculating MIT values of dust, gas, vapor, and their mixtures.

3.1 Standard Experimental Setup

The MIT of a combustible dust cloud is measured using the GG furnace. The dimensions for the GG furnace are specified by the standard EN 50281-2-1 [38]. Figure 5 shows the schematic diagram of the standard GG furnace.

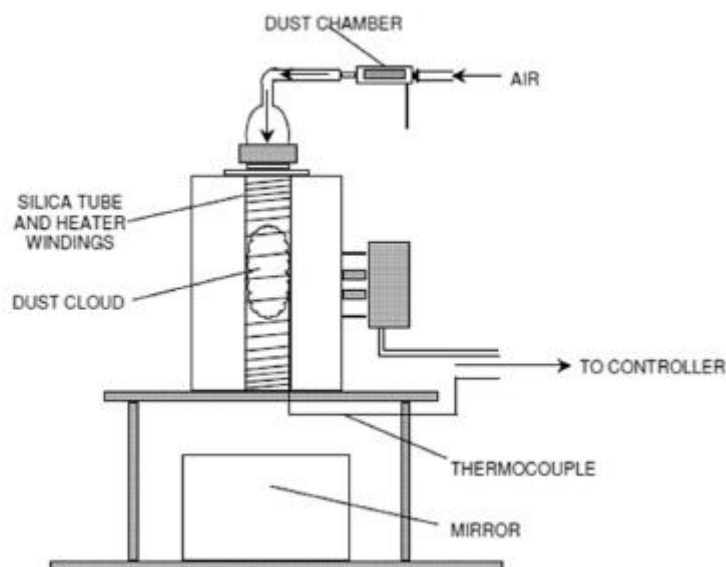


Figure 5: Schematic diagram of the standard GG furnace [5].

The cylindrical furnace in the standard size GG furnace is made up of stainless steel with an internal wall made up of silica tube that can sustain temperatures of up to 1000°C. The standard length of the cylindrical tube is 23cm, and the internal diameter 3.5cm, that is open at the bottom. The volume of the furnace is 0.234L. The wall is

3. Experimental Setup and Procedure

wounded by the resistance wire to maintain a uniform temperature throughout the furnace [5].

3.2 Modified Experimental Setup

For tests with dusts, the GG furnace or the BAM oven are used to find the MIT [38], while for tests with gases or vapors for determination of MIT, the Erlenmeyer flask is used [39]. The MIT of dusts or hybrid mixtures containing combustible dusts cannot be determined using the Erlenmeyer flask. It does not allow the dispersion of dust particles into the flask. Hence, the GG furnace had to be modified in such a way that along with dust-air mixtures, explosive gas-air mixtures or vapor-air mixtures can also be allowed into its furnace tube. With this new modification, it is possible to carry out tests for determining the MIT of two-phase or three-phase hybrid mixtures.

In the present thesis, a modified GG furnace was used, which mainly consists of a steel furnace tube, a pressure regulator, and an air reservoir. Figure 6 shows an image of the modified experimental setup, and figure 7 shows the schematic sketch for the modified experimental setup [2]. Table 3 shows the list of components in the new setup.

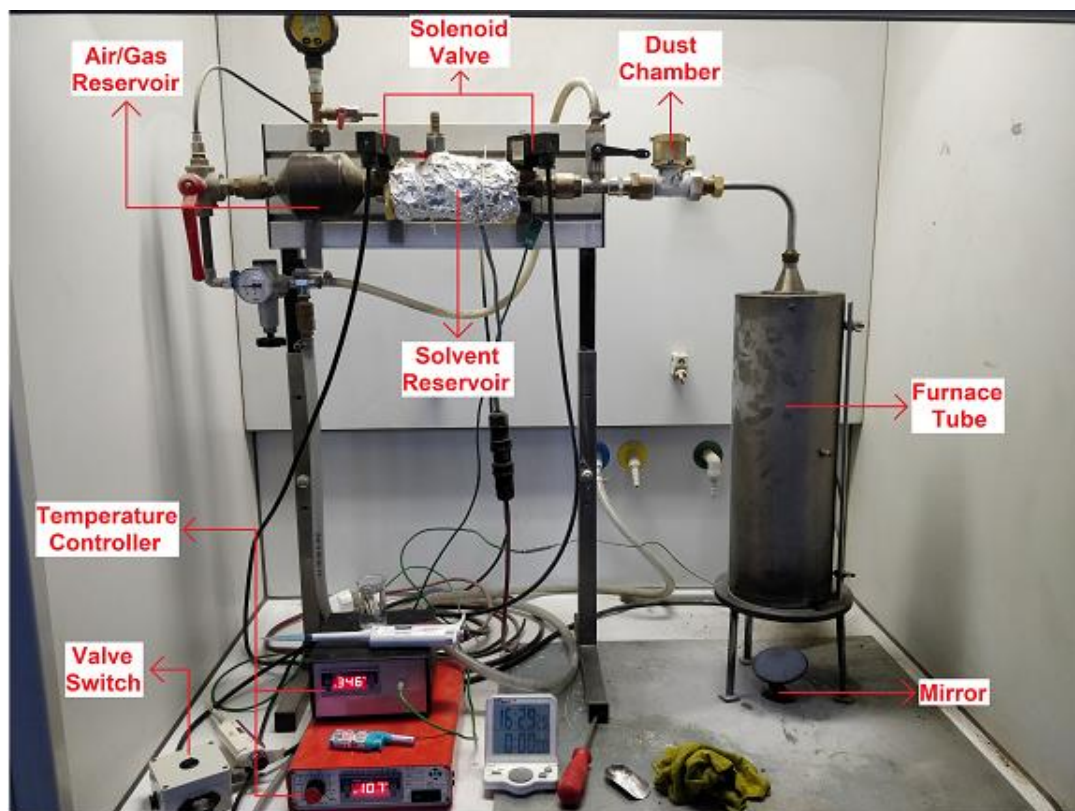


Figure 6: Image of the modified experimental setup.

3. Experimental Setup and Procedure

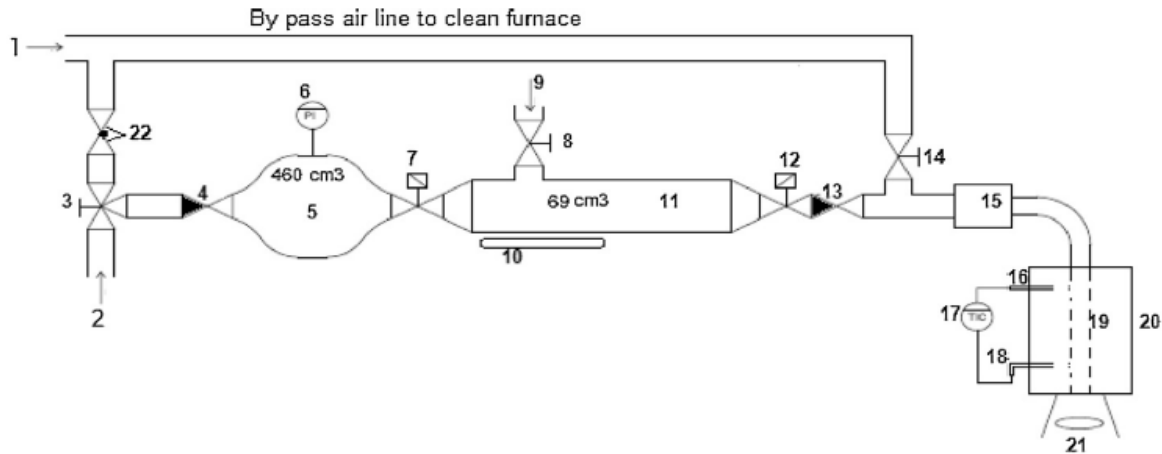


Figure 7: Schematic sketch of the modified experimental setup [2].

Table 3: List of components in the modified setup (Data from [2])

1	Air supply	12	Vacuum control valve
2	Gas supply	13	Check valve
3	T-shape ball valve	14	L-shaped ball valve
4	Check valve	15	Dust chamber/reservoir
5	Air/Gas reservoir	16	Thermocouple
6	Digital pressure gauge	17	Temperature controller
7	Vacuum control valve	18	Electric power supply
8	L-shaped ball valve (two-port)	19	Steel furnace tube
9	Solvent or liquid supply	20	Furnace shell and insulation material
10	Heating filament	21	Mirror
11	Solvent reservoir	22	Air regulating valve

A 42cm (20) long furnace tube was mounted in this furnace, which is twice the length (21.6 cm) as described in the standard EN 50281-2-1 [38] with an internal diameter of 3.5cm. The internal volume of the furnace was 460 cm³, which is heated externally by an electric chrome wire (18). The maximum allowable temperature of the furnace tube was 700°C. A mild steel case is lined with glass wool around the vertically mounted furnace tube and filled with bulk wool to act as thermal insulation. The tube is open at the bottom, and a mirror (21) is kept to observe the flame. Thermocouples (16) are placed inside of the furnace close to the inner wall, which are connected to a PID controller. The apparatus also contains an automatic air charging system and control of the air discharge from the air reservoir for dispersion of dust into the furnace [2].

