

EXPERIMENTAL STUDY OF POLYETHYLENE AND SULFUR
DUST EXPLOSION CHARACTERISTICS

A Thesis

by

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ABSTRACT

Polyethylene and sulfur are widely used substances in today's industries, and therefore, the assessment and control of dust explosion risks associated with their production, storage, and usage are of paramount importance to ensure the safety standards in these industries. Since the petrochemical industry in Qatar is growing vastly to accommodate the Qatar National Vision for 2030, process industries in Qatar face challenges to control the hazards and risks of an explosion of both polyethylene and sulfur dusts which are generated in the polyolefin production process and in the desulfurization units of the gas processing plants, respectively.

Prevention of polyethylene and sulfur dust explosions and the mitigation of their consequences require a deep understanding of the explosive properties of these dusts. This knowledge can be used to reduce the frequency of dust explosions and to minimize the severity of explosions, therefore controlling the risks associated with polyethylene and sulfur dust explosions. Very few data exist in literature regarding dust explosibility properties for high density polyethylene (HDPE) dust and sulfur dust explosions. This experimental work focused on the determination of one of these explosive properties, called the minimum explosion concentration (MEC), for both polyethylene and sulfur dust in the context of the Qatar industry.

To achieve the objectives of this research, the work was divided into three key phases:

- Phase I: Analysis of physical characteristics of both polyethylene and sulfur dust found in local industries.
- Phase II: Experimental determination of MEC values for these dusts using a modified Hartmann tube and a 20-liter sphere.
- Phase III: Determine correlational relationship between dust particle size and MEC of both polyethylene and sulfur dust.

Results of this work gave insights regarding the explosibility of dust samples used in local plants at Qatar. This research generates some new MEC data for both HDPE and sulfur dust using a modified Hartmann tube. It was shown for both sulfur and polyethylene dust, that at smaller particle sizes ($<100 \mu\text{m}$), the particle size does not have a strong influence on MEC values. For larger particle sizes, MEC will clearly increase with particle size.

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I wish to dedicate this thesis first to my parents. I pray that this achievement and the further opportunities which arise from it repay a small part of their sacrifice to make me who I am. I wish to also thank my spouse, Humaira Tasnim, for her immense patience, support, and unconditional love, which she has given me throughout my academic career and life, without which I would not be able to complete this research. Lastly, all the opportunities I received in my life were a gift from Allah, and all praise belongs to Him in opening up doors for me beyond my wildest imaginations.

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Contributors

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This work was supervised by a thesis committee consisting of Professor Luc Véchet and Professor Sam Mannan of the Department of Chemical Engineering and Professor Hazem Nounou of the Department of Electrical Engineering.

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The data analyzed in Section 5.2.3 and Section 5.4 was provided by research carried out by Dr Walid Khalfaoui, Atif Ashraf, and Jack Altwal at the Qatar consortium of the Mary Kay O' Connor Process Safety Center.

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NOMENCLATURE

ASTM	American Society for Testing and Materials
CSB	U.S. Chemical Safety and Hazard Investigation Board
dP/dt	Rate of Pressure Rise
$[dP/dt]_m$	Maximum Rate of Pressure Rise
HDPE	High Density Polyethylene
HSE	Health and Safety Executive
K_{St}	Dust Deflagration Index
LDPE	Low Density Polyethylene
LFL	Lower Flammability Limit
LLDPE	Linear Low Density Polyethylene
LOC	Limiting Oxygen Concentration
MDPE	Medium Density Polyethylene
MEC	Minimum Explosible Concentration
MIE	Minimum Ignition Energy
MIT	Minimum Ignition Temperature
MKOPSC	Mary Kay O'Connor Process Safety Center
MSDS	Material Safety Data Sheet
NFPA	National Fire Protection Association
P_m	Maximum Pressure
P_{max}	Maximum Explosion Overpressure

PP	Polypropylene
QAPCO	Qatar Petrochemical Company
Q-Chem	Qatar Chemical Company
UFL	Upper Flammability Limit
μm	Micrometer
$^{\circ}\text{C}$	Celsius

TABLE OF CONTENTS

	Page
ABSTRACT	ii
ACKNOWLEDGEMENTS	iv
CONTRIBUTORS AND FUNDING SOURCES.....	v
NOMENCLATURE.....	vi
TABLE OF CONTENTS	viii
LIST OF FIGURES.....	xi
LIST OF TABLES	xiv
1 INTRODUCTION	1
1.1 Dust explosion incident statistics	1
1.2 Polyethylene & sulfur production and dust explosion issues.....	5
1.2.1 Polyethylene production and dust explosion issues.....	5
1.2.2 Sulfur production and dust explosion issues.....	6
1.3 Motivation for the project	7
2 FUNDAMENTALS OF DUST EXPLOSIONS.....	8
2.1 Definition of a dust.....	8
2.2 Dust explosion pentagon and dust explosion mechanism.....	9
2.3 Primary and secondary explosions	13
2.4 Prevention and protection strategies	14
2.5 Dust explosibility characteristics	16
2.5.1 Qualitative assessment of dust explosibility in a modified Hartmann tube.....	16
2.5.2 Quantitative assessment of dust explosion characteristics.....	18
2.5.3 Experimental assessment of dust explosion properties.....	21
3 LITERATURE REVIEW OF POLYETHYLENE AND SULFUR DUST PROPERTIES	27
3.1 Polyethylene dust explosion properties.....	27
3.1.1 Minimum Explosible Concentration (MEC) of polyethylene dust.....	27
3.1.2 Maximum pressure (P_m) and rate of pressure rise (dP/dt) _m of polyethylene dust	31

3.1.3	Maximum Explosion Overpressure (P_{max}) and Dust Deflagration Index (K_{St}) of polyethylene dust	36
3.1.4	Minimum Ignition Energy (MIE) of polyethylene dust.....	42
3.1.5	Minimum Ignition Temperature (MIT) of polyethylene dust.....	44
3.2	Sulfur dust explosion properties.....	48
3.2.1	Minimum Explosible Concentration (MEC) of sulfur dust	49
3.2.2	Maximum pressure (P_m) and rate of pressure rise (dP/dt) _m of sulfur dust.....	50
3.2.3	Maximum Explosion Overpressure (P_{max}) and Dust Deflagration Index (K_{St}) of sulfur dust	51
3.2.4	Minimum Ignition Energy (MIE) of sulfur dust	52
3.2.5	Minimum Ignition Temperature (MIT) of sulfur dust	52
3.2.6	Limiting Oxygen Concentration (LOC) of sulfur dust	55
3.3	Summary of the literature review	55
3.3.1	Polyethylene dust explosion properties	55
3.3.2	Sulfur dust explosion properties	57
4	RESEARCH OBJECTIVES AND METHODOLOGY	59
4.1	Research objectives	59
4.2	Methodology	60
4.2.1	Phase I: Analysis and physical characterization of the polyethylene and sulfur dusts found in the local industries of Qatar	60
4.2.2	Phase II: Experimental determination of the MEC for both polyethylene and sulfur dust collected on-site.....	66
4.2.3	Phase III: Study of the effect of particle size distribution on the MEC values of polyethylene and sulfur dust.....	76
5	EXPERIMENTAL RESULTS AND DISCUSSION	80
5.1	Experimental results for sulfur dust	80
5.1.1	Phase I: Analysis and physical characterization of sulfur dusts found on-site	80
5.1.2	Phase II: Experimental determination of the minimum explosible concentration (MEC) for sulfur dust collected on-site.....	91
5.1.3	Phase III: Effect of particle size distribution on the MEC values of sulfur dust.....	97
5.2	Experimental results for polyethylene dust.....	102
5.2.1	Phase I: Analysis and physical characterization of polyethylene dusts found in the local industries of Qatar	102
5.2.2	Phase II: Experimental determination of the minimum explosible concentration (MEC) for polyethylene dust collected on-site	110
5.2.3	Phase III: Effect of particle size distribution on the MEC values of polyethylene dust	117

5.3	Discussion on manual sieving vs. mechanical sieving.....	120
5.3.1	Experimental results with manually sieved samples of polyethylene dust.....	122
5.3.2	Experimental results with manually sieved samples of sulfur dust ..	123
5.4	Comparison of results using a modified Hartmann tube vs 20-liter sphere for a sample of sulfur dust	124
6	CONCLUSIONS	127
6.1	Sulfur dust	127
6.2	Polyethylene dust	128
6.3	Future research	129
	REFERENCES.....	130
	APPENDIX	135

LIST OF FIGURES

	Page
Figure 1. Distribution of dust explosions by material – United Kingdom (1979-1988)....	2
Figure 2. Dust explosions in the United States, 1980-2005	4
Figure 3. Fatalities from dust explosions in United States, 1980-2005	5
Figure 4. Dust explosion pentagon.....	9
Figure 5. Mapping the dust explosion process in sequence	11
Figure 6. Explosion mechanism of dust particles	12
Figure 7. Detailed modified Hartmann tube illustration	18
Figure 8. Dust explosion pentagon in relation to dust explosion properties	19
Figure 9. Illustration of 20-liter dust explosion sphere	22
Figure 10. Mike-3 apparatus	25
Figure 11. Godbert-Greenwald furnace schematic.....	26
Figure 12. Lean flammability limit for polyethylene powder as a function of particle size	29
Figure 13. Variation of MEC with particle size for polyethylene dust.	30
Figure 14. Effect of turbulence on P_m for different dusts	33
Figure 15. P_m data for polyethylene dusts compared with those of methane gas	35
Figure 16. Effect of atmosphere humidity on P_m and $(dP/dt)_m$ for polyethylene dust.....	36
Figure 17. Effect of dust particle size on P_{max} for polyethylene dust.....	39
Figure 18. Effect of particle size on K_{St} of a polyethylene dust sample: ●, average diameter 20 μm ; ○, average diameter 56 μm ; □, average diameter 98 μm ; ■, average diameter 116 μm	40
Figure 19. Effect of particle size on K_{St} of ○ polyethylene and △ silicon.....	40
Figure 20. Effect of particle size on MIE of polyethylene dust.	43

Figure 21. Autoignition data from the 1.2-liter furnace plotted as temperature versus concentration:●, ignitions; ○, non-ignitions (particle size 37 μm).....	44
Figure 22. Variation of MIT with dust concentration for polyethylene (particle size between 106-125 μm).....	45
Figure 23. Particle size dependence for the MIT for polyethylene	47
Figure 24. Influence of dust concentration on P_m and $(dP/dt)_m$	50
Figure 25. Effect of dust concentration on ignition temperature of sulfur dust	53
Figure 26. Influence of water content on ignition energy and ignition temperature of sulfur dust	54
Figure 27. Beckman Coulter LS 13 320 particle analyzer	62
Figure 28. Typical particle size range analysis results	63
Figure 29. SEM pictures of a polyethylene dust sample.....	64
Figure 30. FEI Quanta 400 Scanning Electron Microscope (SEM).....	65
Figure 31. Rigaku ZSX Primus II WDXRF X-ray fluorescence spectrometer.....	65
Figure 32. TAMUQ's modified Hartmann tube.....	67
Figure 33. Schematic of a modified Hartmann tube	68
Figure 34. Schematic of pierced lid positions and its qualitative assumptions (modified Hartmann tube)	68
Figure 35. Picture of the modified Hartmann tube during an MEC measurements	69
Figure 36. Pictorial representation of TAMUQ 20-liter dust explosion sphere	74
Figure 37. Schematic of 20-liter dust explosion sphere	75
Figure 38. Pestle and mortar for dust grinding.....	77
Figure 39. TAMUQ mechanical siever (RoTap RX-29 Sieve-Shaker)	79
Figure 40. Graphical illustration of process for granulated sulfur samples	81
Figure 41. Graphical illustration of process for prilled sulfur samples.....	83
Figure 42. Particle size characterization of sulfur dust collected on site	86

Figure 43. Explosibility results for on-site sulfur dust samples	93
Figure 44. Pictures of the fire / explosion of sulfur dust (collected on-site) in the modified Hartmann tube	96
Figure 45. Graphical summary of explosibility results of sieved sulfur samples	99
Figure 46. Particle size characterization of polyethylene dust collected on-site.....	106
Figure 47. Explosibility results for on-site polyethylene dust samples.....	111
Figure 48. Pictures of the fire / explosion of polyethylene and additives dust (collected on-site) in the modified Hartmann tube	114
Figure 49. Graphical summary of explosibility results of sieved polyethylene samples	119
Figure 50. MEC measurement for QG_Sulfur_WC using a 20-liter sphere	126

LIST OF TABLES

	Page
Table 1. Dust explosions in West Germany, 1965-1980: Fatalities and injuries in a sample of 357 explosions.....	3
Table 2. Dust explosions in the United States, 1900-1956: Fatalities, injuries, and material losses in a sample of 1123 explosions	3
Table 3. Polyethylene production capacity in Qatar	6
Table 4. Means of preventing and mitigating dust explosions.....	16
Table 5. Dust explosion classification based on K_{St} value.....	20
Table 6. Minimum Explosible Concentration (MEC) of polyethylene dust	28
Table 7. 20-liter sphere experiment with polyethylene P_m and $(dP/dt)_m$	32
Table 8. P_{max} and K_{St} of polyethylene dust	37
Table 9. Minimum Ignition Energy (MIE) of polyethylene dust	42
Table 10. Minimum Ignition Temperature (MIT) of polyethylene dust	46
Table 11. Dust explosion properties of sulfur dust	48
Table 12. Limiting Oxygen Concentration (LOC) of sulfur dust.	55
Table 13. Summary of effect of critical parameters on polyethylene dust explosion properties	57
Table 14. Origin and identification of sulfur samples.....	82
Table 15. Origin of prilled sulfur samples	84
Table 16. Summary of particle sizes for granulated sulfur samples	84
Table 17. Chemical composition of sulfur dust samples	90
Table 18. Summary of explosibility results for on-site sulfur dust samples	92
Table 19. Summary of explosibility results of sieved sulfur samples.....	98
Table 20. Location and identification of the dust samples from the polyethylene plant.....	103

Table 21. Summary of particle sizes for polyethylene and additive samples	105
Table 22. Summary of explosibility results for on-site polyethylene dust and additive samples	110
Table 23. Summary of explosibility results for sieved polyethylene samples	118
Table 24. Explosibility results of manually sieved polyethylene dusts	123
Table 25. Determination of MEC in 20-liter sphere (ASTM E1515)	125

1 INTRODUCTION

Dust explosion is one of the most common and widespread hazards faced by the process industry where explosible powders (e.g., organic or metal dust) are handled, stored, processed, or produced¹. A dust explosion involves a rapid combustion of dust particles suspended in air with the generation of a pressure wave². This phenomena can be very energetic and damaging as the generated pressure can cause severe injuries and fatalities, and destroy buildings³. The injuries and fatalities usually occur in two ways. The initial damage is caused in proximity to the explosion, where people exposed to the explosion are burned by the intense heat of the dust cloud. The secondary damage caused to both people and property is by flying objects, falling structures, and debris, which can lead to many injuries and fatalities³.

1.1 Dust explosion incident statistics

The lack of fundamental understanding and improper hazard assessment of primary and secondary dust explosions are frequently the cause of serious incidents in the chemical process industry. Very few statistics of dust explosion incidents are available in the open literature. However, from those that can be found, the severity and catastrophic consequences of these explosions are clearly reflected.

As an example, in the United Kingdom, 485 explosions and 715 fires involving flammable dust were reported between 1965 and 1980. These incidents caused 26 fatalities and 639 injuries⁴. Between the period of 1979 - 1988, 303 dust explosions took

place in the United Kingdom as shown in Figure 1, out of which 92 incidents resulted in injuries⁵. More recently in 2011, information provided by HazardEx reported that nearly 2000 dust explosions occurred in Europe annually, out of which 50 were reported to be from the United Kingdom alone⁶.

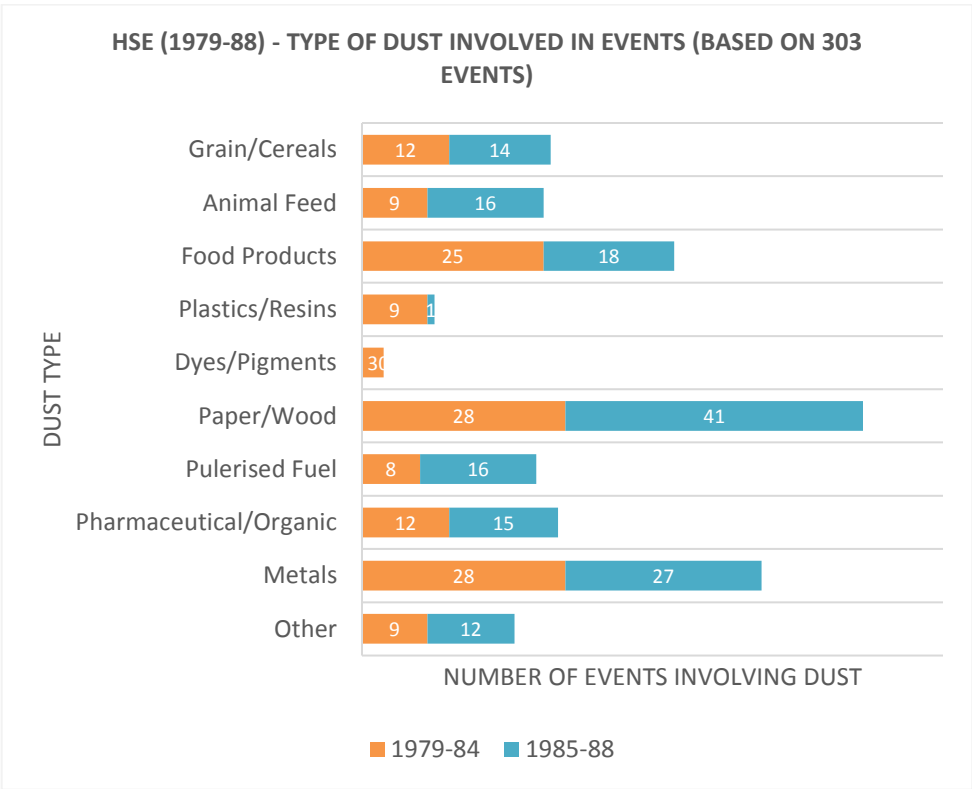


Figure 1. Distribution of dust explosions by material – United Kingdom (1979-1988) Reprinted from ⁵.

In West Germany from 1965 to 1980, 357 explosions were recorded, which resulted in 103 fatalities and 492 injuries as shown in Table 1. The National Fire

Protection Association (NFPA) published a very detailed report concerning dust explosion in the United States from 1900 to 1956, and counted the number of explosions to be totaling 1,123. These explosions resulted in 676 fatalities, 1,770 injuries, and 100 million dollars in material losses as shown in Table 2⁷.

Table 1. Dust explosions in West Germany, 1965-1980: Fatalities and injuries in a sample of 357 explosions
Reprinted from ⁷.

TYPES OF DUST	EXPLOSIONS		FATALITIES			INJURIES		
	No.	(%)	No.	(%)	Per explosion	No.	(%)	Per explosion
Wood and bark	113	31.6	12	11.7	0.11	124	25	1.10
Food and feed	88	24.7	38	36.8	0.43	127	26	1.44
Metals	47	13.2	18	17.5	0.38	91	18.5	1.94
Plastics	46	12.9	18	17.5	0.39	98	20	2.13
Coal and peat	33	9.2	7	6.8	0.21	39	8	1.18
Paper	7	2.0	0	0.0	0.0	0	0	0.0
Others	23	6.4	10	9.7	0.43	13	2.5	0.56
All	357	100.0	103	100.0		492	100.0	

Table 2. Dust explosions in the United States, 1900-1956: Fatalities, injuries, and material losses in a sample of 1123 explosions
Reprinted from ⁷.

TYPES OF DUST	EXPLOSIONS		FATALITIES			INJURIES			MATERIAL LOSSES	
	No.	(%)	No.	(%)	Per explosion	No.	(%)	Per explosion	Million \$	Per explosion
Wood and bark	162	14.5	38	5.6	0.23	160	9.0	0.99	11.4	0.070
Food and feed	577	51.4	409	60.5	0.71	1061	60.0	1.84	75.8	0.131
Metals	80	7.1	108	16.0	1.35	198	11.2	2.48	3.2	0.040
Plastics	61	5.4	44	6.5	0.72	121	6.8	1.98	3.7	0.061
Coal (not mines)	63	5.6	30	4.4	0.48	37	2.1	0.59	1.6	0.025
Paper	9	0.8	0	0.0	0.0	0	0.0	0.0	0.5	0.056
Others	171	15.2	47	7.0	0.27	193	10.9	1.13	4.3	0.025
All	1123	100.0	676	100.0		1170	100.0		100.5	

According to the CSB report in 2006, 281 dust fires and explosions occurred in the United States between 1980 and 2005, causing 119 deaths and 718 injuries as shown in Figure 3³. An interesting point to note from Table 1, Table 2, and Figure 2 is that food and wood dust explosion are the most common type of incidents across all countries and that plastics also constitute a good percentage of the dust explosion incidents. This is of particular interest to this research as one of the dusts this research is going to focus on is polyethylene dust, which is also responsible for dust explosions in the plastics industry.