3. Experimental Setup and Procedure

3.3 Generalised Experimental Procedure

For using the modified GG furnace, few prerequisite requirements must be met in order to get accurate results from the experiments:

- (1) The whole equipment should be cleaned thoroughly and should be in good operating conditions.
- (2) The furnace tube should be properly brushed and cleaned from inside, not to allow any previous residues of materials inside the tube.
- (3) Finally, the solvent reservoir and dust chamber should be flushed throughout with a clean air blast to avoid the admixing of any residual traces of materials used in previous experiments.
- (4) The syringes used in solvent injecting pipettes should be replaced each time operating with a new flammable solvent.
- (5) The operator should maintain a minimum safe distance from the furnace tube and should use personal protective gear wherever necessary.

For tests with dusts, as described by the standard, the dust sample must be pre-treated in such a way that it is homogeneous and representative of the dust to be tested. According to the standard, the same sample must be able to pass through a test sieve made of wire mesh of a square hole perforated plate with openings of 75 μm . It is also necessary to check for coarser particles or jams in the sample. However, it should not contain grains larger than 1000 μm . To avoid high dust agglomeration, the moisture content of the sample should be below 10%. Any changes during the pre-treatment operations should be noted [11] [38]. After the preliminary analysis, the tests for MIT of dusts should be carried as follows:

- (1) The furnace tube should be heated and set at a desired temperature. Pre weighed amount of dust should be placed in the dust chamber. The air reservoir should be filled with air at a desired dispersion pressure.
- (2) The dust sample should be dispersed into the heated furnace with the pressurized air blast. The criterion for an explosion should be an observation of a flame within the furnace or at the bottom of the furnace. This should be done by placing a mirror at the

3. Experimental Setup and Procedure

bottom of the furnace tube. A shower of sparks or a single spark does not cause an ignition to the dust cloud, they cannot be considered as an ignition.

(3) If no ignition occurs, the mass of the dust and pressure of air dispersion should be varied until a vigorous explosion is observed. Mass can be varied from 0.1g to 1g and air pressure can be varied from 0.1bar to 1bar above the atmospheric pressure.

(4) The condition at which a vigorous explosion is witnessed (a specific combination of mass and pressure) should be assumed as the best condition for ignition. This condition should be maintained, and the temperature of the furnace should be lowered until no flame is observed for ten continuous repetitions.

(5) According to the standard, if the igniting temperature is above 300°C, then the furnace temperature should be lowered by intervals of 20°C. If it is below 300°C, the furnace temperature should be lowered by intervals of 10°C.

(6) The minimum ignition temperature is the lowest temperature at which no flame is observed in 10 successive repetitions. When the MIT is obtained, further tests should be performed by the variance of the mass of dust and air pressure below the MIT to confirm non-ignition.

For tests with solvents, the same operating principle, as mentioned for dusts should be used. The only difference, in this case, was, the GG furnace was modified by introducing a solvent reservoir (11) to allow the input of the solvent. The solvent reservoir is heated externally with a heating filament (10) to allow the vaporization of the solvent before being dispersed into the heated furnace. The desired amount of liquid solvent should be measured accurately with a syringe of 1000 µl pipette and placed in the solvent reservoir. It should be allowed for some time to vaporize completely before being dispersed into the furnace. Meanwhile, the air reservoir (5) should be filled with air at the desired dispersion pressure. Upon opening the valve, the pressurized air blast pushes the solvent vapors into the heated furnace tube. If the concentration of the vapor-air mixture is within the explosible range, and the temperature is at the MIT or above the MIT, the mixture will be ignited.

For tests with gases, the same operating procedure should be followed as described for solvent-air testing. In this case, the only difference is that a new gas feed line (2) is introduced in the air reservoir along with the air. The solvent reservoir (11) and the dust

3. Experimental Setup and Procedure

chamber (15) should be kept empty and the air reservoir (5) should be filled with gas and air. This composition of gases is evaluated depending on the partial pressures. For example, 0.7bar is the required absolute total pressure, and if 5 vol% gas is required. The air reservoir should be first filled with 0.665bar of air followed by 0.035bar of flammable gas to make a total pressure of 0.7bar.

For tests with two-phase hybrid mixtures (two-component mixture), the same procedure should be followed as described above. The methods for tests with pure dusts and solvent vapors should be combined. The remaining steps followed for such tests should be the same as that for tests with pure dusts. The temperature decrement intervals should be of 5°C. When the MIT is obtained, further tests should be carried out at 5°C below the obtained MIT by variations of pressure and concentration to see if ignition occurs.

4. Materials and Experimental Work

4.1 Materials

Cornstarch (starch) dust and n-heptane solvent were selected in this thesis for investigating the explosion behaviour of two-component dust-vapor hybrid mixtures.

As stated by Addai [9], particle size is very important when testing for different explosion characteristics of combustible dust. As the particle size increases, the combustion rate also increases. Also, low ignition temperature and explosion limits can be seen if the degree of sub-division of the particle is high. Hence, the dust sample went through CAMSIZER particle size testing. Figure 8 shows the particle size distribution for starch dust and the percentage of particle volume/mass used. 95% of dust particles were under 50µm with a moisture content of 6.65 mass%. The molecular formula for starch dust used was $C_{27}H_{48}O_{20}$, the stoichiometric combustion reaction for starch is given by equation (1). Table 4 shows the preparatory analysis of starch dust used. Properties of n-heptane solvent are shown in Table 5.

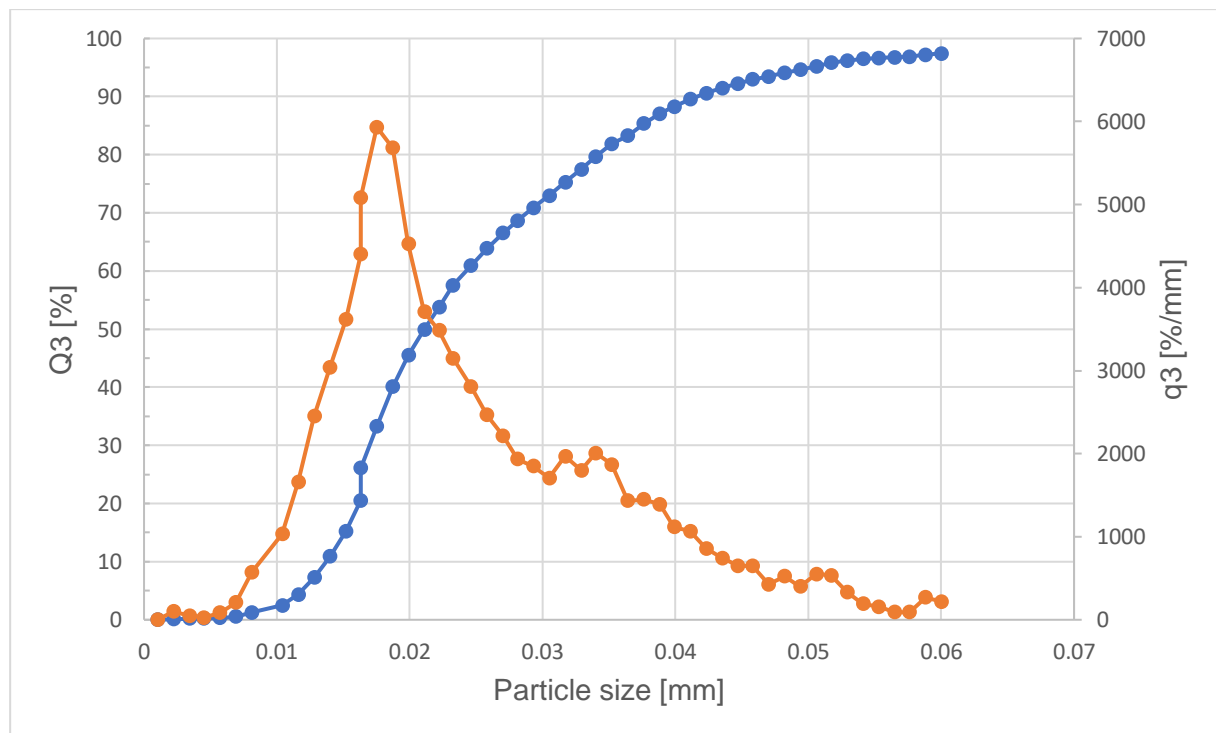


Figure 8: Particle size distribution of starch dust.