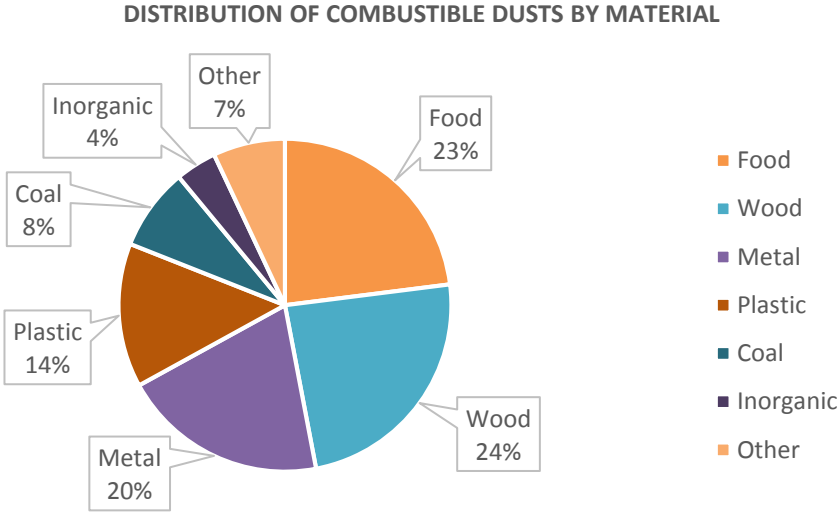


Figure 2. Dust explosions in the United States, 1980-2005
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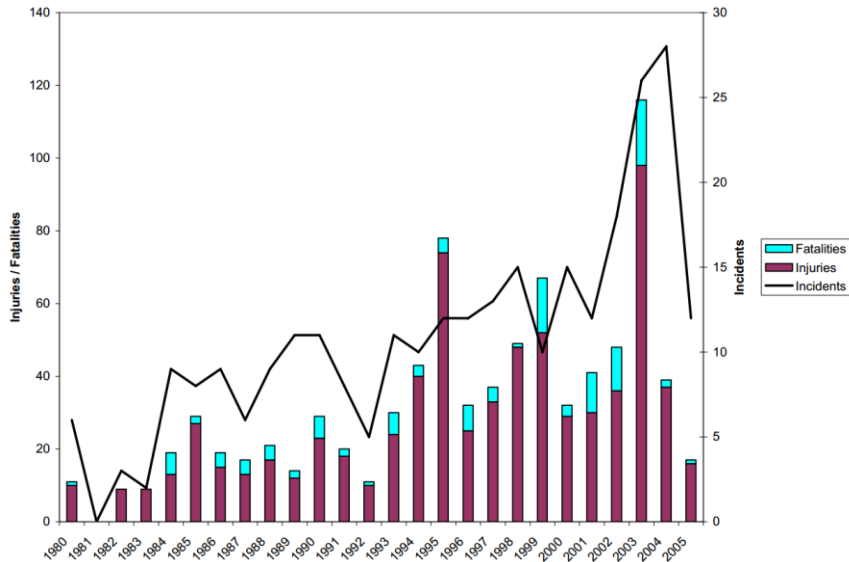


Figure 3. Fatalities from dust explosions in United States, 1980-2005
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1.2 Polyethylene & sulfur production and dust explosion issues

In the State of Qatar, the process industry faces many challenges to control the risks of explosion of mainly two types of explosible dust material in their facilities: polyethylene dusts from polyolefins production processes and sulfur dusts from desulfurization units in gas processing plants.

1.2.1 Polyethylene production and dust explosion issues

The petrochemical industry in Qatar is growing vastly to accommodate the Qatar National Vision for 2030. Three major companies manufacture polyethylene in Qatar (Qatar Petrochemical Company (QAPCO), Qatar Chemical Company (Q-Chem), and Qatofin). Table 3 shows a summary of the polyethylene production capacities of these

companies. The polyethylene pellets production process involves the generation of polyethylene dusts in various stages of the process units. Fine polyethylene dust dispersed in air in sufficient concentration and in the presence of an ignition source, may pose a potential dust explosion hazard.

Table 3. Polyethylene production capacity in Qatar

COMPANY	PRODUCTION CAPACITY
Qatar Petrochemical Company (QAPCO) ⁸	700 KTA of Low Density Polyethylene (LDPE): <ul style="list-style-type: none"> • 2 LDPE plants with a global capacity of 400 KTA • New LDPE 3 plant 300 KTA
Qatofin (Joint venture between QAPCO, Total, and Qatar Petroleum) ⁹	World class petrochemical plant capable of producing 450 KTA of Linear Low Density Polyethylene (LLDPE) in development
Qatar Chemical Company (Q-Chem) ¹⁰	453 KTA high-density and medium-density polyethylene (HDPE & MDPE) plant: <ul style="list-style-type: none"> • New Q-Chem II HDPE plant project to expand Q-Chem

1.2.2 Sulfur production and dust explosion issues

The world production of sulfur in 2011 amounted to 69 million tons, with 15 countries contributing more than 1 million tons each¹¹. It is also projected that the sulfur production in the Middle East will grow by 12.6 million tons from 2009 to 2019¹². Similarly, the sulfur production in Qatar has expanded significantly from 400,000 tons in 2005 to 3.5 million tons in 2015. This high output is driven by the petroleum and natural gas industry in Qatar which produces more than 1 million barrel of crude and gas condensate per day¹³. The main sulfur producing companies are QAPCO, Q-Chem, and

Qatargas. As with polyethylene dust, the production of elemental sulfur produced at various locations of the plant can be involved in a dust explosion.

1.3 Motivation for the project

The prevention of polyethylene and sulfur dust explosions and the mitigation of its consequences require a deep understanding of the explosive properties of such dusts. This knowledge can be used to reduce the frequency of dust explosions and to minimize the severity of the explosion, and therefore control the risks associated with polyethylene and sulfur dust explosions. This thesis answers to a request from the Qatar industry through the industry consortium of the Mary Kay O'Connor Process Safety Center (MKOPSC) to improve the current knowledge of the explosion properties of polyethylene and sulfur dust. The aim of this research is to perform an experimental and theoretical study on polyethylene and sulfur dust explosions in the context of the Qatar industry. The research aimed to fulfill three major objectives.

- The first objective was to conduct the physical characterizations for polyethylene and sulfur dusts found in the local industries of Qatar.
- The second objective focuses on the determination of the minimum explosion concentration (MEC) for polyethylene and sulfur dust in the particular context of the Qatar industry.
- The third objective of the research was to find the effect of particle size distribution of polyethylene and sulfur dust on the MEC.

2 FUNDAMENTALS OF DUST EXPLOSIONS

This section introduces the phenomena involved in a dust explosion along with the dust explosion characteristics to be determined for the control of the risks associated with dust explosion in a process plant.

2.1 Definition of a dust

There is currently no agreed standard definition of dust worldwide. The difference in classification is in regards to the particle size at which the dust exhibits explosibility and flame propagation properties once the required conditions for a dust explosion are met. The definition of a explosible dust, according to the National Fire Protection Association (NFPA 654, 2000), corresponds to “any finely divided solid material which is 420 microns or smaller in diameter (material passing a US No. 40 Standard Sieve) and that it presents a fire or explosion hazard when dispersed and ignited in air”¹⁴. The British Standards Institute in London in 1958 defined materials with particle size less than 1000 microns as powders, and particles with diameter size less than 76 microns as dust¹. The former US Bureau of Mines developed the following classification for dusts based on particle size¹⁴:

- i. Dust related to coalmines - particles smaller than 850 microns.
- ii. Dust related to surface industries^a - particles smaller than 425 microns.

^a Surface industries – Industries which are above ground level

2.2 Dust explosion pentagon and dust explosion mechanism

In order for a dust explosion to occur, five conditions must be met as described in the Dust Explosion Pentagon shown in Figure 4.

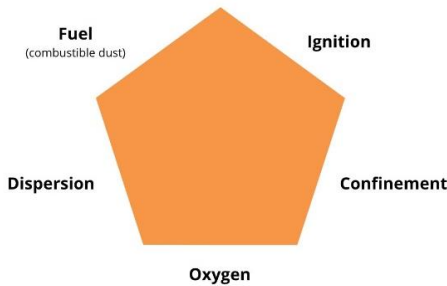


Figure 4. Dust explosion pentagon

- i. **Explosible Dust:** The dust must be explosible and present in a finely divided form in the right concentrations.
- ii. **Oxidant:** An oxidant must be present to enable combustion (e.g., oxygen in the air).
- iii. **Ignition:** An ignition source of sufficient energy must be present. Ignition of a dust cloud can occur in two ways.
- iv. **Dispersion:** The dust particles must be suspended in air and mixed with the oxidant for a dust explosion to occur¹⁵. Suspended dust burns more rapidly, which is what gives rise to an explosion rather than just a fire. The mixing of the

dust with the oxidant also illustrates the key difference between dust explosion and gas explosion, where the dust is a solid fuel rather than a gaseous fuel. In a gas explosion, there is a thorough mixing of both fuel and oxidant. This is because the smallest entities of fuel in air are separated only by molecular distances, and thus, the gravitational effects in such an explosion are negligible. On the other hand, in a dust/air mixture, the dust particles, due to their larger weight, are strongly influenced by gravity. This has a direct effect on the formation of a dust/oxidant suspension which is a key condition for any dust explosion^{16,17}.

- v. **Confinement:** The confinement of the dust cloud is a prerequisite to have a rapid increase in pressure for the explosion to take place¹⁸. The confinement of the dust cloud allows an overpressure to develop, which in turn enables a fast-burning dust flame, which then transitions into a dust explosion¹⁶. This confinement usually occurs in closed spaces, such as, in equipment or buildings, but it could also be caused by self-confinement if the reaction is fast enough. It is also entirely *“possible to have a destructive explosion even in open air if the reaction is so fast that the pressure builds up in the dust cloud faster than it can be released at the edge of the cloud”*¹⁹.

The dust explosion mechanism can be explained by the sequence of events as follows. First, the dust is dispersed in an oxidant rich environment (e.g., air). This dispersed dust finds an ignition source which transfers energy to the particles (through

conduction and radiation). The heat transferred vaporizes the material, forming a layer of combustible vapor at the surface of the dust particle that enters a combustion reaction with the oxidant in the air. The initiated flame propagates to other dust particles and leads to a rapid combustion throughout the dust cloud following the same mechanism. Due to the confined space, this flame propagation keeps on accelerating which gives a rise to a pressure wave. A pictorial representation of this process is shown in Figure 6²⁰. The flame propagation rate of the explosion is dependent on many factors related to the nature of the dust, dust particle size, and the nature of combustion of the by-products formed¹.

The dust explosion pentagon can also be utilized in mapping out the dust explosion process in time. This is obtained by unwrapping the pentagon in the clockwise direction as shown in Figure 5. This figure shows the different process mechanisms, which are arranged based on their time-related position during the evolution of the explosion. Additional mechanisms which affect explosion severity, such as, flame propagation and acceleration have also been added to the flowchart¹⁸.

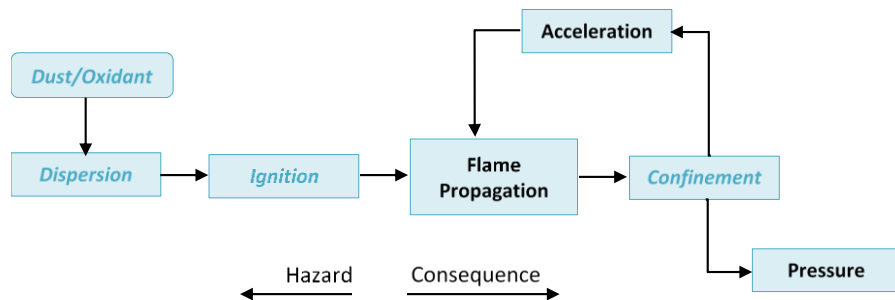


Figure 5. Mapping the dust explosion process in sequence Reprinted from¹⁸.

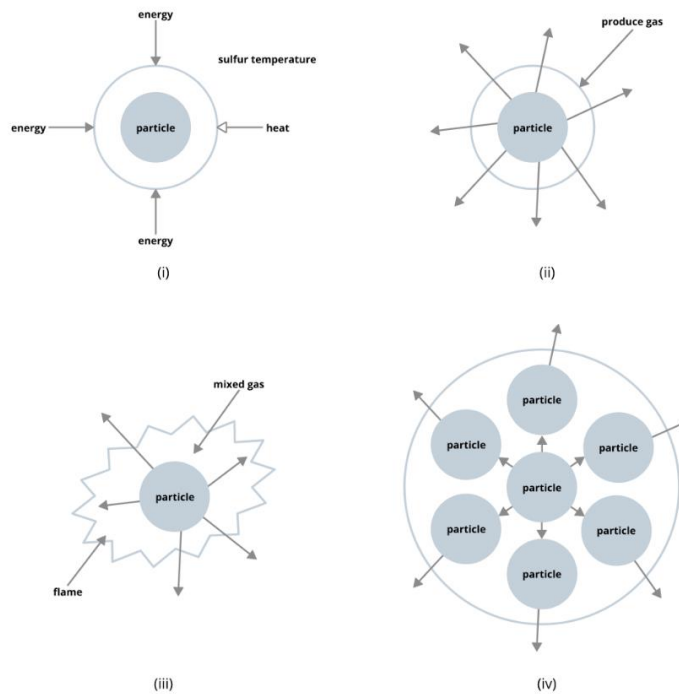


Figure 6. Explosion mechanism of dust particles
Reprinted from ²⁰.

It is also essential to understand that dust explosions and gas explosions significantly differ from each other. It is very likely that in similar conditions, it would be quite easy to produce a gas cloud, but not so easy to produce a dust cloud. One of the reasons is that the propagation of flames in dust/air mixtures is not just limited to the flammable dust concentration range of the clouds, which is the opposite of what happens in gas flame propagation. This is because the deposits of dust actually offer a discrete and different possibility of flame propagation. Other differences are captured in the fact that initial forces in the dust cloud can produce fuel concentration gradients, and that

thermal radiation could be an important contributing factor in the heat transfer of the flame to the unburnt cloud¹.

There is still much more research which needs to be conducted to understand flame propagation in dust clouds. Some of the key factors to consider are flame distortion and the turbulence propagated by the explosion itself. Some of the differences between premixed gases and dust clouds were captured by Rzal and Vessiere in their experiments of a laminar maize starch/air flame using different obstacles, such as, a disk, an annulus, and a sphere. While observing the flame propagation with an annulus, it was observed that there was a flame-quenching phenomenon present, which could be explained by centrifugal separation of dust particles and air. As such, this finding was very significant in proving that burning rate of dust clouds does not respond to turbulence in the same way as the burning rate of a premixed gas does²¹.

2.3 Primary and secondary explosions

A dust explosion can either be classified as a primary explosion or a secondary one. A primary dust explosion usually occurs in closed and confined equipment or room where a dust disperses, ignites, and explodes within the equipment³. One of the key differences between a dust explosion and gas explosion is that gas explosions very rarely occur inside vessels due to a lack of air to support the explosion. However, primary dust explosions generally occur in the process equipment as the condition of confinement is met inside such vessels. Thus, a dust explosion which is severe enough can cause the

vessel to rupture if it has insufficient pressure release devices, or if its design pressure is too low to withstand the overpressure generated by the dust explosion¹.

A secondary explosion occurs when dust accumulated on floors or ceilings or other surfaces is disturbed, lofted, and ignited by a primary explosion. Generally, the melted dust particles in the primary explosion provides the sparks needed for secondary explosions. These secondary explosions accelerate the combustion of the dust as the pressure increases²². The pressure wave from the secondary explosions then cause the accumulated dust in other areas to become suspended in air, which in turn leads to multiple secondary dust explosions. Thus, a weak primary explosion can cause very powerful secondary dust explosions, based on the amount of dust prevalent around in the factory and equipment. Another point to note is that the initiating event for a secondary dust explosion might not be a dust explosion at all. The incidents outlined in the CSB report, such as, the CTA Acoustics, West Pharmaceutical Services, and Ford River Rouge, are all examples of secondary dust explosions initiated by events that were not dust explosions³.

2.4 Prevention and protection strategies

The control and prevention of dust explosion hazards make use of two elements. These are the dust explosion pentagon, and the Basis of Safety approach. The explosion pentagon shown in Figure 4, offer guidance in identifying the factors that cause dust explosions. For example, the explosion pentagon gives several approaches of explosion prevention to the industry. Some examples of these approaches are, “removal of fuel by

good housekeeping and removal of electrostatic ignition sources by grounding and bonding”¹⁶. Similarly, the dust explosion pentagon helps in visualizing the explosion requirements, which leads to identification of measures for explosion mitigation, such as, isolation venting or isolation containment¹⁶.

A Basis of Safety approach is a safeguarding philosophy, which involves specific devices, equipment, and procedures, whose purpose is to greatly weaken or eliminate one of the pentagon sides to such an extent that a dust explosion cannot occur. The approach also takes into account that if the explosion does occur, then employees and facilities are protected from dangerous and adverse effects of the explosion. Generally, the three methods used to prevent dust explosions are either eliminating the ignition sources, or controlling the oxidant required for the explosion to occur, or controlling the fuel required for the dust explosion¹⁵.

Once the prevention strategies fail in stopping the dust explosion, the protection strategies are used as a fallback measure, so that the explosion does not cause substantial damage to life and property. Some of the most common forms of protection strategies are explosion containment, explosion suppression, explosion isolation, and explosion venting. Eckhoff in Table 4 presents some of the prevention and mitigation techniques, which are used, in combating the possibility of a dust explosion. It clearly separates prevention and mitigation as two separate measures to combat a dust explosion hazard⁷.

Table 4. Means of preventing and mitigating dust explosions
Reprinted from ⁷.

PREVENTION		MITIGATION
Preventing ignition sources	Preventing explosible dust clouds	
a. Smoldering combustion in dust, dust flames	f. Inerting by N ₂ , CO ₂ , and rare gases	j. Partial inerting by inert gas
b. Other types of open flames (e.g., hot work)	g. Intrinsic inerting	k. Isolation (sectioning)
c. Hot surfaces	h. Inerting by adding inert dust	l. Venting
d. Electric sparks and arcs, electrostatic discharges	i. Dust concentration outside explosible range	m. Pressure-resistant construction
e. Heat from mechanical impact (metal sparks & hot spots)		n. Automatic suppression
		o. Good housekeeping (dust removal, cleaning)

2.5 Dust explosibility characteristics

The first step in the determination of dust explosion hazards is the simple qualitative assessment of the potential for a dust material to explode. Once this potential is established, a more in-depth analysis of the dust explosibility properties is performed to quantitatively evaluate factors which affect the frequency and the severity of the consequences.

2.5.1 *Qualitative assessment of dust explosibility in a modified Hartmann tube*

A modified Hartmann tube is used to determine whether the dust is explosible or not, and provide a qualitative indication of the intensity of the explosion. The equipment is a 1.2-liter vertical tube mounted onto a dust dispersion system in which the dust is dispersed using an air blast and ignited using an induction ignition system or a coil ignition system. The 1.2-liter tube is made of transparent glass material which allows for

the visual capture of whether the dust ignited or had an explosion. Figure 7 provides a graphical representation of the workings of the modified Hartmann tube. If there is an explosion, then the lid at the top of the tube flips, signifying the severity of the explosion.

The general dust explosibility classification, which has been used for years, is divided broadly in two categories²²:

- Category A – Dust which ignite and propagate a flame
- Category B – Dust which do not propagate a flame in the test apparatus

These classifications replaced an earlier scheme where dusts were divided into Class I, II, or III. Broadly speaking, Class I corresponded to Group A of dusts, while Class II and III corresponded to Group B²³. In the United States, explosible dusts are classified as hazardous materials under the NFPA classification. Class I is given to gases, whereas Class II has been assigned to dusts. The dust classification has been further subdivided into E for metal dusts, F for carbonaceous dusts, and G for flour dusts²³.

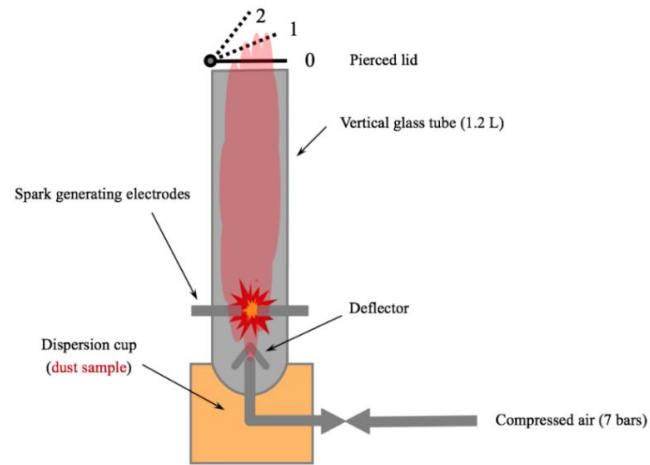


Figure 7. Detailed modified Hartmann tube illustration

2.5.2 *Quantitative assessment of dust explosion characteristics*

Quantitative assessment of dust explosion characteristics refers to the quantification of the properties that are related to the five conditions of the dust explosion pentagon, in that they prove to be critical factors in either preventing a dust explosion or mitigating its consequences as shown in detail in Figure 8.

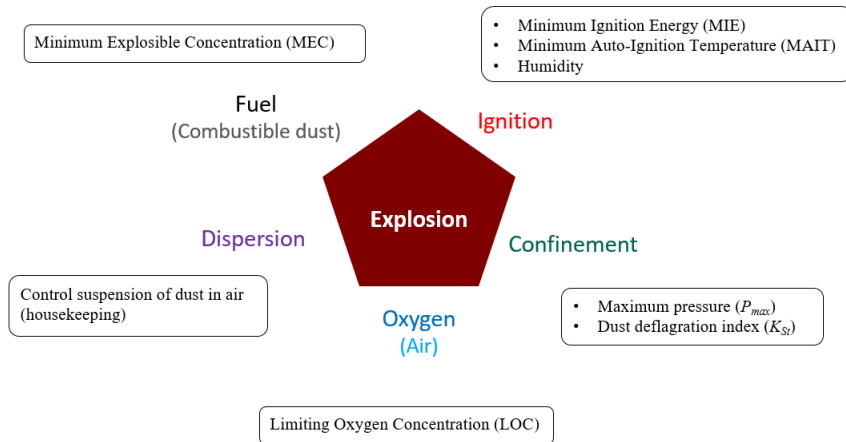


Figure 8. Dust explosion pentagon in relation to dust explosion properties

2.5.2.1 Minimum Explosible Concentration (MEC)

The MEC is “the minimum concentration of a combustible dust cloud that is capable of propagating a deflagration through a well dispersed mixture of the dust and air under the specified conditions of test”²⁴. If the concentration of the dust dispersed in air does not reach the MEC, then there is not enough fuel for the explosion to occur. It is essential that the concentration of suspended dust be within the parameters of the explosible range for an explosion to occur. This can be explained in a similar manner by giving the example of flammability ranges, such as, Lower Flammability Limit (LFL) and Upper Flammability Limit (UFL), which define the minimum and maximum ranges of concentration for commonly used vapors to ignite³. This is helpful in dust prevention mechanisms, especially in confined equipment where the risks of dust explosions are much higher²⁵.

2.5.2.2 Maximum Explosion Overpressure (P_{max})

The maximum explosion overpressure (P_{max}) is the maximum pressure that the dust explosion can develop in a given confined volume. This parameter serves as an indication of the potential destructive pressures that could be generated during a dust explosion of the material being tested, and it is usually used for venting, suppression, isolation, and containment designs²⁶.

2.5.2.3 Dust Deflagration Index (K_{St})

The maximum rate of pressure increase accompanying an explosion $(dP/dt)_m$ indicates the robustness and strength of an explosion. When testing for explosibility characteristics in vessels of different volumes, it was found that “a plot of the logarithm of the maximum pressure slope versus the logarithm of the vessel volume frequently produced a straight line of slope -1/3”. This relationship was named the cubic law²⁷, where K_{St} is the dust deflagration index (Table 5):

$$\left(\frac{dP}{dt}\right)_{max} V^{\frac{1}{3}} = K_{St} \quad \text{Equation 1}$$

Table 5. Dust explosion classification based on K_{St} value
Reprinted from ³.

DUST DEFLAGRATION INDEX (K_{St})	CLASSIFICATION
0	no explosion
$0 < K_{St} < 200$	weak explosion (dust explosion class 1)
$200 < K_{St} < 300$	strong explosion (dust explosion class 2)
$300 < K_{St}$	very strong explosion (dust explosion class 3)

2.5.2.4 Limiting Oxygen Concentration (LOC)

The LOC is defined as the average of the lowest oxygen concentration at which an explosion takes place and the highest oxygen concentration in a dust/air mixture in which an explosion just fails to take place. This property is used to inert vessels to prevent dust explosions.

2.5.2.5 Minimum Ignition Energy (MIE)

The MIE is the minimum amount of energy required to ignite a explosible dust cloud. This is used for the control of ignition sources in areas prone to dust explosions.

2.5.2.6 Minimum Ignition Temperature (MIT)

The MIT is specified as the lowest temperature at which the dust cloud is ignited. This is used for the control of ignition by contact with a hot surface in areas prone to dust explosions (e.g., hot surface on equipment).

2.5.3 Experimental assessment of dust explosion properties

The above explosion properties are generally measured using three equipment:

- 20-liter sphere for MEC, P_{max} , K_{St} , and LOC
- Mike-3 apparatus for MIE
- Godbert-Greenwald furnace for MIT

2.5.3.1 20-liter sphere (MEC, P_{max} , K_{St} , and LOC)

The 20-liter dust explosion sphere is used to measure the P_{max} , K_{St} , and LOC of a dust explosion (Figure 9). It is an enclosed chamber made of stainless steel that measures the overpressure and the rate of pressure rise for an explosion. This rate of pressure rise is directly tied to calculating the dust deflagration index value of the dust explosion. These data are measured by the pressure transducers present in the inner chamber of the sphere. This data is then collected and stored using a high-speed computer-based data acquisition system.

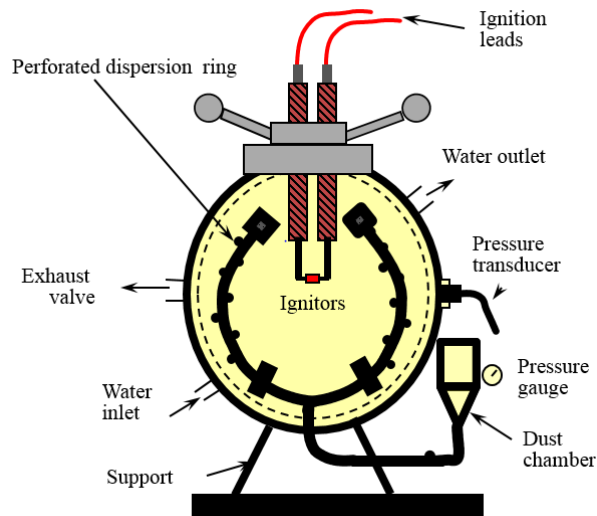


Figure 9. Illustration of 20-liter dust explosion sphere

2.5.3.1.1 Measuring Minimum Explosible Concentration (MEC)

The methodology to measure the MEC using a 20-liter sphere is provided by the ASTM E1515 standard. The standard recommends to start testing a dust sample from a dust concentration of 100 g/m^3 , and if a deflagration occurs, then the dust concentration is reduced till no deflagration occurs. If a deflagration does not occur at 100 g/m^3 , then the concentration is increased until a deflagration occurs. This process is repeated till the lowest concentration at which the dust explodes is found. This lowest concentration at which the dust explodes is the MEC value for that particle size value and range²⁴.

2.5.3.1.2 Measuring P_{\max} and K_{St}

Both the P_{\max} and maximum rate of pressure rise are also measured using pressure transducers in the 20-liter sphere. The ASTM E1226 standard gives the steps required to measure both the P_{\max} and K_{St} values. It is recommended to start testing a dust sample from a dust concentration of 250 g/m^3 . The pressure time curves give “*maximum pressure*” and the rate of pressure rise for that specific dust concentration. Tests are performed with increasing dust concentration by 250 g/m^3 to plot the variation of the “*maximum pressure*” and the *rate of pressure rise* as a function of dust concentration. P_{\max} and $(dP/dt)_m$ represent the maximum values of these curves. Once the maximum values for P_{\max} and $(dP/dt)_m$ have been found, two further tests are conducted at that same dust concentration and at least one test is conducted at each side of the optimal dust concentration to ensure repeatability of results. The $(dP/dt)_m$ and P_{\max} values are normally obtained in the 500 to 1250 g/m^3 range. In many cases, the correct

P_{\max} and $(dP/dt)_m$ values are not found at the same concentrations. The $(dP/dt)_m$ values are then used to calculate the K_{St} values for that dust sample²⁸.

2.5.3.1.3 Measuring Limiting Oxygen Concentration (LOC)

The concentration of oxygen inside the 20-liter sphere can be controlled to measure the LOC as outlined in the ASTM E2931 standards²⁹. It recommends to start the tests from a dust concentration of 500 g/m³. The oxygen concentration in the sphere and the dust concentration are varied to find the following:

- L: The lowest oxidant concentration at which flame propagation is possible for at least one dust concentration.
- H: The highest oxidant concentration at which flame propagation is not possible for the same dust concentration.

The LOC for that dust sample is then defined as:

$$LOC = \frac{L + H}{2} \quad \text{Equation 2}$$

2.5.3.2 Mike-3 apparatus (MIE)

The Mike-3 apparatus (Figure 10) is very similar to the modified Hartmann tube in that the dispersion and ignition occur in 1.2-liter transparent glass tube (see paragraph 2.5.1). The main difference is that the voltage provided to the electrodes can be varied, which is then used to vary the amount of energy being supplied by the electrodes for the ignition to occur³⁰. The MIE is given by the minimum energy of the electrode that was required to ignite a explosible dust cloud.



Figure 10. Mike-3 apparatus
Reprinted from ³¹.

2.5.3.3 Godbert-Greenwald furnace (MIT)

A Godbert-Greenwald furnace is generally used to measure the minimum ignition temperature of a dust ²³. It can also be used in explosibility testing of Class B dusts ²². The common components of a Godbert-Greenwald furnace are a dust chamber to store the dust, a pressure vessel, a heater to heat the oven, and a thermocouple to measure the temperature of the oven. Figure 11 provides a detailed diagram and schematic of a Godbert-Greenwald furnace³². The dust is injected into the vertical tube in the furnace which is held at a constant temperature. When the dust passes through the furnace, if a flame is observed at the end of the furnace, the dust is classified to be explosible. The temperature is varied to measure the lowest temperature at which the

dust would explode. A 20-liter sphere can also be modified by adding thermocouples to find out the MIT of an explosible dust²³.

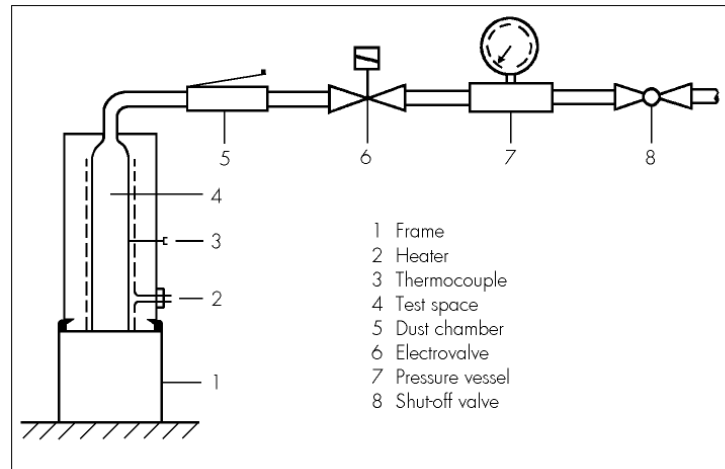


Figure 11. Godbert-Greenwald furnace schematic
Reprinted from ³².

3 LITERATURE REVIEW OF POLYETHYLENE AND SULFUR DUST PROPERTIES

This section summarizes the intensive literature search to gather published explosive properties of polyethylene and sulfur dust. The influence of the dust concentration, dust particle size, and humidity of the properties will be discussed.

3.1 Polyethylene dust explosion properties

3.1.1 *Minimum Explosible Concentration (MEC) of polyethylene dust*

Table 6 presents a summary of all MEC values for polyethylene we found in the literature. The MEC generally ranges from 10 to 500 g/m³ for particle average diameters ranging from approx. 27 to 200 μm. The gathered data have been obtained from different types of equipment and with samples of different particle size. Some of these values are for low density polyethylene dust (LDPE). No MEC data was found for high density polyethylene (HDPE) dust. In many cases, the type of polyethylene used for the experiment is simply not mentioned.

Hertzberg et al. (1982) performed experiments using an 8-liter sphere with polyethylene dust particle size from approx. 27 to 200 μm and found MEC values ranging from approx. 50 to 475 g/m³ (Figure 12). They did not observe a significant variation of the MEC (around 60 g/m³) for dust particle sizes of 15 to 90 μm. Above this range, the MEC values increased until a dust particle size of 200 μm, beyond which the dust sample did not explode regardless of the dust concentration. The authors conclude

that for polyethylene (as for coal dusts), there is a *lower* characteristic diameter below which the explosive behavior of dust particle is independent of the particle size, and the combustion reaction of these dust particles exhibit explosible qualities of an equivalent premixed gas. They also stated that as the dust particle is increased above a *higher* characteristic diameter the dust would not explode regardless of the dust concentration³³.

Table 6. Minimum Explosible Concentration (MEC) of polyethylene dust

MEC	PARTICLE SIZE	POLYETHYLENE TYPE	EQUIPMENT	SOURCE
≈50 g/m ³	≈27 μm (***)	NA	8-liter chamber	33
≈50 g/m ³	≈31 μm (***)	NA	8-liter chamber	33
≈65 g/m ³	≈55 μm (***)	NA	8-liter chamber	33
≈60 g/m ³	≈90 μm (***)	NA	8-liter chamber	33
≈225 g/m ³	≈130 μm (***)	NA	8-liter chamber	33
≈475 g/m ³	≈200 μm (***)	NA	8-liter chamber	33
35 g/m ³	37 μm (**)	NA	20-liter sphere	34
45 g/m ³	106 - 125 μm ^b	LDPE	Modified Hartmann Tube	35
50 g/m ³	125 - 150 μm	LDPE	Modified Hartmann Tube	35
60 g/m ³	150 - 180 μm	LDPE	Modified Hartmann Tube	35
15 g/m ³	62 μm (median)	LDPE	20-liter sphere	36
60 g/m ³	136 μm (d50) (**)	NA	Modified Hartmann Tube	37
10 g/m ³	49 μm (**)	NA	20-liter sphere	38
20 g/m ³	28 μm (**)	NA	20-liter sphere	38
40 g/m ³	103 μm (**)	NA	20-liter sphere	38
500 g/m ³	171 μm (**)	NA	20-liter sphere	38

(**) volume median diameter

(***) surface area weighted mean diameter

^b The authors chose to represent the particle size interval by its upper limit (for example, 106-125 μm is represented by 125 μm).

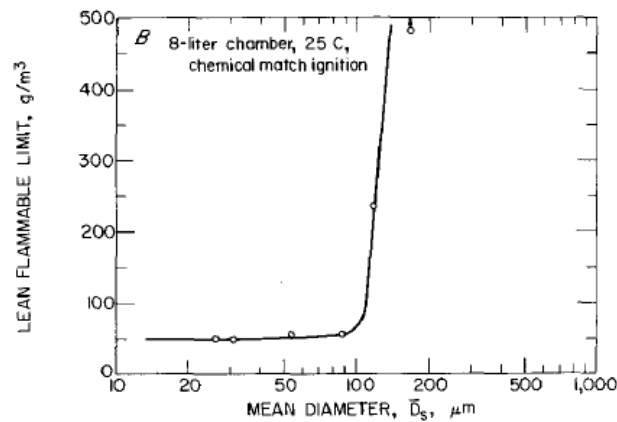


Figure 12. Lean flammability limit for polyethylene powder as a function of particle size
Reprinted from ³³.

Mittal and Guha (1996) studied the relationship between polyethylene dust particle size and MEC using a modified Hartmann tube. This is the only paper which uses a modified Hartmann tube to measure the effect of dust particle size on MEC for polyethylene. It is important to note that the ASTM E1515 standard recommends to either use a 20-liter or 1 m³ vessel to conduct experiments regarding MEC. The authors indicated that the purpose of the experimental work was to only obtain a preliminary range of information on the MEC. For particle size of 125 μm , 150 μm , and 180 μm (note that this is the highest particle size for a given sample, not the surface area mean diameter as used by Hertzberg), they found MEC values of 45 g/m³, 50 g/m³, and 60 g/m³ ³⁵ (These MEC values differ very significantly from Hertzberg's results (by a factor 5 for 125 μm up to a factor 10 for 150 μm). While this difference may be

related to the use of different equipment, Mittal et al. (1996) did not characterize the dust particle size in a way that allows comparison with values obtained by Hertzberg et al. (1982).

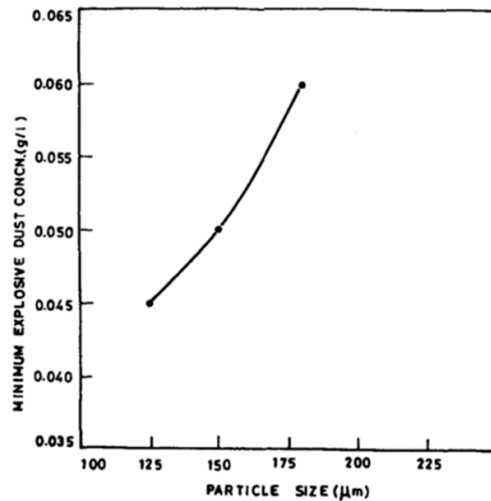


Figure 13. Variation of MEC with particle size for polyethylene dust. Reprinted from ³⁵.

Amyotte et al. (2012) conducted experiments with polyethylene dust particle size in a 20-liter sphere ranging from 28 to 916 μm (volume median diameters). The authors observed that the dust particle sizes above 171 μm did not explode even for dust concentrations of 2750 g/m^3 . The authors concluded that dust particle may be so large that, even at high dust concentrations, the dust sample will not explode as the particle size is too large to be dispersed in air. It was shown that MEC does increase significantly with increasing particle size, but this behavior is not seen for dust particle size at around

under $100\ \mu\text{m}$ ³⁸. While Amyotte and Hertzberg use different definitions for particle size (*volume median diameters* and *surface area mean diameter*, respectively) and the equipment used is different (20-liter and 8-liter spheres, respectively), the MEC values seem comparable on the range of particle size.

Hertzberg et al. (1982) and Amyotte et al. (2012) come to the same conclusion in regards to effect of particle size on MEC of polyethylene dust. Their results show that MEC does increase with particle size, but this behavior can only be seen above a critical diameter of about $100\ \mu\text{m}$. Both the papers then show that the MEC of the polyethylene dust sample would keep increasing until a second critical diameter is reached, beyond which the dust would not explode. Amyotte et al. (2012) found this critical diameter to be about $171\ \mu\text{m}$ (volume median diameter), whereas Hertzberg et al. (1982) found this critical diameter to be approx. $200\ \mu\text{m}$ (surface area weighted mean diameter). The results of these two papers are not comparable to the values obtained by Mittal et al. (1996), since the authors do not test for MEC below $125\ \mu\text{m}$. Due to the difference in particle size characterizations, the exact values are not comparable and thus conclusions and comparisons between these two papers are limited in scope.

3.1.2 Maximum pressure (P_m) and rate of pressure rise (dP/dt)_m of polyethylene dust

Several authors studied the phenomena of dust explosion when performed in a confined volume (e.g., 20-liter sphere) to observe the effect of the dust concentration and humidity on the following parameters:

- Maximum pressure (P_m): this corresponds to the maximum pressure generated in the confined volume as a result of the explosion.
- Maximum rate of pressure rise $(dP/dt)_m$: this is a function of the severity of the explosion.

3.1.2.1 Effect of dust concentration on P_m and $(dP/dt)_m$

Lunn et al. (1986) carried out experiments to measure the P_m of polyethylene (unknown type) dust using a 20-liter sphere. The experiments were conducted using a starting concentration of a specific dust sample from 250 g/m^3 and the dust concentration was gradually increased up to 2000 g/m^3 . The author observed that P_m and $(dP/dt)_m$ would increase with the dust concentration for concentration values below 500 g/m^3 . For concentration values below 500 g/m^3 , P_m and $(dP/dt)_m$ would decrease with the dust concentration³⁹.

Table 7. 20-liter sphere experiment with polyethylene P_m and $(dP/dt)_m$
Reprinted from³⁹.