4. Materials and Experimental Work

Table 4: Preparatory analysis of starch dust [40]

Property name		Sample: Starch dust
Molecular formula		$C_{27}H_{48}O_{20}$
Particle Size (μm)	D ₃₂	17.3
	D ₅₀	21.1
	D ₉₀	41.6
Molecular weight (g/mol)		692.7
Density (g/cm ³)		1.5
Moisture content (% , mass)		6.65
Heat of combustion (KJ/Kg)		15302

Table 5: Properties of n-heptane solvent [41]

Property name		Sample: n-heptane solvent
Molecular formula		C_7H_{16}
Purity (%)		99
Density at 20°C (g/cm ³)		0.68
Boiling point (°C)		98
Melting point (°C)		-91
Explosion limit (Vol%)	LEL	0.8
	UEL	6.7 ± 5
MIT (°C)		220 ± 1.3
Maximum explosion pressure (bar)		9.4
Temperature class		T3
Explosion group		IIA

4. Materials and Experimental Work

4.2 MIT of single substances

All the tests for single substances as well as for hybrid mixtures were carried out on the modified GG furnace. As explained in section 3.3, the method described by EN 50281-2-1 [38] was followed for tests with starch dust, and it was slightly modified for testing of MIT of n-heptane and their hybrid mixture. Apart from that, in the present thesis, tests were carried out for each possible combinations of pressure-concentration. This was done with a view to study the effects of pressure and dust concentrations on the MIT of single substances and also to minimize the possible errors and to achieve accurate results. Each test with the desired pressure-concentration combination had ten repetitions.

4.2.1 MIT of Dust

The furnace was heated at a fixed starting temperature of 400°C, and the pre-weighed starch was placed in the dust chamber. To determine the MIT, the mass of starch dust sample and the air pressure were varied from 0.1g to 0.7g and 0.3bar to 0.7bar with intervals of 0.2, respectively. The furnace temperature was progressively lowered with intervals of 20°C. To study the effects of dust concentration on MIT, the dust concentrations in g/m³ were estimated using the relation shown by equation 2:

$$\text{Dust concentration (g/m}^3\text{)} = \frac{1000}{0.460} \times m \quad (2)$$

Where,

m = mass of starch sample in g

0.460 = volume of furnace in litre and 1000 = conversion factor

Table 6 shows the starch dust concentrations for different weights used.

Table 6: Starch dust concentrations for different weights used.

Mass of starch (g)	Starch concentration (g/m ³)
0.1	217
0.3	652
0.5	1086
0.7	1521
1.0	2173

4. Materials and Experimental Work

Before starting the tests, all the necessary preparations were done. The whole equipment was cleaned thoroughly, and blasts of fresh air were passed through the dust chamber to remove the previous residues. Then the following procedure was followed:

- 1) The furnace was heated to a starting temperature of 400°C and was allowed to heat uniformly for some time. 0.5g starch was placed in the dust chamber. The air reservoir was filled with air at a pressure of 0.5bar above the atmospheric pressure.
- 2) Upon activating the solenoid valves via a switch, the starch dust was then dispersed by a blast of air into the heated furnace tube. A dust cloud is formed inside the tube, which is then ignited by the hot surface around.
- 3) The ignition was confirmed and observed at the bottom of the tube through a mirror. This same test was repeated for nine more times.
- 4) Upon confirmation of the ignitions, the mass of starch was changed from 0.5g to 0.7g, 0.3g, and 0.1g, and pressure was varied from 0.5bar to 0.3bar and 0.7bar. Tests were carried out for all the possible combinations of the aforementioned masses and pressures.
- 6) The furnace temperature was then lowered by 20°C, and the same tests were carried out, as mentioned in step 1 and step 4. Upon observing similar ignitions, the temperature was further lowered by 20°C.
- 7) This was done until no ignitions were observed. The same tests were repeated below the MIT by varying pressure and concentrations for confirming non-ignition.

The flowchart of the method followed for the MIT of starch is shown in figure 9.

4.2.2 MIT of Vapor

Two syringes of capacities 100µl and 1000µl were used for accurate measurement of liquid n-heptane. The desired amount of solvent was measured and injected in the solvent reservoir, which was closed by an L-shaped ball valve to avoid any loss of solvent in the atmosphere. To allow complete vaporisation of n-heptane, the solvent reservoir was heated with a heating filament at a temperature of 105°C, which is above the boiling point of n-heptane. The furnace was set at a starting temperature of 240°C. The volume of n-heptane was varied from 275µl to 425µl with intervals of 25µl, and the

4. Materials and Experimental Work

pressure was varied from 0.3bar to 0.7bar with intervals of 0.2bar. The furnace temperature was lowered by intervals of 5°C.

Before starting the tests, all the necessary preparations were done. The Blasts of fresh air were passed through the whole equipment for removal of any previous residual materials. The dust chamber and the gas inlet were kept closed during the tests to avoid loss of air. The measurement method described by EN 50281-2-1 [38] was slightly modified, and the same procedure used for tests with dust was used. The only difference was the dust chamber was kept empty; only the solvent reservoir was used. The following procedure was then followed:

- 1) The furnace was heated at a starting temperature of 240°C, and 275µl n-heptane was placed in the solvent reservoir. The Air reservoir was filled with air at a pressure of 0.5bar.
- 2) The liquid solvent was allowed to vaporize for 50-60sec. Then upon activating the solenoid valves via a switch, the vaporized solvent was dispersed into the heated furnace tube by a blast of air. As per the standard, the same test was repeated for nine more times.
- 3) Upon observation of a flame, the solvent volume was increased by 25µl to 300µl. Keeping the pressure constant, the same procedure was followed.
- 4) The same tests with ten repetitions each were carried out for volumes 325µl, 350µl, 375µl, 400µl, 425µl at a constant pressure of 0.5bar above the atmospheric pressure.
- 5) Upon confirmation of ignitions, the furnace temperature was lowered by 5°C from 240°C to 235°C, and the same procedure as mentioned in the above steps was followed.
- 6) Repetitions were done until no further ignitions were observed. Once MIT was determined, the same tests were carried out by varying the pressure and concentrations to confirm non-ignition.

The flowchart of the method followed for the MIT of n-heptane is shown in figure 10.

4. Materials and Experimental Work

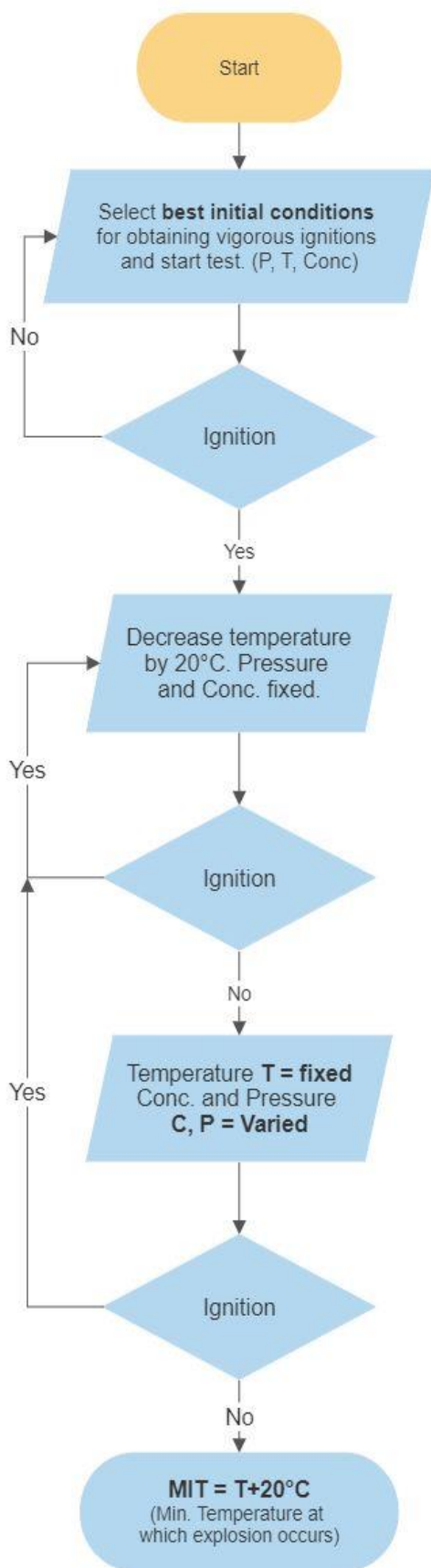


Figure 9: Flowchart of the method followed for MIT of dust.