Concentration (g/m^3)	P_m (bar)	$(dP/dt)_m$ (bar/s)
250	6.75	410
500	7.21	508
750	6.21	434
1000	5.76	441
2000	4.38	266

This conclusion was confirmed by Kaufmann et al. (1992) in their experiments with polyethylene dust using a 0.95 m³ sphere (Figure 14)^c. However, the absolute values are different to the values obtained by Lunn et al. (1986) using a 20-liter sphere. It is difficult to explain these differences, due to the equipment being different and also because Kauffman et al. (1992) did not mention the particle size of the sample tested.

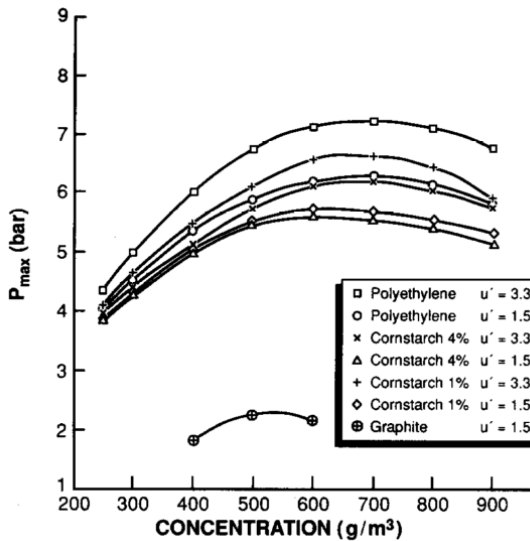


Figure 14. Effect of turbulence on P_m for different dusts
Reprinted from ⁴⁰.

Lunn et al. (1986) and Kauffman et al. (1992) did not conduct tests with dust concentration below 250 g/m³. This was done by Cashdollar (2000) for dust concentrations at approximately 25 g/m³ to 4000 g/m³ in a 20-liter sphere (Figure 15). He

^c The authors refer to P_m using the annotation P_{max} . Note that this P_{max} differs from the definition of P_{max} as used by the ASTM and used in this thesis.

observed the same explosion behavior. The results showed that P_m increases sharply for concentration between 25 g/m^3 and 200 g/m^3 . Cashdollar also showed that polyethylene dust even explodes at concentration as high as 4000 g/m^3 (Figure 15). The author explains that at such high concentrations, there is a high amount of uncertainty in how well and effectively the dust is being dispersed. Cashdollar also theorizes that the decrease in the P_m and $(dP/dt)_m$ at such higher concentrations might be the result of both the probable decrease in turbulence and an increased heat sink because of the very large dust concentrations¹⁹.

The conclusion in regards to the effect of dust concentration on P_m for the three authors above observed that the P_m and $(dP/dt)_m$ rise with increasing dust concentration up to a certain dust concentration (500 g/m^3 for Lunn et al., $\approx 750 \text{ g/m}^3$ for Kaufmann et al., and $\approx 500 \text{ g/m}^3$ for Cashdollar), and above this dust concentration, both the P_m and $(dP/dt)_m$ values decrease when the dust concentration increases. The values obtained by Cashdollar are different compared to both Lunn et al. and Hertzberg et al., and again, all these results are not comparable due to lack of information by both Kauffman et al. (1992) and Cashdollar (2000) regarding the dust particle size.

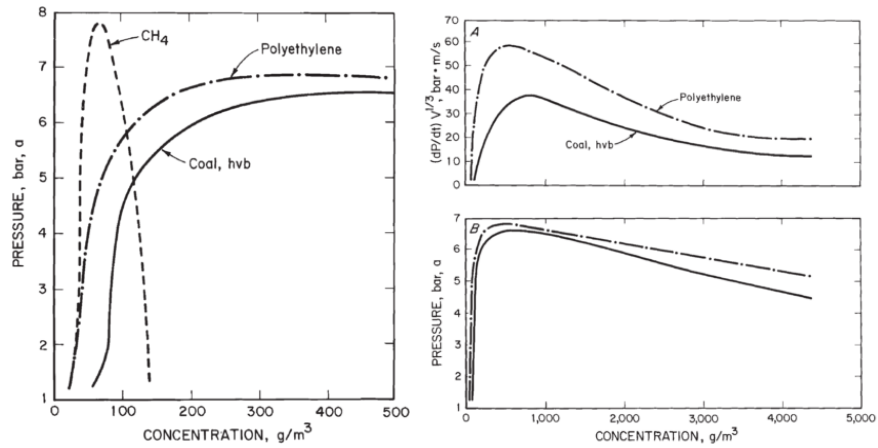


Figure 15. P_m data for polyethylene dusts compared with those of methane gas Reprinted from ¹⁹.

3.1.2.2 Effect of water content on P_m and $(dP/dt)_m$

Traore et al. (2009) measured P_m and $(dP/dt)_m$ of polyethylene dust in a 20-liter sphere while varying the amount of water which was injected into the 20-liter sphere. As shown in Figure 16, the conclusion of the experiments was that both maximum pressure (P_m) and rate of pressure rise ($[dP/dt]_m$) decrease when the humidity increases³⁷.

Eckhoff (2003) provides an explanation of the effect of moisture content on the dust explosible properties. One is that, it reduces the ignition sensitivity of the dust, meaning that it will be harder to ignite the dust. Secondly, it also reduces the explosion violence of the explosion as the water acts as an inhibitor in the oxidation reaction and the water can act as a heat sink⁷.

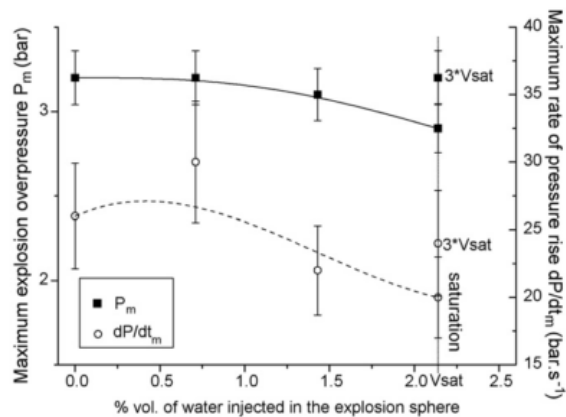


Figure 16. Effect of atmosphere humidity on P_m and $(dP/dt)_m$ for polyethylene dust Reprinted from ³⁷.

3.1.3 Maximum Explosion Overpressure (P_{max}) and Dust Deflagration Index (K_{St}) of polyethylene dust

The maximum value of $P_m = f$ (dust concentration) curve correspond to P_{max} , and equally the maximum value of $(dP/dt)_m = f$ (dust concentration) curve is used to calculate the K_{St} of a dust material (refer to 2.5.3.1).

Table 8 gather all the P_{max} and K_{St} of polyethylene dust found in the literature. The table shows that, for polyethylene, P_{max} ranged between 5.9 and 8.8 bar.

A very wide range of K_{St} values was found for polyethylene (from 5 to 200 bar-m/s. This puts polyethylene in the ST1 dust explosion classification (weak explosion).

Note: as stated earlier, even a weak primary explosion can give rise to much stronger secondary explosion as in the case of the accident at West Pharmaceuticals.

Table 8. P_{max} and K_{St} of polyethylene dust

P_{max}	K_{St}	PARTICLE SIZE	POLYETHYLENE TYPE	EQUIPMENT	SOURCE
≈(0 - 8.5 bar)	≈(0 - 200 bar-m/s)	≈[0 - 350 μm]	NA	1 m ³ vessel	⁴¹
7.21 bar	138 bar-m/s	<10 μm – weight % - 1.7 >10<20 μm – weight%-70.7 >20<36 μm – weight%-70.7	NA	20-liter sphere	³⁹
8.8 bar	193 bar-m/s	NA	NA	20-liter sphere	⁴²
NA	≈20 bar-m/s	116 μm ^d	NA	26-liter sphere	⁴³
NA	≈45 bar-m/s	98 μm	NA	26-liter sphere	⁴³
NA	≈80 bar-m/s	≈70 μm	NA	26-liter sphere	⁴³
NA	≈80 bar-m/s	≈60 μm	NA	26-liter sphere	⁴³
NA	≈80 bar-m/s	56 μm	NA	26-liter sphere	⁴³
NA	≈80 bar-m/s	≈45 μm	NA	26-liter sphere	⁴³
NA	≈120 bar-m/s	20 μm	NA	26-liter sphere	⁴³
NA	≈150 bar-m/s	≈10 μm	NA	26-liter sphere	⁴³
≈7 bar	NA	NA	NA	1.22 m diameter sphere	⁴⁰
6.6 bar	71 bar-m/s	< 500 μm d ₉₀ <250 μm	HDPE	20-liter sphere	⁴⁴
≈6.9 bar	≈55 bar-m/s	NA	NA	20-liter sphere	¹⁹

^d The authors chose to represent the particle size as the average of the sieve sizes between which each dust sample was held.

Table 8. continued.

P_{max}	K_{St}	PARTICLE SIZE	POLYETHYLENE TYPE	EQUIPMENT	SOURCE
8.5 bar	131 bar-m/s	62 μm (median)	LDPE	20-liter sphere	³⁶
NA	$\approx 5 - 130$ bar-m/s	$\approx 5 - 900$ μm	NA	20-liter sphere	⁴⁵
6.9 bar	137 bar-m/s	28 μm (**)	NA	20-liter sphere	³⁸
6.7 bar	104 bar-m/s	49 μm (**)	NA	20-liter sphere	³⁸
6.9 bar	78 bar-m/s	103 μm (**)	NA	20-liter sphere	³⁸
5.8 bar	15 bar-m/s	171 μm (**)	NA	20-liter sphere	³⁸

(**) volume median diameter

Bartknecht (1981) measured values of P_{max} and K_{St} for polyethylene in a 1 m³ sphere for samples of particle size approximately between 25 and 350 μm . He observed that both P_{max} and K_{St} decrease as the particle size increases⁴¹ (Figure 17).

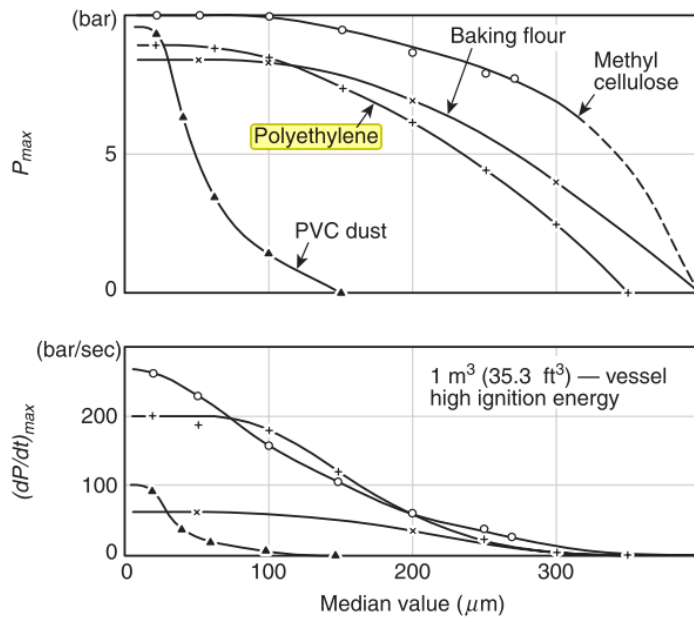


Figure 17. Effect of dust particle size on P_{max} for polyethylene dust
Reprinted from ⁴¹.

Britton et al. (1989) conducted tests to measure the effect of particle size (from 20 to 116 μm – see below for the meaning of this diameter) on the K_{St} of polyethylene dust using a 26-liter sphere (no values of P_{max} are reported). They observed that the higher the particle size, the lower the K_{St} (Figure 18 and Figure 19). The maximum values of K_{St} for polyethylene sample tested remained under 200 bar-m/s ⁴³.

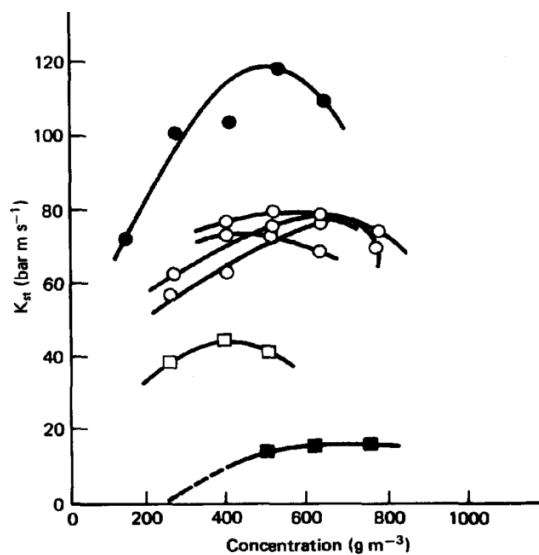


Figure 18. Effect of particle size on K_{St} of a polyethylene dust sample: ●, average diameter 20 μm ^e; ○, average diameter 56 μm ; □, average diameter 98 μm ; ■, average diameter 116 μm .
Reprinted from ⁴³.

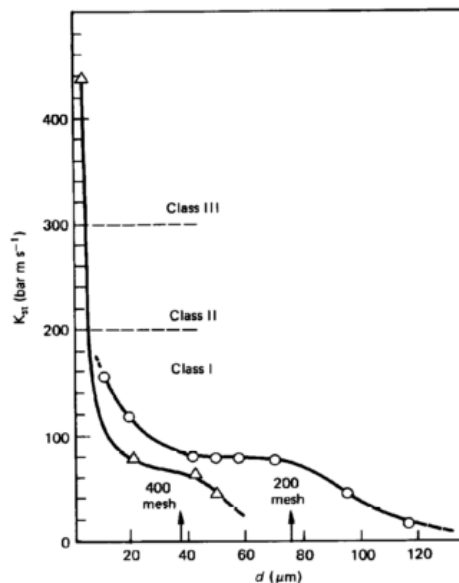


Figure 19. Effect of particle size on K_{St} of ○ polyethylene and Δ silicon
Reprinted from ⁴³.

^e The authors chose to represent the particle size as the average of the sieve sizes between which each dust sample was held.

In a research report from 2003, HSE (UK) gives a P_{\max} value of 8.5 bar and K_{St} of 131 bar-m/s for a particle size of 62 μm (median), and this value is comparable to the results by Bartknecht³⁶. Both Bartknecht (1981) and HSE only characterize the dust particle size using median value without mentioning if it is related to volume, mass or surface area of the dust particle.

Amyotte et al. (2012) measure P_{\max} and K_{St} for polyethylene dust particle size from 28 to 916 μm (volume median diameters) (Table 8) according to the ASTM E1226 standard. They did not have any explosion for dust particle sizes above 276 μm , therefore no P_{\max} and no K_{St} . They observed that K_{St} value increased from 15 to 137 bar-m/s as the dust particle size became smaller for the samples that exploded. For particle diameter between 28 to 103 μm , P_{\max} value was not sensitive to the particle size ($P_{\max} = 6.9$ bar). For diameters of 171 μm , the P_{\max} decreased to 5.8 bar. These values are different compared to the ones obtained by Bartknecht (1981) (P_{\max} of approx. 3 bar for dust particle size of 276 μm ; no explosion was observed by Amyotte et al. (2012) with this particle size). Here, again we can observe differences in the way to report the median diameter of the samples. Amyotte et al. (2012) characterizes the dust particles by volume median diameter and no details of whether Bartknecht used the volume, mass, or surface area to characterize the dust particle size could be obtained. It should also be noted that while Amyotte et al. (2012) uses a 20-liter sphere for their experiments, Bartknecht has used a 1 m^3 vessel for his experiments³⁸.

All the authors cited come to the same conclusion that with increasing dust particle size, the K_{St} decreases, and therefore the severity of the explosion also decreases.

In general, it was observed that P_{\max} tends to decrease above a particle size of approx. 100 μm . It is difficult to compare the results from different authors due to their differing methods of characterizing the dust particle size.

Another difference to note is the fact that Amyotte et al. (2012) use a 10 kJ chemical ignitor as an ignitor source in the 20-liter sphere, Britton et al. (1989) use “two centrally-placed, electric match-head igniters with a calorimetric energy of about 140 J each”⁴³ as an ignitor source in the 26-liter sphere, which represents a much higher ignition energy that may influence the results.

3.1.4 Minimum Ignition Energy (MIE) of polyethylene dust

There is in fact very few data related to the MIE of polyethylene dust in the literature. The ranges of MIE found in the literature were from 10 mJ to 1 J. The extreme variance in these values can be attributed to the differing dust particle sizes used by the experiments to find the MIE.

Table 9. Minimum Ignition Energy (MIE) of polyethylene dust

MIE	PARTICLE SIZE	Polyethylene type	EQUIPMENT	SOURCE
10 mJ	25 μm (*)	NA	Mike-3	7
1000 mJ	250 μm (*)	NA	Mike-3	7
500 mJ	136 μm (d50)	NA	Modified Hartmann Tube	37
10 - 30 mJ	28 μm (**)	NA	Mike-3	38
10 - 30 mJ	49 μm (**)	NA	Mike-3	38
10 - 30 mJ	103 μm (**)	NA	Mike-3	38

(*) mass median diameter

(**) volume median diameter

Bartknecht (1987) showed that MIE tends to increase linearly with the particle size (for 25 μm and 250 μm , MIE was found to be 10 mJ and 1000 mJ approximately, Figure 20).

The MIE for polyethylene dust measured by Traore et al. (2009) using a modified Hartmann tube was 500 mJ for a dust particle size of 136 μm .

Amyotte et al. (2012) showed that the MIE of polyethylene dust seems to be insensitive to the particle size for particle diameter of less than 103 μm (MEC between 10 to 30 mJ) but increases for particle diameter higher than 171 μm ³⁸.

The combined information from the authors above seem to indicate that the higher the particle size, the higher the MIE.

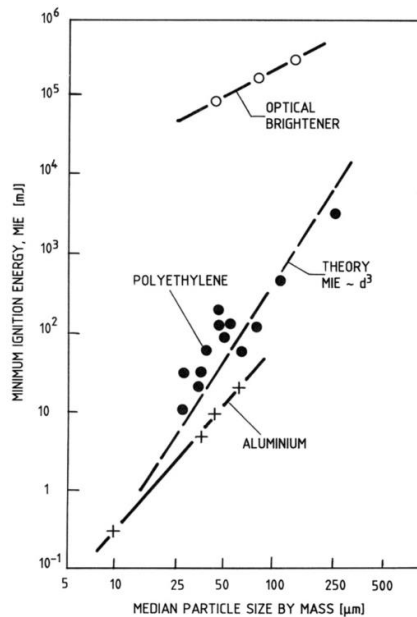


Figure 20. Effect of particle size on MIE of polyethylene dust. Reprinted from ⁷.

3.1.5 Minimum Ignition Temperature (MIT) of polyethylene dust

Hertzberg (1991) showed, using a 1.2-liter furnace, that for dust concentration below 100 g/m^3 (particle size of $37 \text{ }\mu\text{m}$ mean diameter), the ignition temperature decreases sharply as the dust concentration increases⁴⁶. Above this concentration, the ignition temperature seems to be constant. The MIT found is around 400°C (Figure 21)⁴⁶.

Mittal et al. (1996) made the same experimental observation with a Godbert-Greenwald furnace (Figure 22)³⁵ for a sample of particle size between $106\text{-}125 \text{ }\mu\text{m}$ (no information of the distribution is given)³⁵. However, he got a MIT slightly higher than Hertzberg (1991) (440°C).

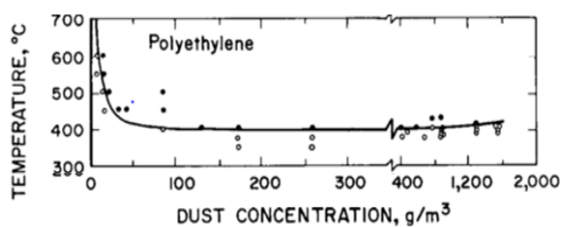


Figure 21. Autoignition data from the 1.2-liter furnace plotted as temperature versus concentration: ●, ignitions; ○, non-ignitions (particle size $37 \text{ }\mu\text{m}$)
Reprinted from ⁴⁶.

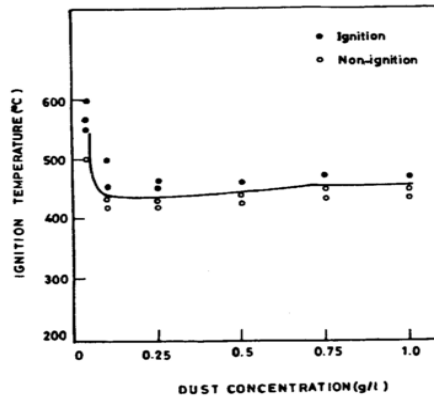


Figure 22. Variation of MIT with dust concentration for polyethylene (particle size between 106-125 μm)
Reprinted from ³⁵.