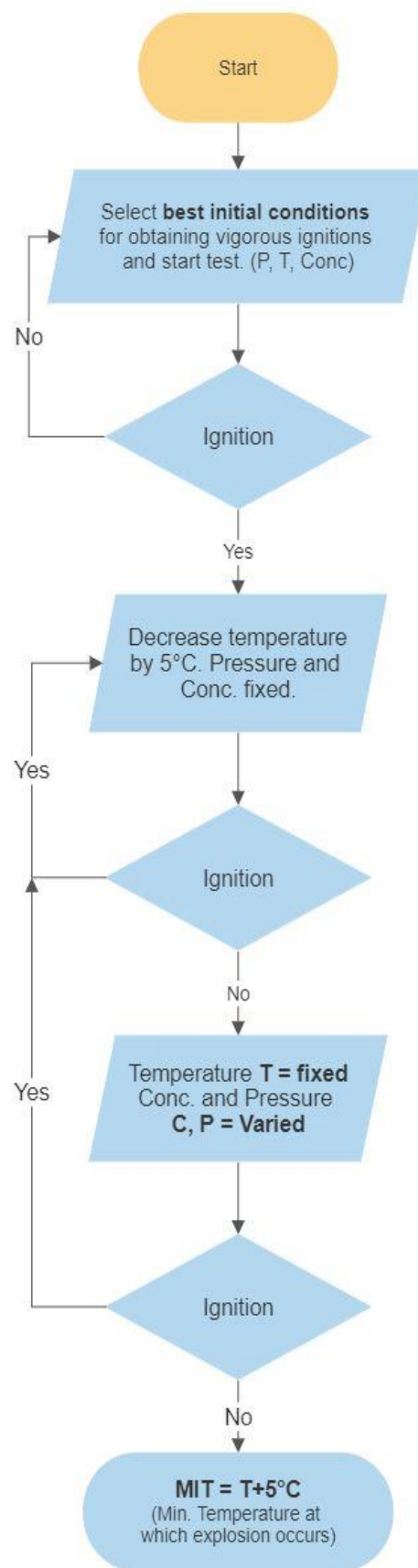


Figure 10: Flowchart of the method followed for MIT of solvent.

4. Materials and Experimental Work

4.3 MIT of hybrid mixture

The same experimental principles, as explained earlier in section 4.2.1 and section 4.2.0, were followed. In this case, the test methods for dust and solvent were combined. The remaining steps followed the same operating principle for pure dust. The starting temperatures in both the cases were kept below their individual MIT values obtained in their pure form, to see if the mixture becomes ignitable below their individual MIT. The temperature was lowered progressively with an interval of 5°C.

4.3.1 Effect of addition of vapor on MIT of dust

In this case, the mass of starch was kept constant at 1gm, which is non-ignitable at this concentration, and the volume of n-heptane added was varied. This was done to observe the effect on the MIT of dust after the addition of solvent, that formed a hybrid dust-solvent vapor mixture.

Test conditions for MIT of starch - n-heptane hybrid mixture:

Initial starting temperature of the furnace: 350°C

Temperature of solvent reservoir: 105°C

Mass of starch: 1 g (constant)

Air pressure: 0.3bar, 0.5bar, 0.7bar, 1bar

Volume of n-Heptane: 25µl, 50µl, 100µl, 200µl

No. of repetitions for each combination: 10

4.3.2 Effect of addition of dust on MIT of vapor

Similar to the previous section, to observe the effects of the addition of dust to solvent vapor, the volume of n-heptane solvent was kept constant at 240µl, which itself is non-ignitable at this concentration. The concentration of starch added was varied along with variations in pressure and temperature.

Test conditions for MIT of n-heptane - starch hybrid mixture:

Initial starting temperature of the furnace: 230°C

Temperature of solvent reservoir: 105°C

4. Materials and Experimental Work

Volume of n-heptane: 240 μ l (constant)

Air Pressure: 0.3bar, 0.5bar, 0.7bar, 1bar

Mass of starch: 0.1g, 0.3g, 0.5g, 0.7g

No. of repetitions for each combination: 10

Figure 11 shows a flowchart representing the method followed in the present thesis for testing the MIT of hybrid mixtures.

4. Materials and Experimental Work

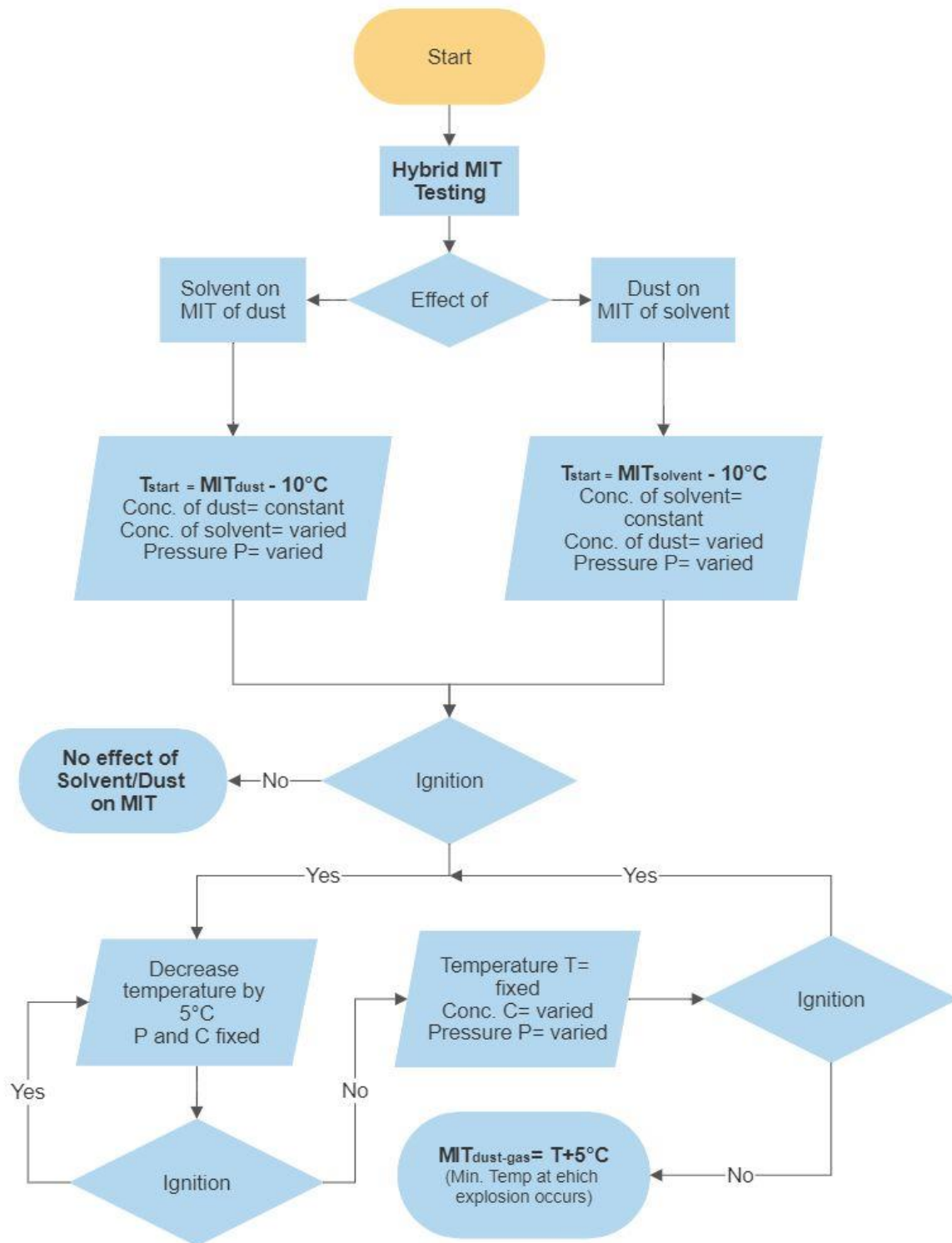


Figure 11: Flowchart representing the method followed for testing MIT of hybrid mixtures.

5. Results and Discussion

5.1 Results for MIT of single substances

Tests were carried out on a modified GG furnace by making slight modifications in the method described by the standard EN 50281-2-1, consequently, which allowed the testing of solvent as well. The results for each component are discussed further in detail.

5.1.1 Result for MIT of dust

The result obtained for MIT of starch dust clouds using a modified GG furnace is shown in figure 12. In order to validate our experimental results for the MIT of hybrid mixtures, the obtained value for MIT of pure starch is compared to available literature values. The comparison is shown in figure 12. The obtained MIT of starch is 360°C, whereas the value determined by Addai et al. [9] is 370°C and the value given in the ignition handbook is 380°C [8].

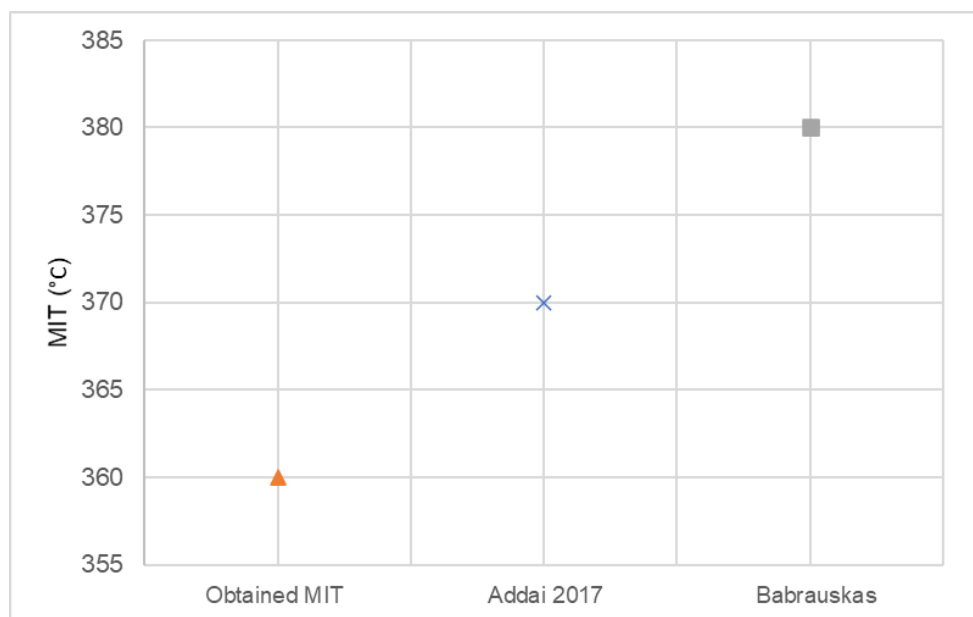


Figure 12: Obtained MIT of starch and comparison with MIT values given by Addai et al. [9] and Babrauskas [8].