Table 10 summarizes the MIT data found in the literature. The MIT values found by Hartmann (1948) are mentioned in a paper by Mittal et al. (1997). It was reported that for experiments with increasing particle size from 116 to 328 μm , the MIT values increased from approx. 549 to 629°C. No other information about characterization of dust particle size or equipment used to conduct these tests were obtained.

Hertzberg (1991) showed, using a 1.2-liter furnace, that for particle size below 60 μm , the MIT of polyethylene is constant (400°C), and below 60 μm , MIT increases with the particle size (Figure 23).

Table 10. Minimum Ignition Temperature (MIT) of polyethylene dust

MIT	PARTICLE SIZE	Polyethylene type	EQUIPMENT	SOURCE
549°C	116 µm	NA	Godbert-Greenwald furnace	47
549°C	165 µm	NA	Godbert-Greenwald furnace	47
579°C	196 µm	NA	Godbert-Greenwald furnace	47
569°C	231 µm	NA	Godbert-Greenwald furnace	47
599°C	275 µm	NA	Godbert-Greenwald furnace	47
629°C	328 µm	NA	Godbert-Greenwald furnace	47
≈400°C	≈27 µm (*)	NA	1.2-liter furnace	46
≈400°C	≈55 µm (*)	NA	1.2-liter furnace	46
≈425°C	≈125 µm (*)	NA	1.2-liter furnace	46
≈450°C	≈310 µm (*)	NA	1.2-liter furnace	46
≈450°C	≈450 µm (*)	NA	1.2-liter furnace	46
440°C	106-125 µm ^f	LDPE	Godbert-Greenwald furnace	35
440°C	125-150 µm	LDPE	Godbert-Greenwald furnace	35
440°C	150-180 µm	LDPE	Godbert-Greenwald furnace	35
460°C	180-212 µm	LDPE	Godbert-Greenwald furnace	35
470°C	212-250 µm	LDPE	Godbert-Greenwald furnace	35
480°C	250-300 µm	LDPE	Godbert-Greenwald furnace	35
500°C	300-355 µm	LDPE	Godbert-Greenwald furnace	35
480°C	136 µm (d50)	NA	Godbert-Greenwald furnace, Modified Hartmann tube	37
370°C	28 µm (**)	NA	BAM Oven	38
400°C	49 µm (**)	NA	BAM Oven	38
410°C	103 µm (**)	NA	BAM Oven	38
375°C	≈ 75 µm (*)	NA	NA	48

(*) mass median diameter

(**) volume median diameter

^f The authors chose to represent the particle size interval by its upper limit (for example 106-125 µm is represented by 125 µm).

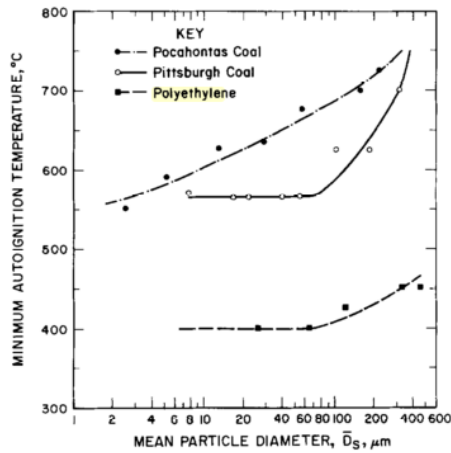


Figure 23. Particle size dependence for the MIT for polyethylene Reprinted from ⁴⁶.

Mittal et al. (1996) conducted a number of experiments using a Godbert-Greenwald furnace by varying the polyethylene dust particle size from 125 to 325 μm and the MIT value found for the lowest dust particle size was 440 $^{\circ}\text{C}$. As the particle size kept increasing, the MIT value kept increasing up to a value of 560 $^{\circ}\text{C}$.

Amyotte et al. (2012) used a BAM oven to measure the MIT for particle size range from 28 to 916 μm , and it was found that the dust particle sizes above 171 μm did not even ignite. The smaller particles below 171 μm did ignite and the MIT values kept decreasing from 410 $^{\circ}\text{C}$ to 370 $^{\circ}\text{C}$.

While all three papers come to the same conclusion regarding effect of dust particle size on MIT, it is difficult to compare their absolute values because of the difference of equipment used and particle characterization method.

3.2 Sulfur dust explosion properties

The literature has very few data for sulfur dust explosion properties and the effect of particle size, dust concentration, and polydispersity on them. Table 11 gives an overview of the sulfur dust explosion properties found in the literature.

Table 11. Dust explosion properties of sulfur dust

MEC (g/m ³)	P _{max} (bar)	K _{St} (bar-m/s)	MIE (mJ)	MIT (°C)	PARTICLE SIZE	EQUIPMENT	SOURCE
≈ 25	NA	NA	NA	280	NA	1.2-liter ceramic furnace	⁴⁶
100	5	28	NA	NA	35 μm (median)	20-liter sphere	⁴⁹
NA	NA	NA	NA	260	50 mm	BAM oven	⁷
NA	NA	NA	1.8 - 2.3	NA	NA	modified Mike-3	⁵⁰
NA	6.8	151	NA	NA	20 μm (*)	1 m ³ vessel	⁴¹
NA	7	113	NA	NA	20 μm	Tube-shaped burner	⁵¹
NA	NA	NA	4.5 - 5.9	NA	NA	Modified Hartmann Tube	³⁰
NA	NA	NA	1 - 3	NA	NA	Mike-3 apparatus	³⁰
2.5	5.3	214	1	210	25.7 μm (d50)	NA	⁵²
NA	NA	NA	6	270	110 μm (d50)	Godbert-Greenwald furnace (MIT), Modified Hartmann Tube (MIE)	⁵³
NA	NA	NA	NA	240	38 μm (*)	BAM oven	⁴⁸
NA	NA	NA	NA	260	38 μm (*)	Godbert-Greenwald furnace, 6.8-liter furnace	⁴⁸
NA	NA	NA	NA	290	38 μm (*)	1.2-liter furnace	⁴⁸

Table 11. continued.

MEC (g/m ³)	P _{max} (bar)	K _{St} (bar-m/s)	MIE (mJ)	MIT (°C)	PARTICLE SIZE	EQUIPMENT	SOURCE
NA	NA	NA	NA	250	NA	Ultrasonic waves	⁵⁴
30	6.8	251.3	0.38	210	<75 μm (mean = 35 μm)	20-liter sphere	²⁰
NA	6.3	204.3	3.4	230	75 μm (mean)	20-liter sphere	²⁰
NA	5.9	168.7	<1.3×10 ⁴	400	285 μm (mean)	20-liter sphere	²⁰
100 - 150	5.6	107.6	<1.3×10 ⁴	490	1400 - 1680 μm (mean = 1540 μm)	20-liter sphere	²⁰

(*) mass median diameter

(**) volume median diameter

3.2.1 Minimum Explosible Concentration (MEC) of sulfur dust

The range of MEC for sulfur dust was found to be between 2.5 to 150 g/m³, which is a very wide range. Just four authors give five values of MEC for sulfur dust throughout the literature. Hertzberg (1991) gives a value of around 25 g/m³, without mentioning the particle size of the dust sample tested. Cashdollar (1994) lists the MEC of sulfur dust to be approx. 100 g/m³, for a median particle size of 35 μm. Áñez et al. (2012) on the other hand gives an extremely low value of MEC (2.5 g/m³) for a median particle size of 25.7 μm. Yanqiu et al. (2014) is the only paper which gives two different MEC values for different particle sizes of sulfur dust, which are 30 g/m³ for a mean particle diameter of 35 μm, and an MEC between 100-150 g/m³ for a mean particle diameter of 1540 μm. With the limited amount of data and the different ways used by

the authors to characterize the dust particle size, no clear conclusion can be made in regards to the effect of particle size on MEC for sulfur dust.

3.2.2 Maximum pressure (P_m) and rate of pressure rise ($(dP/dt)_m$) of sulfur dust

The relationship between the dust concentration of sulfur and its P_m and $(dP/dt)_m$ values were shown by experimental results using a 20-liter sphere by Yanqiu et al. (2014). Similar to the correlation found with polyethylene dust, this paper concludes that for sulfur dust, both the P_m and $(dP/dt)_m$ increase with increasing dust concentration till a certain concentration where it reaches a critical value (800 g/m^3 for P_m , 1200 g/m^3 for $(dP/dt)_m$). Above this dust concentration, the P_m and $(dP/dt)_m$ decrease with increasing concentration (Figure 24)²⁰.

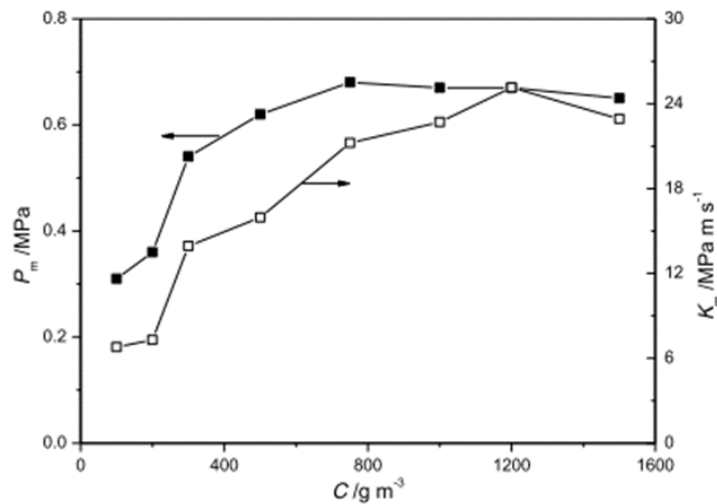


Figure 24. Influence of dust concentration on P_m and $(dP/dt)_m$
Reprinted from ²⁰.

3.2.3 *Maximum Explosion Overpressure (P_{max}) and Dust Deflagration Index (K_{St}) of sulfur dust*

The literature gave a range of P_{max} values from 3 to 7 bar and the values of K_{St} for sulfur ranged from 28 bar-m/s to 251.3 bar-m/s. Cashdollar (1994) just gives a single value of P_{max} and K_{St} of 5 bar and 28 bar-m/s respectively for a median particle size of 35 μm . Similarly, both NFPA (2007) and Silvestrini (2008) give similar P_{max} values of 6.8 and 7 bar respectively for a particle size of 20 μm . The K_{St} mentioned are quite different (151 bar-m/s and 113 bar-m/s respectively). Áñez et al. (2012) also just provides an individual value of P_{max} (5.3 bar) and K_{St} (214) for a median particle size of 25.7 μm . They do not provide information on the equipment used to measure these values. These values are different compared to both values reported by NFPA (2007) and Silvestrini et al. (2008) and differences in characterization of particle size and use of different equipment might explain the difference in these values.

Yanqiu et al. (2014) is the only paper that measures the effect of sulfur dust particle size on the P_{max} and K_{St} values, by varying the dust particle size from 35 to 1540 μm , and whereas the P_{max} values kept decreasing from 6.8 bar to 5.6 bar with increasing particle size, the K_{St} kept decreasing from 251.3 to 107.6 bar-m/s with increasing particle size²⁰. The K_{St} values obtained by Yanqiu et al. (2014) place sulfur in the St2 category of dust explosions, which translates to a strong explosion. OSHA, on the other hand, in a research paper classifies sulfur in the St1 category of dust explosions, without giving any specific K_{St} data for sulfur dust linked to particle size. The general

characteristic of St1 explosion is that it is a weak explosion and its K_{St} varies from 0 to 200 bar-m/s²⁰.

3.2.4 *Minimum Ignition Energy (MIE) of sulfur dust*

The range of MIE found in the literature for sulfur dust was between 0.38 mJ to 13 J. This is quite a varied range, and most of this variance comes from a single paper²⁰, and these results needs to be scrutinized and recreated in order to test the veracity of the data and the conclusions of the paper.

Randeberg et al. (2006) state the MIE of sulfur dust using a Mike-3 to be between 1.8 - 2.3 mJ. Similarly, Janes et al. (2008) give an MIE of 1-3 mJ using a Mike-3 and a MIE of 4.5 - 5.9 mJ using a modified Hartmann tube. None of these authors provide any other information in regards to dust particle size to draw any meaningful conclusions on the effect of dust particle size on MIE.

Whereas Áñez et al. (2012) provides a single MIE value of 1 mJ for a median particle size of 25.7 μm , Dufaud et al. gives an MIE value of 6 mJ for a median particle size of 110 μm . Yanqiu et al. (2014) is the only paper who varied the dust particle size from 35 to 1540 μm , and found that with increasing particle size, the MIE value increased from 0.38 mJ to more than 13 J²⁰.

3.2.5 *Minimum Ignition Temperature (MIT) of sulfur dust*

The relationship between the dust concentration and the ignition temperature of sulfur dust was shown by Hertzberg (1991) with experimental results. The equipment he

used was a 1.2-liter ceramic furnace which works very similar to a Godbert-Greenwald furnace. As the dust concentration increases when dust concentration is below 25 g/m³, the ignition temperature decreases significantly. The ignition temperature mostly remains constant as dust concentration is increased above 25 g/m³ (Figure 25)⁴⁶, and MIT value is approx. 320°C.

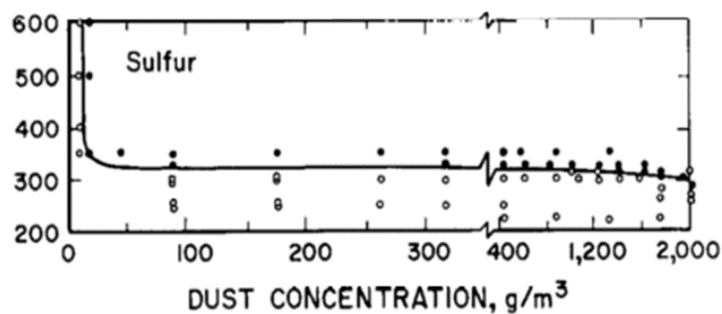


Figure 25. Effect of dust concentration on ignition temperature of sulfur dust Reprinted from ⁴⁶.

Many authors give singular values of MIT for a specific dust particle size. Some of them are: Eckoff (2003) reporting an MIT value of 260°C for 50 μm dust particle size using a BAM oven; Áñez et al. reporting a value of 210°C for a median particle size of 25.7 μm; and Dufuad et al. (2012) reporting a value of 270°C for a median particle size of 110 μm using a Godbert-Greenwald furnace. The ASTM E2109 standard gives different values of MIT for different equipment used, such as, BAM oven, 1.2-liter

furnace and a Godbert-Greenwald furnace for a particle size of 38 μm (mass median diameter).

As with other sulfur dust explosion properties, Yanqiu et al. (2014) is the only paper which measures the effect of dust particle size on MIT, and as the dust particle size was increased from 35 to 1540 μm (mean value), the MIT increased from 210°C to 490°C²⁰.

Yanqiu et al. also measured the effect of water content on both the ignition energy and ignition temperature for sulfur dust. They showed that with higher water content, both the ignition temperature and ignition energy keep rising, thus reducing the probability of a dust explosion occurring in the first place (Figure 26)²⁰.

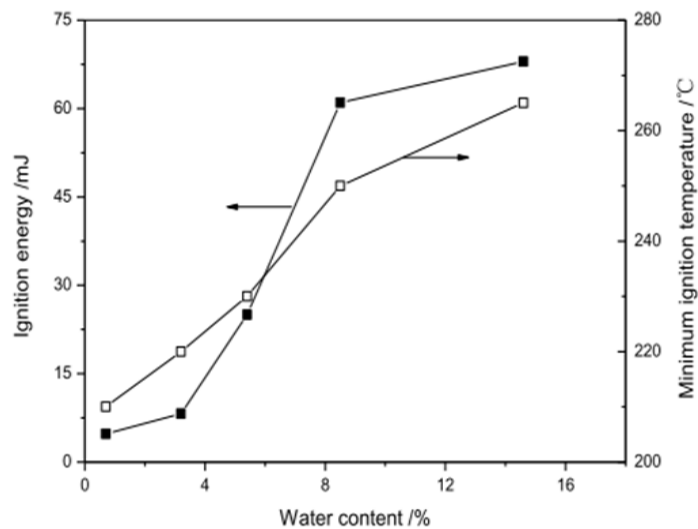


Figure 26. Influence of water content on ignition energy and ignition temperature of sulfur dust
Reprinted from ²⁰.

3.2.6 Limiting Oxygen Concentration (LOC) of sulfur dust

The range of limiting oxygen concentration (LOC) from the literature was found to be between 5% and 9%. *Áñez et al. (2013)* in their paper, report the LOC for sulfur to be about 5%. The sulfur dust used in their tests had a moisture of 0.1% and the $d_{0.1}$, $d_{0.5}$, and $d_{0.9}$ particle sizes were 6.1, 25.7, and 94.5 μm respectively²⁰. On the other hand, *Yanqiu et al.* reported the LOC to be about 9% for sulfur dust particle of size below 75 μm ²⁰. These LOC values are not comparable to each other because both *Yanqiu et al. (2014)* and *Áñez et al. (2013)* does not specify what particle size characterization they have used.

Table 12. Limiting Oxygen Concentration (LOC) of sulfur dust. Reprinted from ²⁰.

LOC	PARTICLE SIZE	EQUIPMENT	SOURCE
9%	< 75 μm	hot plate	²⁰
5%	25.7 μm		²⁰

3.3 Summary of the literature review

3.3.1 Polyethylene dust explosion properties

The literature gives a reasonable amount of data in regards to the effect of dust particle size on the dust explosion properties of polyethylene (MEC, MIE, MIT, P_{max} and K_{St}) (

Table 13). The issues associated with these data are the following:

- Most of the time the type of polyethylene dust used for the experiments is not mentioned. When the type of polyethylene is mentioned, most of the time the experiments were done with low density polyethylene (LDPE). Only one value of P_{\max} and K_{St} are available for HDPE. It is important to note that Qatar produces only LDPE but also HDPE, LLDPE, and MDPE. Therefore, applicability of the data found in the literature to polyethylene other than LDPE can be questioned.
- Most of the times, these papers do not mention the specific details of this particle size range. This would include no to little data on the d_{25} , d_{50} , d_{90} , mean, median, or mode of the dust particle size sample. The particle size is linked to usually either the volume of the dust particle, the mass of the dust particle, or the surface area of the dust particle. Many studies fail to mention which of these has been used to characterize the dust particle size. Therefore, the comparison between data is difficult.
- Dust explosions are measured using different equipment (e.g., 8-liter chamber, 20-liter sphere, and the 26-liter sphere), with different ignition methods and dust dispersion methods, which may generate difference in the measured values.

There is no study on the effect of humidity on MEC, MIE, and MIT for polyethylene dust. Only one author showed the variation of water content on P_{\max} and K_{St} (

Table 13).

Table 13. Summary of effect of critical parameters on polyethylene dust explosion properties

	MEC	MIE	MIT	P_{max}	K_{St}
Increase in Particle Size	Increases(#)	Increases	Increases(#)	Decreases (##)	Decreases
Increase in Water Content	No data found	No data found	No data found	Decreases	Decreases

(#)Increases only after a certain particle size
 (##)Decreases only after a certain particle size

3.3.2 Sulfur dust explosion properties

There is clear lack of data on Sulfur Dust Explosion Properties in the literature.

Only four authors give five values of MEC for sulfur dust throughout the literature. With the limited amount of data and the different ways used by the authors to characterize the dust particle size, no clear conclusion can be made in regards to the effect of particle size on MEC for sulfur dust.

There is some data for the MIT, MIE, P_{max}, and K_{St}, but the literature has little data in correlating the effect of these dust properties to the dust particle size.

Here again, for the available data, the following limitations are found:

- Different equipment are used to measure the explosion properties, which may generate difference in the measured values.

- It is difficult to compare the data due to the lack of or differences in particle size characterization.

No study of other important explosion characteristics, such as, MEC, P_{max} , and K_{St} , for sulfur dust hazards have been conducted which include humidity considerations. Only one author studied the influence of humidity on MIE and MIT.

4 RESEARCH OBJECTIVES AND METHODOLOGY

4.1 Research objectives

The objective of the thesis is to perform the experimental study of the minimum explosion concentration (MEC) of polyethylene and sulfur dust. The project aims to characterize the MEC for typical samples collected on-site at polyethylene and sulfur production facilities in Qatar, and study the influence of the particle size distribution of the polyethylene and sulfur dust on the MEC. In order to reach the above objectives, the research is structured as follows:

- Phase I: Sampling and physical characterization of the polyethylene and sulfur dusts found in the local industries of Qatar.
- Phase II: Experimental determination of the MEC for both polyethylene and sulfur dust collected on-site.
- Phase III: Study of the effect of particle size distribution on the MEC values of polyethylene and sulfur dust.

The generated experimental data focuses on filling the current gap in such data in the published literature and directly supports the Qatar industry in the improvement of the safety of polyethylene and sulfur production facilities. These data also provide a solid indication of the potential of dust generated by sulfur and polyethylene processes at different locations to create explosive atmospheres and the associated MEC for better risk control. Another unique and important outcome of this thesis is the characterization of the effect of particle size distribution on the MEC. The methodology to achieve these

objectives is described in detail in the sections below, which discuss the three phases of the project.

4.2 Methodology

4.2.1 Phase I: Analysis and physical characterization of the polyethylene and sulfur dusts found in the local industries of Qatar

Since this research was conducted to address the needs of the local industry in Qatar, it was central to understand the physical characteristics of the polyethylene and sulfur dusts found in the process facilities.

4.2.1.1 Sample Collection

In regards to polyethylene dust, samples were given to the research team by Q-Chem. Some of these samples were directly taken from the process areas when the company was undergoing a turnaround, and some of the samples were taken directly from the final product storage of the facility. Even if the samples were not collected by our research team directly, we visited the plant to better understand the polyethylene production process areas and interact with the process engineers.

The sulfur sample collection was different in that it was collected on-site from two different companies (Q-Chem and Qatargas). The research team visited both the plants and met the process engineers to gain an understanding of their process through technical documents. The samples of sulfur dust were taken directly by the research

team at locations prone to dust accumulation (following the indication of the process engineers).

4.2.1.2 Characterization of dust samples

The physical characterization of dust samples involved the measurement of particle size distribution, particle shape, chemical composition, and moisture content of the dust sample using a variety of different equipment.

4.2.1.2.1 Particle size distribution

The size of the dust particle was analyzed by using a laser diffraction analyzer (Beckman Coulter LS 13 320). This equipment can measure particle size measurement ranges from 0.017 μm to 2000 μm . It uses Mie theories of light scattering to determine the volume of a particle; the particle size is assumed to be the diameter of a sphere of same volume as the particle (Figure 27). The results of the analysis are displayed graphically as a volume fraction percent (or surface area percent or number percent).

A typical particle size analysis using this equipment is shown in Figure 28. Most of the particle size distribution tend to typically exhibit a bell shape curve.

The distribution is described using the diameters “dx” (d10, d25, d50, d75, d90) which indicates that x% (in volume) of the sample has a diameter less than “dx”. As an example, on Figure 28, d75 is 79.71 μm which means that 75% of the sample (volume) has a diameter of less than 79.71 μm . By definition, d₅₀ corresponds to the median of the distribution.

The mode is the most frequently occurring diameter of the distribution (peak of the distribution).

The mean of the distribution is the arithmetic average of the particle diameter.

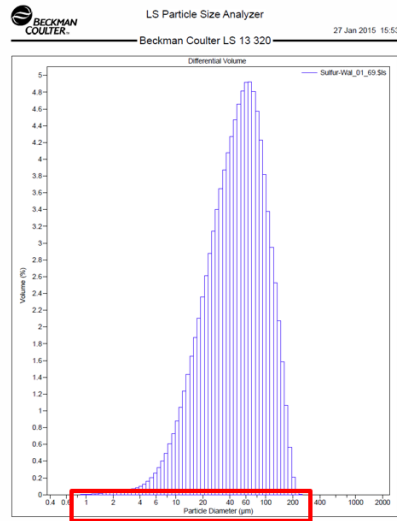
The distribution is characterized using both the variance (averaged of squared differences from the mean) and standard deviation (square root of the variance).

The relative distribution span can also be assessed to represent the distribution:

$$\text{Relative distribution span} = \frac{d_{90} - d_{10}}{d_{50}} \quad \text{Equation 3}$$



Figure 27. Beckman Coulter LS 13 320 particle analyzer



Laser Diffraction Particle Analyzer

Volume Statistics (Arithmetic)		Sulfur-Wal_01_69.\$ls		
Calculations from 0.375 µm to 2000 µm				
Volume:	100%	S.D.:	39.23 µm	
Mean:	57.89 µm	Variance:	1539 µm ²	
Median:	49.08 µm	C.V.:	67.8%	
Mean/Median ratio:	1.179	Skewness:	1.017 Right skewed	
Mode:	66.44 µm	Kurtosis:	0.706 Leptokurtic	
<10%	<25%	<50%	<75%	<90%
15.53 µm	27.30 µm	49.08 µm	79.71 µm	114.7 µm

d_{10} d_{25} d_{50} d_{75} d_{90}

Figure 28. Typical particle size range analysis results

4.2.1.2.2 Particle shape

The particle shape of the dust particle (Figure 29) was analyzed by a scanning electron microscope (SEM) FEI Quanta 400. This SEM uses energy dispersive spectrometry, wavelength dispersive x-ray spectroscopy, and electron backscatter diffraction to capture pictures of particle at even sizes of 10 µm.

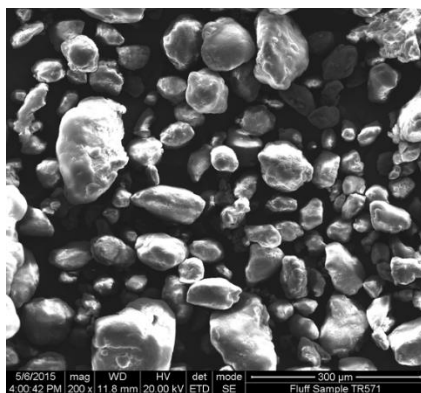


Figure 29. SEM pictures of a polyethylene dust sample

4.2.1.2.3 Sample chemical composition

The chemical composition of the sulfur dust samples is analyzed using x-ray fluorescence spectroscopy coupled with a wavelength-dispersive x-ray spectroscopy (XRF-WDS). The analyzer provides a quantitative determination of all the major and minor elements in the sample (Figure 31). The chemical analysis was not performed for the polyethylene samples.



Figure 30. FEI Quanta 400 Scanning Electron Microscope (SEM)

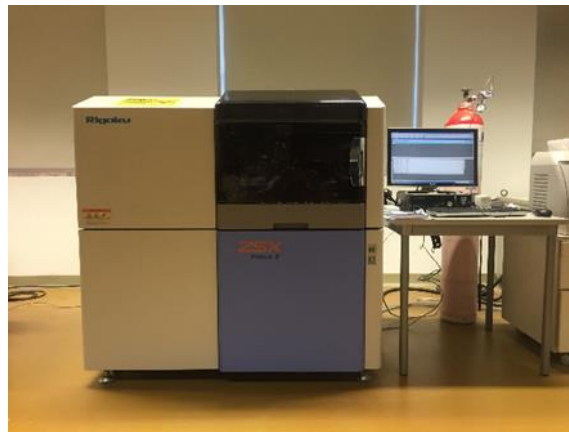


Figure 31. Rigaku ZSX Primus II WDXRF X-ray fluorescence spectrometer

4.2.1.2.4 *Sample humidity*

An attempt was done to assess the moisture content in the sulfur sample. The idea was to put the dust samples in oven at around 75°C and measure the weight change of the sample with time using a precision balance (1 mg precision).

4.2.2 *Phase II: Experimental determination of the MEC for both polyethylene and sulfur dust collected on-site*

Most of the MEC experiments were conducted using the modified Hartmann tube, for which a brief description was given in section 2.5.1. While the modified Hartmann tube is not the standard equipment to measure MEC, it was used as a screening tool to obtain a first estimation of the MEC. As mentioned in section 2.5.3.1, MEC is measured in a 20-liter sphere. At the time of the thesis, one of my tasks was to progress the installation of the 20-liter sphere in the laboratory. The sphere was successfully installed only toward the end of my research.

4.2.2.1 Experiments with the modified Hartmann tube

4.2.2.1.1 Description of the Equipment

A brief description of the modified Hartmann tube was given in section 2.5.1. Figure 32 shows the equipment at TAMUQ. It is composed of a 1.2-liter vertical plexiglass tube mounted onto a dust dispersion system (the dispersion cup). The tube is equipped with two electrodes between which a permanent spark (10 J) is generated

(approx. 10 cm above the dispersion cup). The dispersion cup is connected to a compressed air hose.

During the experiment, a given quantity of dust is dispersed by a single blast of air through the dispersion cup and the vertical tube, creating a dust cloud that is ignited or not by the permanent spark (Figure 33). The severity of the explosion is qualitatively recorded by a hinged cover on the top of the tube. The overpressure generated by the explosion may be sufficient to lift the pierced lid (with a 3 cm diameter hole) at the top of the tube. Two possible positions of the cover are recorded: Level 1 (lid lifted by a maximum angle of 45°), and Level 2 (lid lifted by an angle of $> 45^\circ$), each giving a qualitative indication of the severity of the explosion (Figure 34).

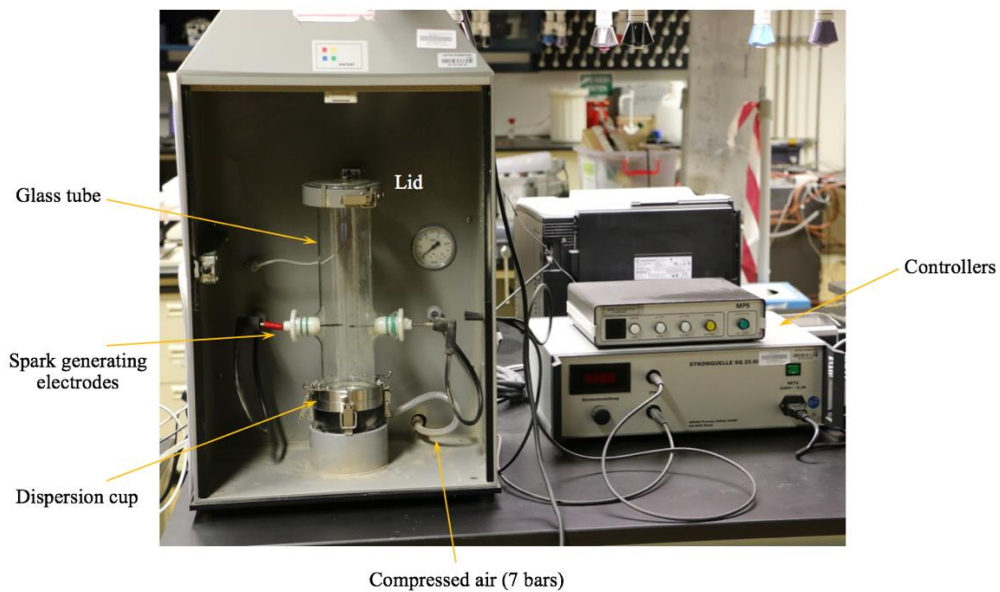


Figure 32. TAMUQ's modified Hartmann tube

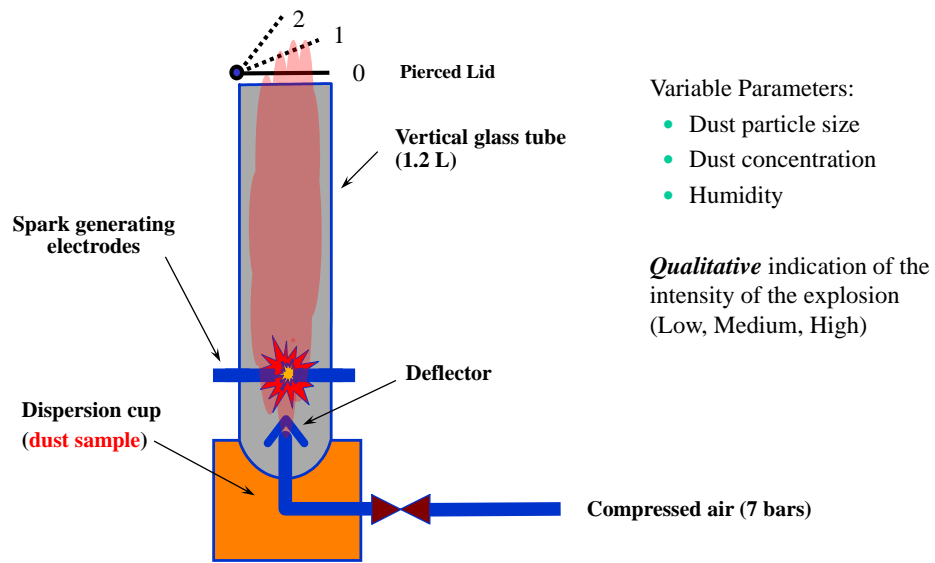


Figure 33. Schematic of a modified Hartmann tube

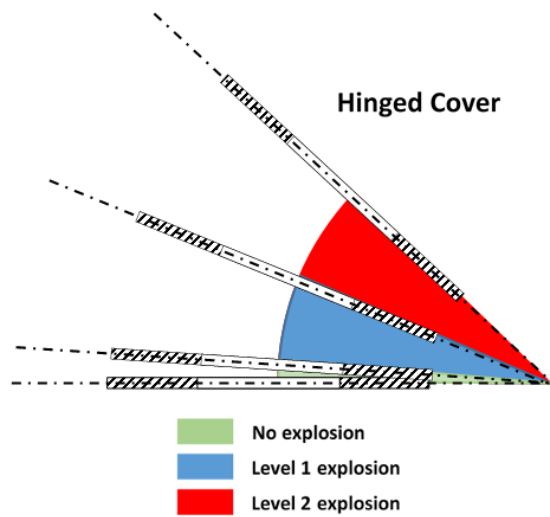


Figure 34. Schematic of pierced lid positions and its qualitative assumptions (modified Hartmann tube)

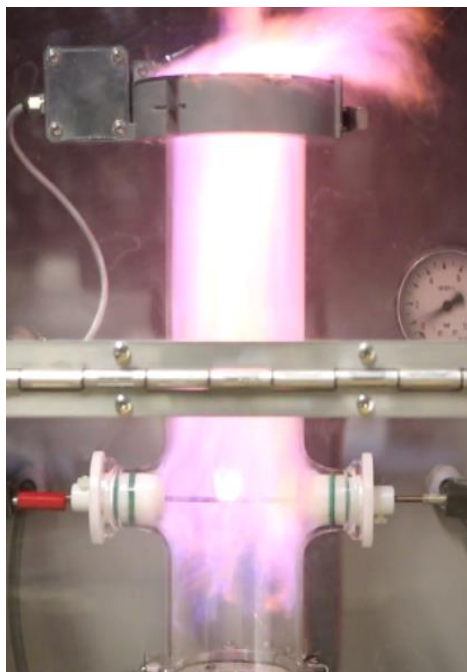


Figure 35. Picture of the modified Hartmann tube during an MEC measurements

4.2.2.1.2 *Safety*

Before conducting any experiments with the modified Hartmann tube, a comprehensive project hazard analysis of the experimental work to be performed was done with polyethylene and sulfur. I received proper training on the equipment from Dr. Luc Vechot (Responsible for the Lab), and I had to demonstrate that I understood the safe operation of the equipment.

4.2.2.1.3 Experimental procedure for MEC testing

The tests were carried out by varying the dust concentration of a given sample (collected on-site or of a specific dust particle size range) and observing the potential ignition and explosion of the sample.

The dust concentration was calculated as follows:

$$\text{Dust Concentration} = \frac{\text{Sample mass}}{\text{Volume of the tube}} \quad \text{Equation 4}$$

A petri dish was used to prepare the sample. The mass of the petri dish was taken *without the sample, with the sample, and without the sample after transfer to the dispersion cup*. These measures were taken to have an accurate estimate of the sample mass transferred to the dispersion cup.

For a given sample, the initial dust concentration was 100 g/m³. The concentration was increased by increments of 100 g/m³ (or less) until the ignition of the dust and its potential explosion occurred. The tests would be done up to 1000 g/m³ and then stopped if nothing was observed. On an average, up to 10 tests with the modified Hartmann tube needed to be performed to find the MEC of a single sample.

Each test was videotaped.

Every three or four tests, the plexiglass tube needed to be cleaned of any residual dust. When testing with polyethylene, a tissue was enough to clean the tube. Experimenting with sulfur was much more difficult and extremely time-consuming as melted sulfur stuck to the inner walls of the tube.

4.2.2.1.4 Interpretation of the results

The outcomes of the test were as follows:

- The dust would not ignite (no propagating fire observed).
- The dust would ignite and propagate a fire but without the generation of a pressure enough to lift the pierced lid. The dust concentration at which this happened was chosen to be the MEC value for that dust sample. The assumption was that this MEC would correspond to a lower flammability limit, leading to an overpressure in a confined environment (e.g., in a 20-liter sphere).
- The dust would explode and generate an overpressure that lifts the lid of the Hartmann tube to the Level 1 position. This could initially be classified as a weak explosion⁵⁵.
- The dust would explode and generate an overpressure that lifts the lid of the Hartmann tube to the Level 2 position. This could initially be classified as a strong explosion⁵⁵.

4.2.2.1.5 Quantification of uncertainties of MEC values

The data generated in these experiments, as with all data measured in any experiment, had some range of uncertainties in its final MEC values. These errors can be attributed to four sources:

- The first uncertainty is related to weighing the sample using the balance. The balance had a precision of +/- 1 mg, and as such, all samples weighed would also be affected this error. This error would have no significant effect on the results

obtained by the experiments in the modified Hartmann tube. This is because even for the smallest size samples, the lowest MEC value exhibited was at around 40 mg of dust, which would amount to a 2.5% error in the values reported. For larger samples, with larger MEC values, the error would be even lower in the decimal digits.

- The second source of error is related to the transfer of all the dust from the petri dish to the modified Hartmann tube. Some of the dust might have fallen to the sides or blown away while transferring the dust. While any dust stuck to the petri dish was accounted for in the final mass calculations, the dust blown or fallen to the sides have not been taken into account. While it is difficult to quantify this uncertainty, we did not observe dust being ejected from the tube following the experiment, and even if that was the case, we could not fully and reliably measure this.
- The third source of uncertainty has the largest impact on the final MEC results. The MEC values of the samples were measured by either increasing or decreasing the concentration of the dust, until a fire was observed. The difference between the concentration at which the dust is *ignited and propagates a fire* and the lower concentration of dust at which the dust *does not ignite* represents the recordable uncertainty in the final MEC values listed.
- The final source of uncertainty is related to the difficulty of the experimentalist to recognize a propagating fire in the tube. Two typical observations can be made when the dust is ignited:

- The dust ignites and shows a slow and limited propagation throughout the dust cloud in the tube. We refer to this observation as Beginning of Fire (BF).
- The dust ignites and shows a clear and fast propagation throughout the dust cloud in the tube. We refer to this observation as Propagating Fire (PF).

The difference between the concentrations at which BF and PF happens also represents the recordable uncertainty in the final MEC values listed.

4.2.2.2 Experiments with the 20-liter dust explosion sphere

The 20-liter dust explosion sphere is the standard equipment used to measure the MEC, P_{\max} , K_{St} , and LOC.

4.2.2.2.1 Description of the Equipment

The 20-liter sphere apparatus at TAMUQ (manufactured by Khuner, Switzerland) consists of a closed steel combustion chamber. The test chamber is a hollow sphere made of stainless steel, with a volume of 20 L and designed for a continuous operating pressure (design pressure) of 30 bar. A water jacket serves to remove the heat generated by the deflagration as to maintain thermostatically controlled test temperatures. For testing, the dust is dispersed into the sphere from a pressurized storage chamber ($V = 0.6$ L) via an outlet valve and a rebound nozzle. The outlet valve is

pneumatically opened and closed by means of an auxiliary piston; the valves for the compressed air are activated electrically.

Prior to dispersing the dust, the 20-liter sphere is partially evacuated (using a vacuum pump) to 0.4 bar absolute. This evacuation of the 20-liter sphere by 0.6 bar, together with the air contained in the dust storage chamber (+20 bar; 0.6 L), results in the desired starting pressure (1 bar) for the test.

When dispersed, the dust cloud is then ignited using two pyrotechnic igniters located in the center of the sphere with a total energy of 10 kJ (2×5 kJ). Piezoelectric pressure sensors are used to measure the pressure increases in the chamber during the experiment.