The testing was done using the dust concentration variations 0.1g, 0.3g, 0.5g and 0.7g i.e. 217g/m³, 652g/m³, 1086g/m³, 1521g/m³ respectively. Figure 13 shows the effect of dust concentration on the MIT of starch. It can be clearly seen that as the dust concentration increases gradually from 217 g/m³ to 1521 g/m³, the MIT value decreases. The MIT values were observed as constant until the dust concentration

5. Results and Discussion

was progressively increased to 1521g/m^3 (0.7g). All the varied masses were ignited at 380°C at a pressure of 0.5bar above atmospheric pressure. But as the pressure was lowered to 0.3bar and temperature to 360°C , two ignitions were observed at the highest varied concentration, i.e., 1521g/m^3 . It can be concluded that the possibility of an ignition is high at lower temperatures as the concentration is increased up to a certain range. The result agrees with the argument made in section 2.3.1, which explains the effect of dust concentration on MIT.

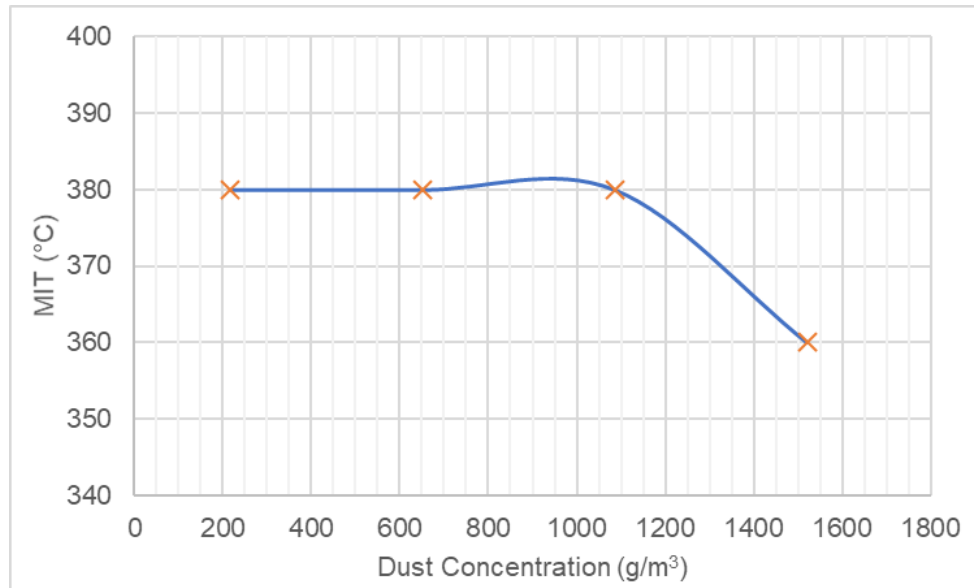


Figure 13: Effect of dust concentration on MIT of starch.

Table 7 shows the detailed results of all the tests carried out for determining the MIT of starch. The number of ignitions obtained for each variation of pressure-concentration for a total of ten repetitions is also shown in the table. Tests were started at a fixed starting temperature of 400°C . Finally, two ignitions were seen at 360°C ($P = 0.3\text{bar}$ and $C = 1521\text{ g/m}^3$). No ignitions were seen at a temperature of 340°C . Hence the previous temperature that witnessed an ignition was considered as the minimum ignition temperature of starch, i.e., 360°C .

5. Results and Discussion

Table 7: Detailed results of all tests carried out for the determination of MIT of starch.

Temperature (°C)	Starch Concentration (g/m ³)	Pressure (bar.g)	Ignition	No. of ignitions	No. of non-ignitions
400	217	0.3	N	0	10
		0.5	N	0	10
		0.7	N	0	10
	652	0.3	Y	7	3
		0.5	Y	6	4
		0.7	Y	8	2
	1086	0.3	Y	3	7
		0.5	Y	6	4
		0.7	Y	7	3
	1521	0.3	Y	4	6
		0.5	Y	5	5
		0.7	Y	5	5
380	217	0.3	N	0	10
		0.5	N	0	10
		0.7	Y	1	9
	652	0.3	Y	7	3
		0.5	Y	5	5
		0.7	Y	8	2
	1086	0.3	Y	2	8
		0.5	Y	3	7
		0.7	Y	5	5
	1521	0.3	Y	1	9
		0.5	Y	3	7
		0.7	Y	3	7
360	217	0.3	N	0	10
		0.5	N	0	10
		0.7	N	0	10
	652	0.3	N	0	10
		0.5	N	0	10

5. Results and Discussion

		0.7	N	0	10
	1086	0.3	N	0	10
		0.5	N	0	10
		0.7	N	0	10
	1521	0.3	Y	2	8
		0.5	N	0	10
		0.7	N	0	10
340	217	0.3	N	0	10
		0.5	N	0	10
		0.7	N	0	10
	652	0.3	N	0	10
		0.5	N	0	10
		0.7	N	0	10
	1086	0.3	N	0	10
		0.5	N	0	10
		0.7	N	0	10
	1521	0.3	N	0	10
		0.5	N	0	10
		0.7	N	0	10

5. Results and Discussion

5.1.2 Result for MIT of solvent vapor

Tests were carried out to determine the MIT of n-heptane solvent using the modified GG furnace according to the modified procedure described earlier. The starting temperature of the furnace was kept at 240°C. A constant pressure of 0.5bar was maintained while the concentration was varied from 275 μ l to 425 μ l. Vigorous ignitions were obtained in these tests. Hence, as per the procedure, the furnace temperature was further lowered by 5°C. No ignitions were obtained at 235°C. To confirm non-ignitions, the same tests were carried out by varying the dispersion pressure to 0.3bar and 0.7bar. Finally, the MIT of n-heptane was determined as 240°C. Figure 14 shows the MIT of n-heptane solvent. It also shows the obtained ignitions and non-ignitions during the tests.

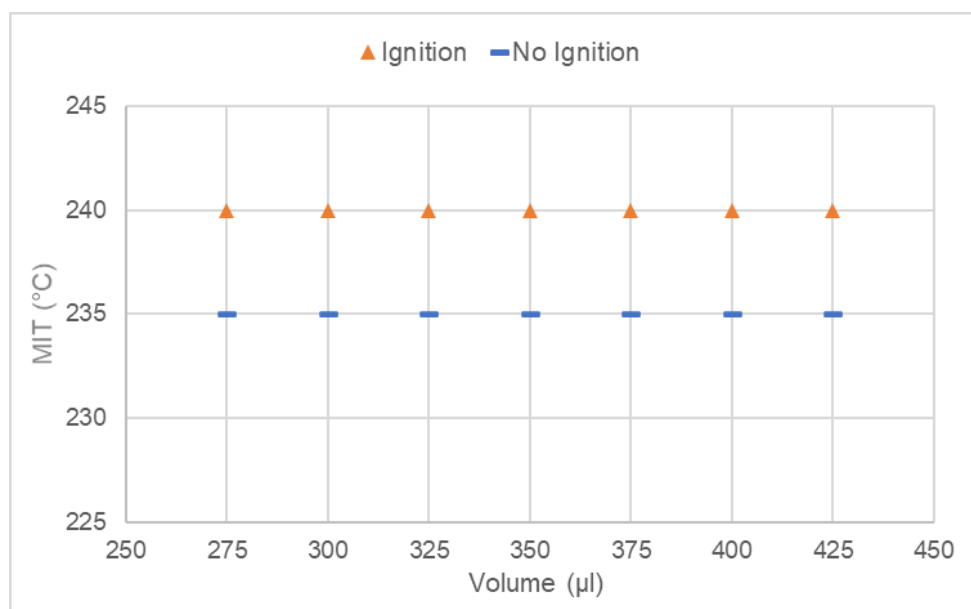


Figure 14: MIT of n-heptane solvent.