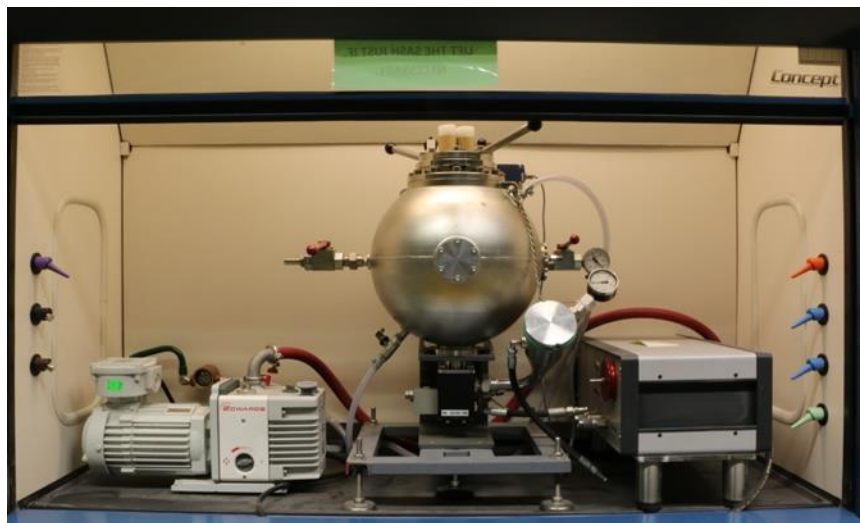


Figure 36. Pictorial representation of TAMUQ 20-liter dust explosion sphere

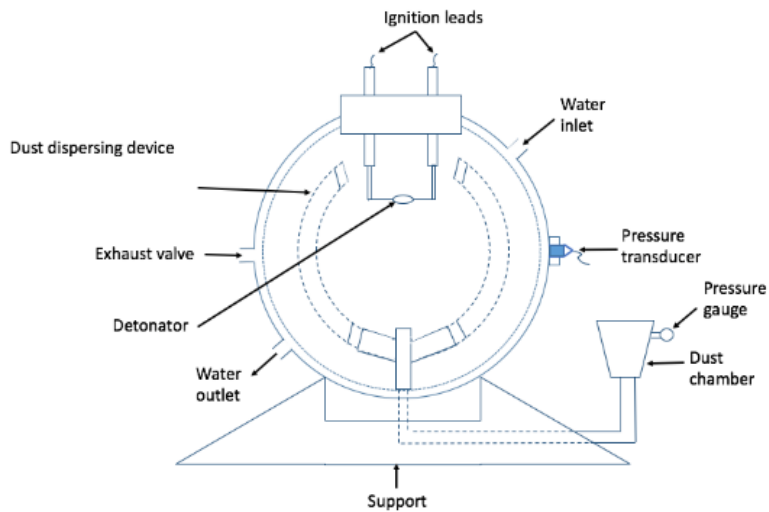


Figure 37. Schematic of 20-liter dust explosion sphere

4.2.2.2.2 *Safety*

A comprehensive project hazard analysis of the experimental work with the sphere was performed. The entire research group participated to a What-If Analysis to identify the hazards and assess the risks associated with the use of the sphere.

4.2.2.2.3 *Experimental procedure for MEC testing*

The ASTM E1515 standard was followed in conducting tests in the 20-liter sphere. The ASTM E1515 standard recommends to start testing a dust sample from a dust concentration of 100 g/m^3 , and if a deflagration occurs, then the dust concentration is reduced till no deflagration occurs. If a deflagration does not occur at 100 g/m^3 , then the concentration is increased until a deflagration occurs. This process is repeated till the

lowest concentration at which the dust explodes is found. This lowest concentration at which the dust explodes is the MEC value for that particle size value and range.

4.2.3 Phase III: Study of the effect of particle size distribution on the MEC values of polyethylene and sulfur dust

The third phase of the thesis involved the study of the effect of particle size distribution of polyethylene and sulfur dust samples on the value of the MEC. This required the production of samples of given particle size range from the on-site collected samples by sieving these on-site samples using calibrated sieves.

4.2.3.1 Preparation of the sample

In general, for one dust sample, about 1000 to 1500 mg amount of dust was needed to conduct the tests over the full range of dust concentrations in the modified Hartmann tube. In order to achieve this mass of dust, the larger particle sizes were grinded to a smaller particle size using a pestle and mortar (Figure 38). This is an extremely time-consuming process, and in order to achieve the desired mass for the finer particle size range, the original sample would have to be grinded for multiple hourly sessions over a period of days.



Figure 38. Pestle and mortar for dust grinding

4.2.3.2 Sieving methodologies

Two sieving methodologies were used in order to conduct experiments in Phase III, which were manual sieving and mechanical sieving. A sample for which we had a reasonable amount of quantity of dust was chosen to be sieved. The objective of the sieving process was to create relatively narrow particle size distribution.

4.2.3.2.1 Manual Sieving

The grinded dust was transferred onto a stack of three sieves. The sieves were stacked in order of decreasing particle size (e.g., 350 μm sieve, 250 μm sieve, 200 μm sieve). The top sieve would be taped up using a plastic or plumbers tape so that no dust flies or spills over while carrying out the sieving. The three sieves were shaken manually inside a fumehood, where a dust collector was placed beneath the bottom sieve to collect

the finer dust particle size. The sieves would then be shaken up to around 2 hours each day over multiple days to get two samples with two different particle size ranges (e.g., 250-350 μm , and 200-250 μm). Once about 1500 gm of each dust sample was collected, the process was repeated to get dust samples of different particle size ranges.

4.2.3.2.2 Mechanical sieving

The equipment used for sieving was the RoTap mechanical sieve shaker which oscillates horizontally at 287 oscillations per minute (Figure 39). Four sieves were stacked in order of decreasing particle size. The grinded dust was placed on top of the sieve and the sieves were vertically tapped at 150 taps per minute. The sieves used were the standard ASTM E-11 sieves which ranged from 20 to 425 μm . After about an hour of sieving, the mass of each sieve containing the dust sample was weighted and the sieving resumed. This process continued till there was no change in the mass of dust samples in the sieves. The average sieving time of about 300 grams of dusts was about 3 hours. The whole process was repeated till about 1500 gm of dust was collected for each particle size range.



Figure 39. TAMUQ mechanical sieve (RoTap RX-29 Sieve-Shaker)

4.2.3.3 Experimental determination of the MEC of the sieved sample

The sieved samples were then tested in the modified Hartmann tube with the same methodology as described in Section 4.2.2. This work also provided an opportunity to compare the manual or mechanical sieving process in the performance of the tests conducted with the modified Hartmann Tube.

5 EXPERIMENTAL RESULTS AND DISCUSSION

5.1 Experimental results for sulfur dust

5.1.1 Phase I: Analysis and physical characterization of sulfur dusts found on-site

5.1.1.1 Collection of Samples

The sulfur samples were collected from two different plants in Qatar with two different types of final sulfur product processes:

5.1.1.1.1 Granulated sulfur production process

Sulfur granules are produced in a granulation drum using a water spray technology. Liquid sulfur (at high temperature) is fed to the granulator and submitted to a water spray to form spherical granules of solid sulfur which fall on a rotating belt. These sulfur granules are scrapped off the rotating belt onto a dust discharge belt conveyor that transfers them onto a vibrating screen where the fine and coarse sulfur particles are separated. The granules from the vibrating screen equipment then are transferred to a silo (storage) via successive conveyor belts. The granules are moving from one conveyor belt to the other by gravity. This transfer occurs in an enclosed space represented by the dotted lines on Figure 40.

The experimental tests were conducted on five out of seven on-site samples of sulfur taken from the plant which produced granulated sulfur. Table 14 also details from which areas of the plant these dust samples were taken from. Most of the tests performed in this thesis were done with these sulfur samples.

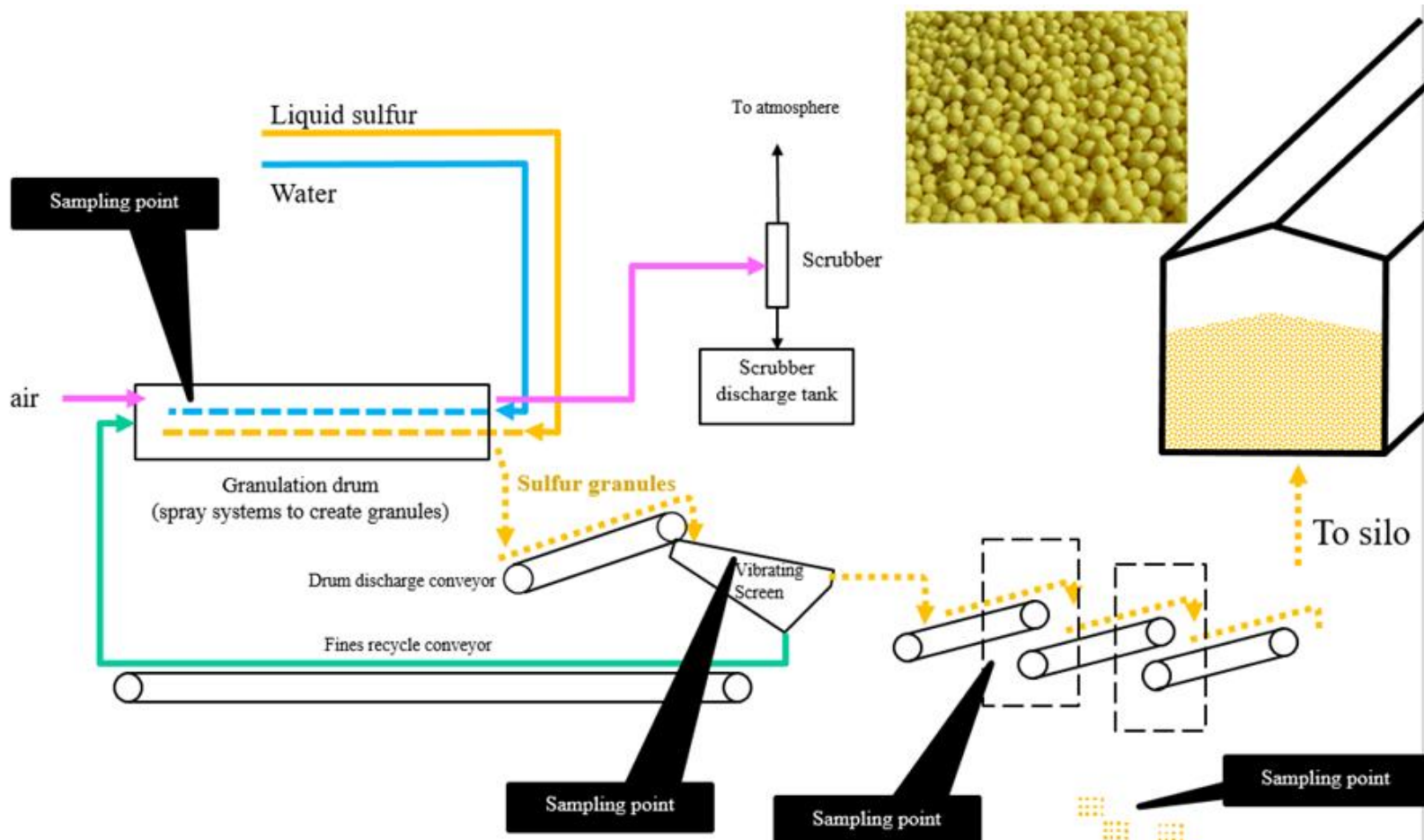


Figure 40. Graphical illustration of process for granulated sulfur samples

Table 14. Origin and identification of sulfur samples

ORIGIN	DESCRIPTION	DUST SAMPLE ID
Granulator	Sulfur dust from the granulator	QG_Sulfur_G
Scrappers	Sulfur dust spillage from Scrappers	QG_Sulfur_Scrp
Vibrating screen	Sulfur dust from the vibrating screen	QG_Sulfur_VS
Conveyor belt	Sulfur dust Spillage from conveyor belt	QG_Sulfur_CB
Wall on chute	Sulfur dust in the wall on chute	QG_Sulfur_WC
Conveyor to structure	Sulfur dust on conveyor to structure	QG_Sulfur_C-S
Storage	Sulfur granules final product from storage	QG_Sulfur_FP

5.1.1.1.2 Prilled sulfur production process

Hot liquid sulfur is fed into an accumulator where droplets of liquid are generated. These droplets fall by gravity onto a water-cooled conveyor belt where they solidify, thus forming sulfur pellets. These pellets are scrapped off the belt and dropped into buckets of a bucket conveyor system. The sulfur pellets are conveyed to the top of the sulfur storage silo where they fall onto a pile of sulfur.

Table 15 also details from which areas of the plant all of these dust samples were taken from. These on-site samples were not used by this research to measure their MEC values. Therefore, no dust particle size characterization was done for all of these samples. These dust samples were used for subsequent tests by the research team at the center.

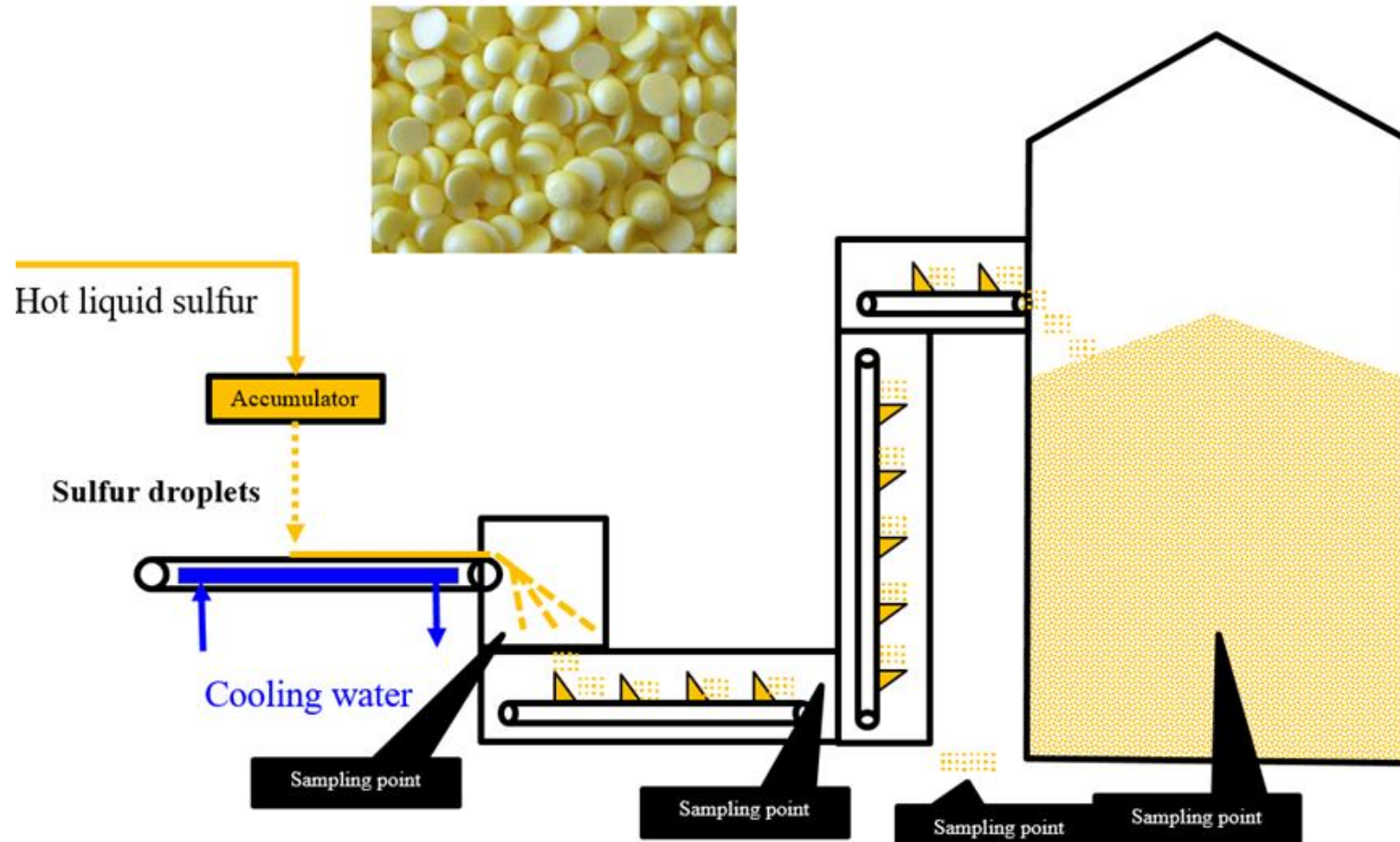


Figure 41. Graphical illustration of process for prilled sulfur samples

Table 15. Origin of prilled sulfur samples

ORIGIN	DESCRIPTION	DUST SAMPLE ID
Storage Silo	Sample was taken from the storage silo	QChem_Sulfur_S
Buckets	Sample was taken from buckets which contain the scrapped prilled sulfur dust	QChem_Sulfur_B
Conveyor line	On the floor (sulfur dust spillage) around the conveyor line	QChem_Sulfur_C
Scrapping Area	Scrapping area at the end of the water-cooled belt	QCHEM_Sulfur_Scrp

5.1.1.2 Particle size distributions

Table 16. Summary of particle sizes for granulated sulfur samples

PARTICLE SIZE DISTRIBUTION	Particle size range (μm)	D10 (μm)	D25 (μm)	D50 (μm)	D90 (μm)	Relative span (μm)	MEAN (μm)	MODE (μm)	Particle Shape
QG_Sulfur_FP	-	2522	3556	4641	6285	0.81	5828	5580	-
QG_Sulfur_G	-	904.9	1127	1420	2326	1	2169	1512	-
QG_Sulfur_C-S	1-800	13.47	22.58	40.62	125.6	2.76	67.46	45.75	Irregular
QG_Sulfur_WC	1-200	15.53	27.30	49.08	114.7	2.02	57.89	66.44	Irregular
QG_Sulfur_CB	4-<2000	444.4	762.4	1156	1834	1.20	1144	1739	Spherical
QG_Sulfur_Scrp	4-<2000	376.3	810.2	1263	1848	1.16	1187	1909	Irregular
QG_Sulfur_VS	8-<2000	74.11	107.9	189.7	910	4.40	336.4	127.6	Irregular

The sample of QG_Sulfur_FP contained final products of sulfur granules (4.6 mm in size) from the storage silos and as such did not contain much dust particle (Table 16).

Three other samples taken from the plant (QG_Sulfur_Scrp, QG_Sulfur_CB, and QG_Sulfur_G) contained mostly sulfur granules with a mean particle size of more than 1000 μm , and very few dust particle sizes below 420 μm , above which the NFPA does not consider the particles to be dust sizes (Figure 42).