Figure 15 shows the comparison of obtained MIT of n-heptane solvent with MIT determined by Brandes et al. [41] using the Erlenmeyer flask, which is described in the standard EN 14522:2005 [39] and literature value given by Babrauskas [8]. The values agreed with work done by Brandes and Babrauskas, showing a deviation of maximum +20°C. It should be noted that the method mentioned in this work for the determination of MIT of solvent does not seek to replace the standard method used for the MIT of gases and solvents. It could be used to determine the MIT of hybrid mixtures. The reason for higher MIT value can be a result of turbulence, which intensifies the heat transfer inside the gas so that the local overheating of the gas is prevented [42].

5. Results and Discussion

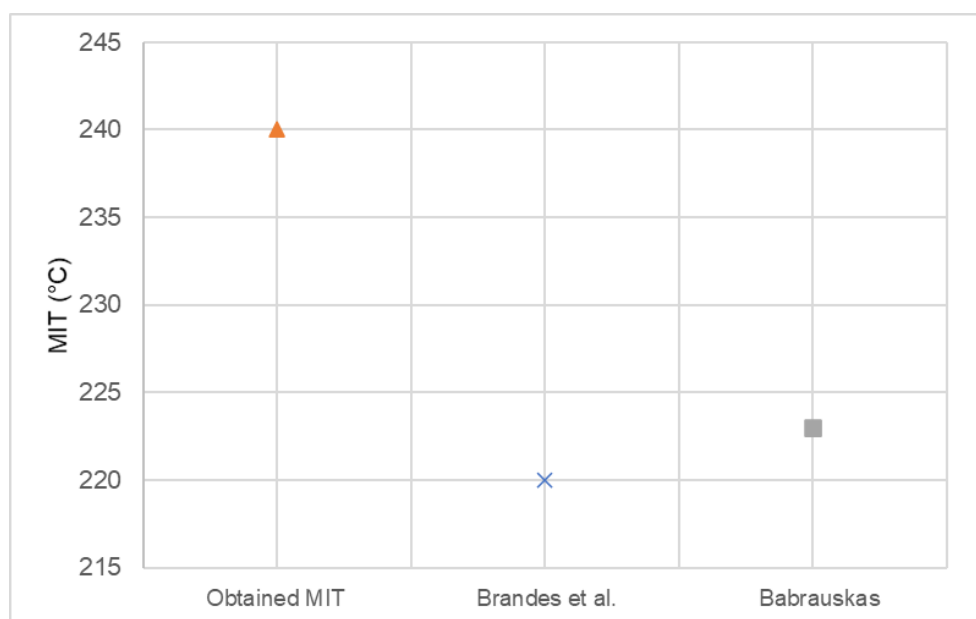


Figure 15: Obtained MIT of n-heptane and comparison with MIT values given by Brandes et al. [41] and Babrauskas [8].

Table 8 shows the detailed results of all the tests carried out to determine the MIT of n-heptane solvent. The number of ignitions obtained for each variation of pressure-concentration for ten repetitions is also shown in the table. The best ignition condition was achieved at 240°C at a constant pressure of 0.5bar. Keeping the pressure constant, the temperature was reduced by 5°C to 235°C, no ignitions were seen at 235°C. Hence, the pressure was varied along with the concentration to confirm the non-ignition of n-heptane. 240°C was the minimum ignition temperature obtained for n-heptane.

Table 8: Detailed results of all tests carried out for the determination of MIT of n-heptane.

Temperature (°C)	Pressure (bar.g)	Volume (μl)	Ignition	No. of Ignitions	No. of non-ignitions
240	0.5	275	Y	1	9
		300	Y	1	9
		325	Y	7	3
		350	Y	6	4
		375	Y	9	1
		400	Y	6	4
		425	Y	9	1

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235	0.5	275	N	0	10
		300	N	0	10
		325	N	0	10
		350	N	0	10
		375	N	0	10
		400	N	0	10
		425	N	0	10
235	0.3	275	N	0	10
		300	N	0	10
		325	N	0	10
		350	N	0	10
		375	N	0	10
		400	N	0	10
		425	N	0	10
235	0.7	275	N	0	10
		300	N	0	10
		325	N	0	10
		350	N	0	10
		375	N	0	10
		400	N	0	10
		425	N	0	10

5. Results and Discussion

5.2 Results for MIT of Hybrid mixture

MIT of a hybrid mixture of flammable solvent and combustible dust was considered. The MIT of the hybrid mixture was tested by mixing combustible dust, which is below its minimum explosible concentration or non-ignitable at that temperature, to solvent vapor, or by mixing a flammable solvent vapor to combustible dust which is below its lower explosion limit. Two variables were considered during the tests, the effect of the addition of solvent vapor on the MIT of dust and the effect of combustible dust on the MIT of solvent vapor. For this, the MIT of flammable solvent and combustible dust as a single substance was initially determined experimentally, as shown in previous sections.

5.2.1 Effect of addition of solvent vapor on MIT of dust

The tests carried out to investigate the effects of adding n-heptane solvent on MIT of starch dust were done to see if a mixture of solvent with starch dust can be ignited at a temperature that is below its minimum ignition temperature. Hence, testing was done at a starting temperature that was already below the obtained MIT of starch dust. In this case, a dust concentration of 2173g/m^3 i.e., 1g dust, was kept constant, and the volume of n-heptane was varied during the tests. The effect of the addition of n-heptane solvent to starch dust is shown in figure 16. The MIT of starch dust in its pure form was determined as 360°C . It was decreased further to 350°C after the addition of 0.35 vol%, i.e., $50\mu\text{l}$ of n-heptane, which was below its LEL value.

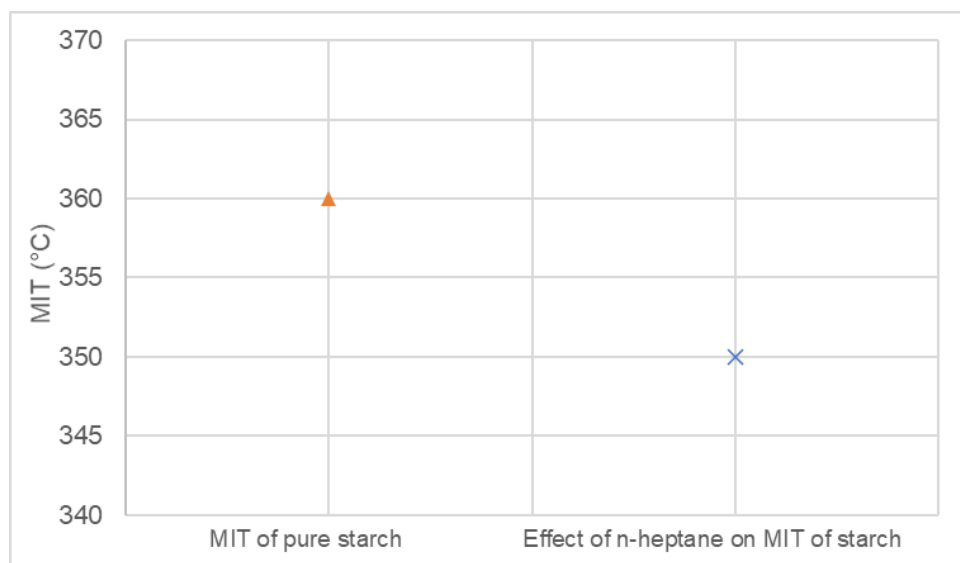


Figure 16: Effect of addition of n-heptane solvent on the MIT of starch dust.

5. Results and Discussion

Total five ignitions were observed at three different pressure-concentration combinations. The volume of n-heptane added were selected such that they themselves do not form an ignitable mixture with air in the GG furnace, they were non-ignitable. One ignition was seen at a pressure of 0.5bar when 25 μ l, i.e., 0.17 vol% of n-heptane solvent, was added. On the other hand, when 0.35 vol%, i.e., 50 μ l of n-heptane, was added to 1g of dust, i.e., concentration of 2173g/m³, three ignitions were seen at a pressure of 0.5bar above the atmospheric pressure. Another thing worth noting was that when both pressure and volume of n-heptane added were increased, it caused an ignition of starch dust. One ignition was seen at a pressure of 0.7bar above the atmospheric pressure when 0.63 vol%, i.e., 100 μ l n-heptane was added to starch dust. This could be due to the reason that, with an increase in the volume of injected n-heptane, it increased the concentration of n-heptane vapor in the mixture, which consequently increased the volatile content in the mixture. In section 2.3.1, it is mentioned how the concentration of volatile content affects the MIT value of a substance. Figure 17 depicts the ignitions observed during the testing of MIT of starch – n-heptane hybrid mixture with pressure-concentration being varied.

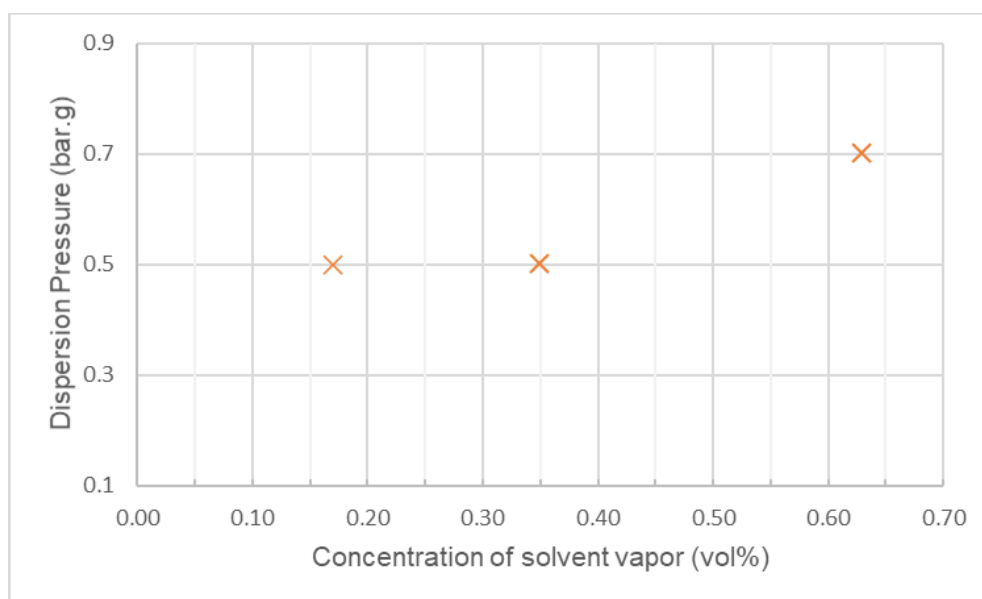


Figure 17: Observed ignitions during tests with n-heptane - starch hybrid mixture.

Table 9 shows the detailed results of all the tests carried out during the testing of n-heptane – starch hybrid mixture. The starting temperature for the tests was lowered by 10°C from the MIT of pure starch. Starch dust was ignited even below its MIT after a small amount of n-heptane was introduced in the furnace. No further ignitions were observed at lower temperature. Hence, 350°C was found as the reduced MIT of starch.

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Table 9: Detailed results of all tests carried out to determine the effect of addition of n-heptane on the MIT of starch.

Temperature (°C)	Volume of n-Heptane (μl)	Pressure (bar.g)	Ignition	No. of Ignitions	No. of non-ignitions
350	25	0.3	N	0	10
		0.5	Y	1	9
		0.7	N	0	10
		1	N	0	10
	50	0.3	N	0	10
		0.5	Y	3	7
		0.7	N	0	10
		1	N	0	10
	100	0.3	N	0	10
		0.5	N	0	10
		0.7	Y	1	9
		1	N	0	10
	200	0.3	N	0	10
		0.5	N	0	10
		0.7	N	0	10
		1	N	0	10
345	25	0.3	N	0	10
		0.5	N	0	10
		0.7	N	0	10
		1	N	0	10
	50	0.3	N	0	10
		0.5	N	0	10
		0.7	N	0	10
		1	N	0	10
	100	0.3	N	0	10
		0.5	N	0	10
		0.7	N	0	10
		1	N	0	10
	200	0.3	N	0	10
		0.5	N	0	10
		0.7	N	0	10
		1	N	0	10

5. Results and Discussion

5.2.2 Effect of addition of dust on MIT of solvent vapor

The effect of adding combustible dust on the MIT of a flammable solvent was investigated using the modified GG furnace. Testing of the hybrid mixture was done to see if flammable n-heptane solvent vapors can be ignited by the addition of combustible starch dust below its minimum ignition temperature. In this case, the starting temperature of the heated furnace was maintained below the MIT of n-heptane. The volume of n-heptane was kept constant at 240 μ l, which is non-ignitable at this volume. The mass of starch added was varied from 0.1g to 0.7g in intervals of 0.2g. The effect of the addition of starch dust on the MIT of n-heptane is shown in figure 18. The MIT of n-heptane was determined as 240°C in its pure form. It was decreased to 230°C after addition of 217g/m³, i.e., 0.1g of starch, which itself is not even ignitable at this temperature.

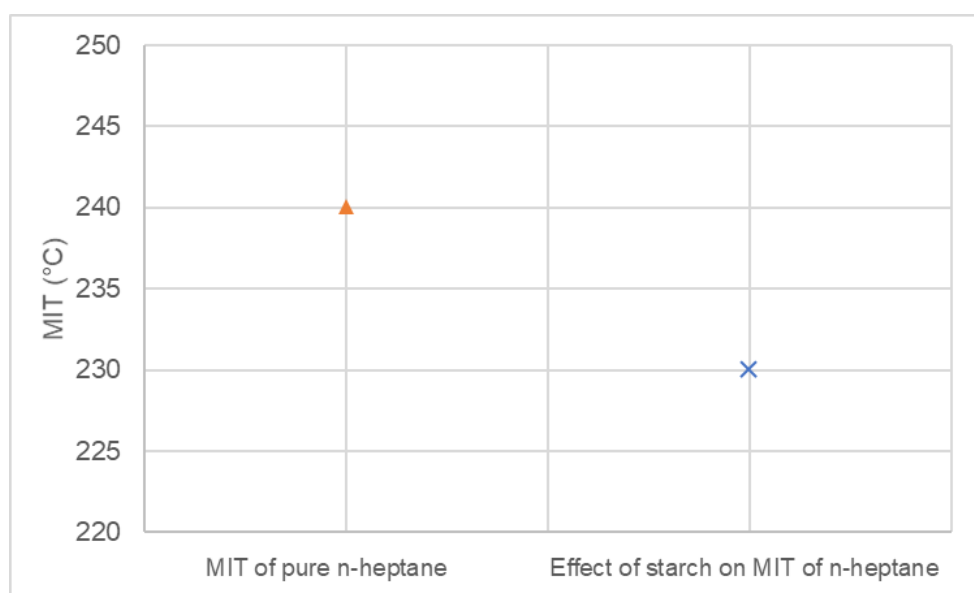


Figure 18: Effect of addition of starch dust on MIT of n-heptane solvent.

Tests were carried out by varying both pressure and dust concentration. Total five ignitions were seen under three different pressure-concentration combinations. At dispersion pressure of 0.5bar above the atmospheric pressure when 0.3g, i.e., 652g/m³ of starch dust was mixed with n-heptane, two ignitions were seen out of ten repetitions. No ignitions were observed at a pressure lower than 0.5bar. But, on the other hand, when the dispersion pressure was set to 0.7bar above the atmospheric pressure, two ignitions were seen when a very small quantity, i.e., 0.1g or 217g/m³ of starch dust was introduced in the furnace along with a constant volume of 240 μ l n-

5. Results and Discussion

heptane. Also, on ignition was observed when 0.3g starch was added to n-heptane the same pressure.

These results are found to agree with the results of Addai [9]. Addai et al. found a similar effect of adding a small amount of combustible dust to a flammable solvent. The minimum ignition temperature is reduced by introducing a small amount of dust to flammable solvent vapors. Figure 19 shows the observed ignitions during tests with starch – n-heptane hybrid mixtures.

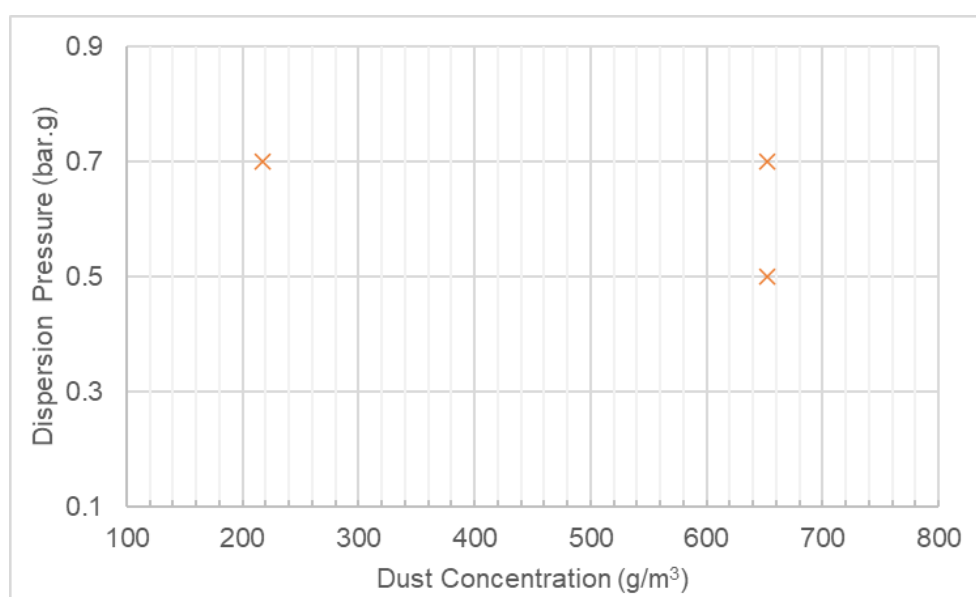


Figure 19: Observed ignitions during tests with starch – n-heptane hybrid mixtures.

Table 10 shows the detailed results of all the tests carried out for testing the effects of adding starch on the MIT of n-heptane. The starting temperature was lowered by 10°C below the obtained MIT of n-heptane. This was done to test the ignitability of n-heptane solvent, which is below its LEL when a small amount of dust is added to it, which itself is below its MEC. It was found that the addition of starch dust has an influence on the MIT of n-heptane. MIT decreased by 10°C by adding starch dust. The reason for this can be the production of volatile matter or combustible gases after the organic particles present in the dust are heated. Such volatile matter formation could be due to the pyrolysis or devolatilization of the dust to a gas. This volatile matter gets added to the solvent vapors, which in turn results in increasing the ignitability of the mixture. Table 10 shows the detailed results of all tests carried out to determine the effect of addition of starch on the MIT of n-heptane.

5. Results and Discussion

Table 10: Detailed results of all tests carried out to determine the effect of addition of starch on the MIT of n-heptane.

Temperature (°C)	Mass of starch (g)	Pressure (bar.g)	Ignition	No. of Ignitions	No. of non-ignitions
230	0.1	0.3	N	0	10
		0.5	N	0	10
		0.7	Y	2	8
		1	N	0	10
	0.3	0.3	N	0	10
		0.5	Y	2	8
		0.7	Y	1	9
		1	N	0	10
	0.5	0.3	N	0	10
		0.5	N	0	10
		0.7	N	0	10
		1	N	0	10
	0.7	0.3	N	0	10
		0.5	N	0	10
		0.7	N	0	10
		1	N	0	10
225	0.1	0.3	N	0	10
		0.5	N	0	10
		0.7	N	0	10
		1	N	0	10
	0.3	0.3	N	0	10
		0.5	N	0	10
		0.7	N	0	10
		1	N	0	10
	0.5	0.3	N	0	10
		0.5	N	0	10
		0.7	N	0	10
		1	N	0	10
	0.7	0.3	N	0	10
		0.5	N	0	10
		0.7	N	0	10
		1	N	0	10

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5.3 Error Analysis

The results obtained during the testing of the MIT of single substances show little deviation from the available literature values. The obtained value for MIT of starch dust cloud in this thesis is 360°C, which is lower by 20°C from the literature value given by Babrauskas [8]. The reason for this can be the modification made in the GG furnace used in this thesis. The length of the heated furnace tube of the furnace was twice the length described in the standard EN 50281-2-1 [38], this may have caused the dust to remain for more period of time in the heated atmosphere as compared to the standard GG furnace. Hence, this could increase the probability of ignition of the dust cloud even at a temperature lower than the literature MIT value. The tests for MIT of starch dust were carried according to the standard procedure, where the temperature was progressively lower by 20°C as prescribed by the standard. Ignitions were seen at 360°C. Hence the temperature was lowered to 340°C where no ignitions were observed. Hence, 360°C was decided as the MIT of starch. In order to get more accurate value of MIT, the tests can be repeated with intervals of 5°C from 360°C to 340°C.

The result for MIT of n-heptane solvent was obtained on the GG furnace by modifying the equipment as well as the standard procedure used for testing for MIT of dust clouds of combustible dusts. The result showed a maximum deviation of 20°C from the available MIT value obtained by the standard procedure described by EN 14522:2005 [39], which is used for testing for MIT of gases and vapors. Also, it should be noted that the procedure followed in this thesis does not seek for a replacement to the standard procedure. Rather, it could be used for the testing of hybrid mixtures consisting of combustible dusts and flammable gases/solvents. According to Hattwig et al. [42], turbulence could also be a reason for the higher value of MIT. It intensifies the heat transfer inside the furnace tube, which prevents the local overheating of gases. Another reason for obtaining a greater MIT value could be the improper vaporization of n-heptane liquid solvent or due to loss of vapors from the solvent reservoir, which could result in less concentration of volatile matter in the vapor-air mixture inside the furnace.

Test conditions of hybrid mixtures largely depended on the results of the MIT of single substances. Hence, the results obtained for tests with hybrid n-heptane-starch

5. Results and Discussion

mixtures were according to the results obtained in the tests with single substances. The overall results for the effects of the addition of dust/solvent to another solvent/dust to form a hybrid mixture obtained in this work agree with the results of Addai et al. 2016 [2], 2017 [9], which conclude that both dust and vapors can be ignited below their minimum ignition temperature if a second component is added below its minimum ignition concentration or lower explosion limit.

5.4 Suggestion of general testing procedure

Some basic points are suggested to determine the MIT of hybrid mixtures, and few modifications/additions in the present procedure are suggested as follows:

- 1) It should be made mandatory to manually clean the dust chamber and the L-shaped nozzle that connects the dust chamber to the furnace tube each time before starting a new experiment with different materials. Also, thorough cleaning is suggested after 50 successive repetitions irrespective of the materials tested. Thus, most of the residual substances will be removed, giving more accurate results.
- 2) The residence time for the vaporization of the liquid solvent in the solvent chamber should be of at least 120-150 seconds. This will allow the solvent to vaporize completely, leaving no traces of partially vaporized or non-vaporized liquid. Otherwise, if it is not vaporized completely, it may increase the moisture content when mixed with dust, affecting the MIT of a dust/vapor/air mixture.
- 3) In this work, it was found that sometimes when the dust chamber was kept open for more than a minute, the moisture content inside the dust chamber would sometimes increase, which resulted in the deposition of dust on the inner walls of the dust chamber and nozzle connecting the chamber to the furnace tube. Hence, it is suggested to avoid unnecessary opening of the dust chamber and keep the dust storage container closed all the time unless needed.
- 4) The temperature of the furnace lowered during the tests until no ignitions are observed, should be in the intervals of a minimum of 5°C to a maximum of 10°C instead of 20°C. Also, tests should be carried at intervals of 2°C (if the primary interval is 10°C) or 1°C (if the primary interval is 5°C). This could help in getting more accurate results. For this, the starting temperature for the testing should be a maximum of 25°C above the literature/theoretical value. Since in many pieces of research, it has been found

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that values obtained in GG furnace usually deviate from 5°C to 20°C from the available literature.

5) A particular minimum number of ignitions out of ten repetitions should be set as a primary requirement for a pressure-concentration combination to consider as an ignition. E.g., Minimum three or five ignitions are necessary for a combination of a particular pressure-concentration to be considered as an ignition. This is because sometimes unexpected/accidental ignitions are observed at such pressure-concentration, which generally would be non-ignitable. This generally happens at dispersion pressure lower than 0.5bar, when sometimes the whole volume in the solvent and dust chamber is not dispersed into the furnace tube due to low pressure. Such a scenario was observed in this work, in which some moist dust particles settled/accumulated near the dust chamber or in the corners of the nozzle or near the upper opening of the furnace tube. Hence, these accumulations resulted in the distortions of the concentrations in the latter tests, which had to be repeated again.

6. Conclusion

Hybrid mixture explosions are usually encountered in many industries that process, handle or produce combustible dusts, flammable gases, or solvents. Currently, there are no universal set of values of MIT for hybrid mixtures that could be referred to as standard values, owing to the complexity involved in such mixtures. MIT is not a physical constant. Rather it largely depends on the dust concentration, turbulence of the dust cloud, and size-shape of the hot surface. Hence, while designing safety and control systems, these values cannot be directly derived theoretically. To prevent or mitigate such ignition hazards, deeper knowledge is required about the behaviour of substances when mixed with another substance that can lead to an explosion when present in a heated environment.

The main purpose of this work was to extend the knowledge about the ignition of hybrid mixtures and to make the dependence of explosibility or the probability of an ignition on the minimum ignition temperature more comprehensible while dealing with two-phase hybrid mixtures. The effect of dust concentration on MIT was investigated. MIT decreases when the dust concentration is increased up to a certain value. It was found that there can be an influence on MIT when two substances of different phases are mixed. The MIT of starch dust was reduced by 10°C when a small amount of n-heptane solvent was added to it, which was below its LEL or which was non-ignitable in its single form. Similarly, the addition of a small quantity of starch dust below its MEC to n-heptane solvent reduced the MIT of n-heptane by 10°C. Hence, it can be said that the probability of an ignition can increase even if a second component is added in a small amount or even below its own MIT.

Results from the present work don't show very strong effects on MIT, as not many ignitions were seen during the testing of hybrid mixtures. Hence, not all the results can be thoroughly explained. As a result, more combinations are needed to be tested before coming to a general conclusion about the ignition of hybrid mixtures. Based on the results obtained in this work, the following primary conclusions can be made:

- MIT of dust can be decreased when a small amount of solvent is added, which is either below its LEL or non-ignitable at the temperature at which it is added, or vice-versa.

6. Conclusion

- The likelihood of an ignition in a two-component mixture increases even when both the components are present below their individual MIT and/or individual LEL/MEC.
- MIT of hybrid mixtures cannot be directly predicted just by over-lapping the effects of single substances.

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