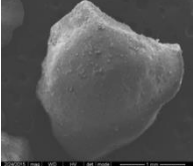
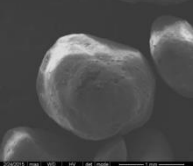
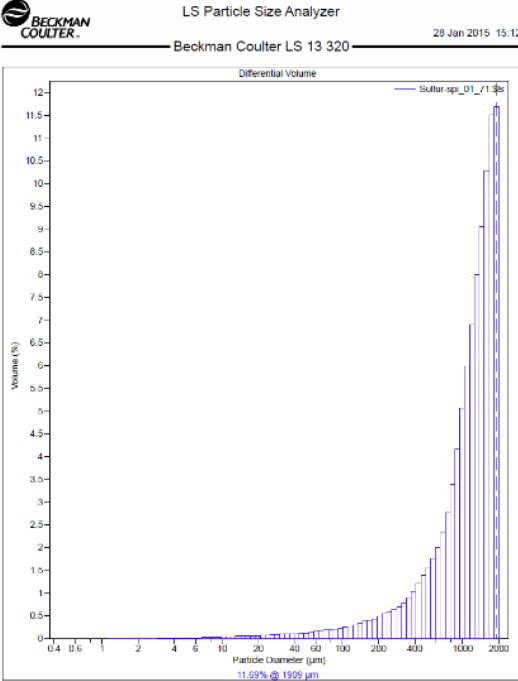
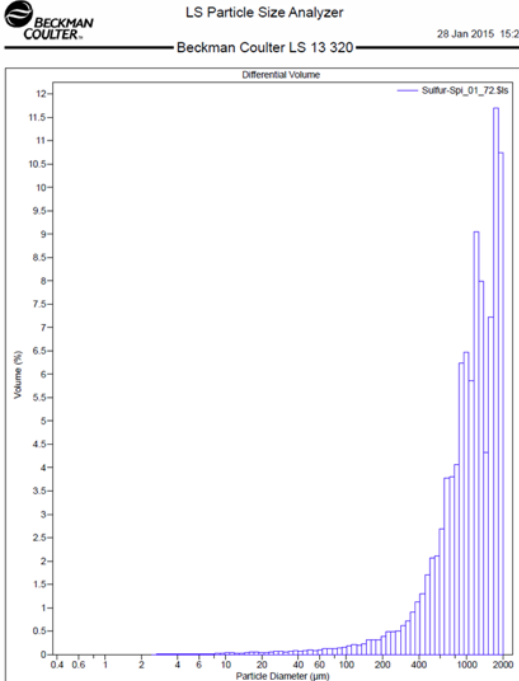
Sample	QG_Sulfur_Scrp		QG_Sulfur_CB	
Photo				
SEM Picture				
Particle size distribution				

Figure 42. Particle size characterization of sulfur dust collected on site



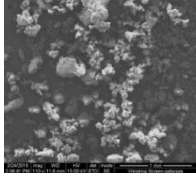
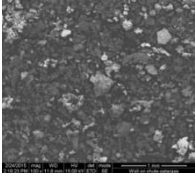

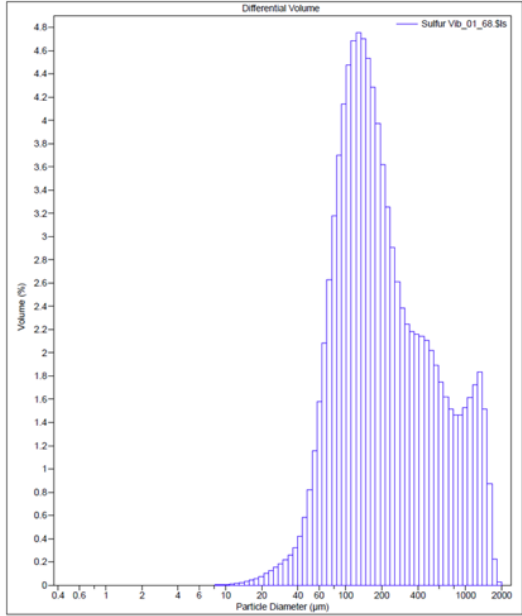

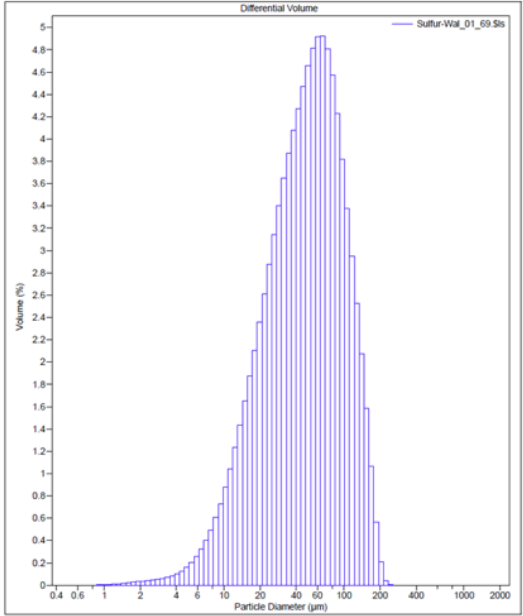
Sample	QG_Sulfur_VS	QG_Sulfur_WC
Photo		
SEM Picture		
Particle size distribution	<p data-bbox="569 634 1087 688">  LS Particle Size Analyzer Beckman Coulter LS 13 320 27 Jan 2015 15:44 </p> 	<p data-bbox="1257 634 1776 688">  LS Particle Size Analyzer Beckman Coulter LS 13 320 27 Jan 2015 15:53 </p> 

Figure 42. continued.


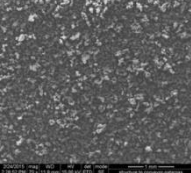
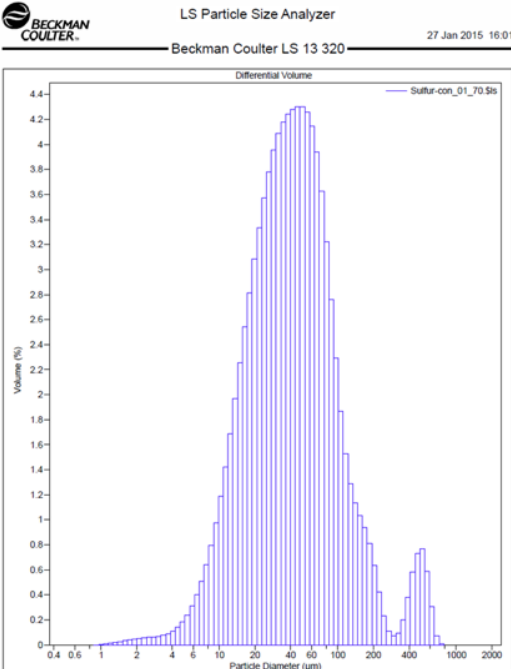
Sample	QG_Sulfur_C-S
Photo	
SEM Picture	
Particle size distribution	<div style="text-align: center;">  </div>

Figure 42. continued.

The sample collected from the vibrating screen (QG_Sulfur_VS), had a very wide range of 4 to 2000 μm , but its d50 was quite low at 189 μm . The distribution shows two peaks, which may indicate potential contamination. It is expected to find both a wide particle size range and a low d50 for this sample, as this dust has been taken from the vibrating screen equipment, and the primarily role of this equipment is to separate the fine and coarse particles.

The samples of QG_Sulfur_C-S and QG_Sulfur_WC had very fine particle sizes with d50 of 40 μm and 49 μm respectively. The striking detail in comparing the particle sizes of these samples is that both these samples have very similar mean, mode, d10, d25, d50, d75, d90, and relative span, but their range of particle sizes are very different. When looking at the particle size distribution in **Error! Reference source not found.**, QG_Sulfur_WC has two peaks. The second peak on the right seems to indicate that QG_Sulfur_WC was contaminated with a small quantity of sample having a mode diameter around 500 μm . This may affect the values of mean, mode, d10, d25, d50, d75, and d90 for this sample.

If we look more carefully at the particle size distribution in **Error! Reference source not found.**, it seems that QG_Sulfur_C-S has finer particles than QG_Sulfur_WC. This being said QG_Sulfur_C-S and QG_Sulfur_WC really look alike. QG_Sulfur_C-S has a range of up to 800 μm , whereas QG_Sulfur_WC had a range of just up to 200 μm . This would indicate that up to 90% of the particles in these samples are of similar size, but in the QG_Sulfur_C-S sample, the rest 10% of particles are of a very large size. As such, it would be interesting to compare the explosibility result of

these samples and see if these large particles had any effect on the explosibility and MEC results.

5.1.1.3 Chemical composition analysis

Table 17 gives the chemical composition of all the sulfur samples collected on-site. The prilled and granulated sulfur samples both have similar chemical composition and the same crystallographic properties (XRD).

Table 17. Chemical composition of sulfur dust samples

	S	C	O	Na	Cl	Mg	Al	Si	P	K	Ca	Fe
Qchem_Prilled Sulfur_FP	90.1	7.98	1.67		0.0578	0.0189	0.0529	0.0527			0.0483	0.0097
QG_Sulfur_FP	98.9		0.909	0.0226	0.0762	0.019	0.0104	0.0141			0.0171	
QG_Sulfur_G	90.1	2.09	5.03	0.87	1.56	0.183	0.0792	0.0209		0.0304	0.0356	
QG_Sulfur_C-S	89.1	3.8	5.75	0.175	0.234	0.146	0.12	0.275	0.0045	0.0207	0.329	0.0307
QG_Sulfur_WC	84.3	11.1	4.23	0.0719	0.0828	0.0461	0.0371	0.0762	0.0041	0.0574	0.0754	0.0101
QG_Sulfur_CB	95.3	2.67	1.79		0.035	0.0175	0.0272	0.0543		0.0076	0.0518	
QG_Sulfur_Scrp	91.8	1.98	5.47		0.0303	0.0902	0.0654	0.252		0.0093	0.263	
QG_Sulfur_VS	37.8	60.1	1.81	0.0327	0.337	0.0293	0.0405	0.0538	0.0038	0.0031	0.0433	0.0049

All the samples contain more than 84% of sulfur, except the sulfur collected at the vibrating screen which only has 38% of sulfur. Carbon is the major contaminant for

all the samples except for the sample collected at the storage facilities. A reason for carbon being the major contaminant could be that the carbon from the conveyor belts gets mixed with the sulfur dusts being transported throughout the unit. The purest sample in the subset is that of QG_Sulfur_FP, which contains more than 99% sulfur, which is expected as this is the final product found in the storage silos.

5.1.1.4 Humidity

The samples were also tested for the moisture content before conducting experiments in the Hartmann tube. The sulfur dust was placed in a petri dish and left in a heating oven for around 7 days at 50°C. The weight of the samples was chosen to be around 500 mg to minimize the error in measurement of the potential water lost. There was no weight change in the sulfur sample after heating, and thus it was concluded that there was minimal moisture in the samples as there was no discernible difference in the weights before and after heating the sample.

5.1.2 Phase II: Experimental determination of the minimum explosible concentration (MEC) for sulfur dust collected on-site

Three of the five samples collected from the plant (granulated sulfur) recorded a fire or an explosion in the modified Hartmann tube.

Table 18 and Figure 43 both give a detailed overview of these results. The table and the figure indicate the volumetric mean, median, and mode for each sample (please

refer to Table 16 for the complete particle size distribution). For each of the samples, we recorded the concentration for each of the following:

- A fire is propagated; this concentration was taken as the MEC.
- An explosion Level 1 and Level 2 occurs; this information gives a qualitative indication of the severity of the explosion.

Table 18. Summary of explosibility results for on-site sulfur dust samples

SAMPLE	Mean particle size diameter (μm)	Median particle size diameter (μm)	Mode particle size diameter (μm)	Propagating Fire observed / MEC (g/m^3)	LEVEL 1 explosion (g/m^3)	LEVEL 2 explosion (g/m^3)
QG_Sulfur_Scrp	1187	1263	1909	No Fire	No Fire	No Fire
QG_Sulfur_CB	1144	1156	1739	No Fire	No Fire	No Fire
QG_Sulfur_VS	336.4	189.7	127.6	94.16 (-14) [§]	No Explosion	No Explosion
QG_Sulfur_WC	57.89	49.08	66.44	75 (-25)	87.5 (-11)	170 (-24)
QG_Sulfur_C-S	67.46	40.62	45.75	54.16 (-16)	89.16 (-34)	112.5 (-14)

[§] The numbers in the brackets indicate the possible error in measuring these values using the modified Hartmann tube.

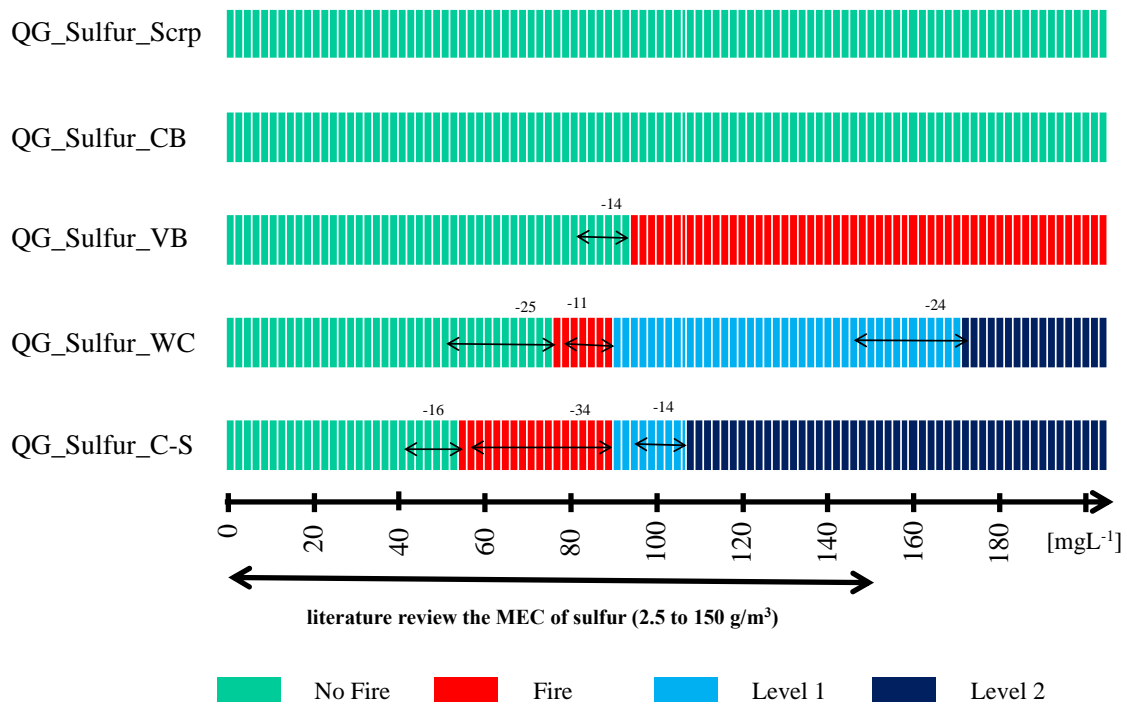


Figure 43. Explosibility results for on-site sulfur dust samples

The sulfur samples taken from the scrapper and the conveyor belt (QG_Sulfur_Scrp and QG_Sulfur_CB) did not ignite in the modified Hartmann tube. The d10 for these sample were 376 μm and 444 μm respectively, making these samples one of the larger sulfur dust samples. In fact, NFPA stops classifying particles as dust at more than 420 μm , and as such, NFPA would not consider these samples as dusts. These results show the dust generated at the scrapper and the conveyor belt (with large particle size) are not likely to be involved in an explosion.

The sample for the vibrating screen (QG_Sulfur_VS) ignited and propagated a fire at 94.6 g/m^3 but did not generate an overpressure in the tube even at very high

concentrations (up to 1000 g/m³). The median for this sample is 189.7 μm but the distribution shows two peaks, which may indicate potential contamination. This sulfur sample though was heavily contaminated with carbon (60% of the content – see Table 17) and as such it is very difficult to draw any conclusions or compare any of this data to the one present in the literature. Carbon dust itself is a explosible dust which has generally quite a low MEC. There is no data present in the literature which measure MEC for a mixture of carbon and sulfur dust. This result shows that dust collected at the vibrating screen can be involved in a dust explosion, but we cannot say that this is entirely due to sulfur since the sample is contaminated with carbon.

The samples from the wall on chute (QG_Sulfur_WC, 85% sulfur) contained very fine particles (median diameter of 49.08 μm). It ignited at values at around 75 g/m³. Taking into account the uncertainty, we found an MEC between 50 g/m³ and 75 g/m³. For this sample, we observed a Level 1 explosion for quite a wide range of concentrations (from 87.5 to 170 g/m³).

The samples from the conveyor to structure (QG_Sulfur_C-S, 89% sulfur) contained very fine particles (median diameter of 40.62 μm). It ignited at values at around 54 g/m³. Taking into account the uncertainty, we found an MEC between 38 g/m³ and 54 g/m³. For this sample, the transition from Level 1 to Level 2 explosions occurred over a narrow range of dust concentrations (from 89.16 to 112.5 g/m³), unlike QG_Sulfur_WC. The potential contamination of QG_Sulfur_C-S as explained in section 0 may explain the differences of MEC values and explosions behavior for levels 1 and 2

with QG_Sulfur_WC. QG_Sulfur_C-S also has finer particles than QG_Sulfur_WC, which may lower the MEC.

The results above clearly show that the dusts generated at the wall on chute and conveyor to structure are very prone to dust explosion, and we can qualitatively say that the explosion may be more severe than dust generated at the vibrating screen. It is vital to remember that the dust in the wall on chute is present in an enclosed space which would certainly affect the severity of the dust explosion at this location.

There is no literature data on MEC in the range of particle size of QG_Sulfur_WC and QG_Sulfur_C-S ($40\ \mu\text{m} < \text{median diameter} < 50\ \mu\text{m}$), so our results give a first indication of the MEC for fine sulfur dust ($38\ \text{g/m}^3 < \text{MEC} < 75\ \text{g/m}^3$).

However, these results do not represent pure sulfur.

Figure 44 shows pictures of the fire / explosion of sulfur dust in the modified Hartmann tube for QG_Sulfur_VS, QG_Sulfur_WC, and QG_Sulfur_C-S.

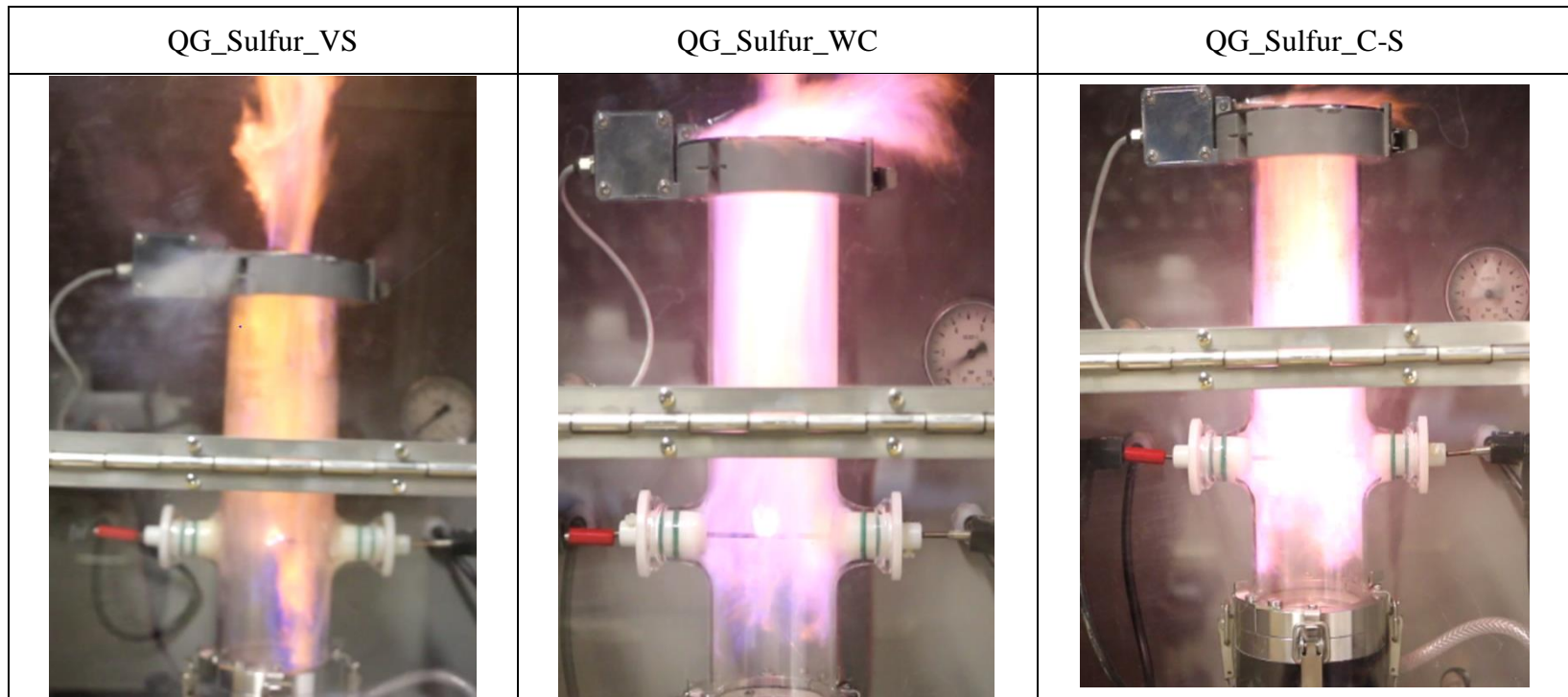


Figure 44. Pictures of the fire / explosion of sulfur dust (collected on-site) in the modified Hartmann tube

5.1.3 Phase III: Effect of particle size distribution on the MEC values of sulfur dust

The sulfur sample that was chosen to be mechanically sieved was the granulated sulfur sample, which was the final pure sulfur product received in granular form (QG_Sulfur_FP). This dust sample was grounded painstakingly for days and then mechanically sieved into 6 particle size ranges. These were 0-53 μm , 63-70 μm , 90-106 μm , 125-150 μm , 150-180 μm , and 355-425 μm .

These sieved dust samples could not be analyzed in the particle size analyzer as not enough amount of dust could be sieved to both conduct experiments and measure the samples through the particle size analyzer.

Therefore, we decided to present the results as a range of dust particle size on the basis of the sieves used for the sieving or the arithmetic mean assuming a perfect bell shape distribution.

Generally, for the results obtained using the six sieved samples, there is not much data in the literature to compare our results to. Only one of these samples did not either exhibit a fire or an explosion in the modified Hartmann tube.

Table 19 gives a summary of all the explosibility results of the six sieved samples and Figure 45 provides a graphical representation of this data.

For the on-site samples, we observed very clear propagation of fires, so the conclusion on MEC was somehow easy to obtain. For the sieved sample, this decision on whether we have an MEC or not was not that clear. Indeed, because of the narrow range of particle size, there were 2 typical observations when the dust ignited:

- The dust ignites and shows a slow and limited propagation throughout the dust cloud in the tube. We refer to this observation as Beginning of Fire (BF).
- The dust ignites and shows a clear and fast propagation throughout the dust cloud in the tube. We refer to this observation as Propagating Fire (PF).

Therefore, we concluded that the MEC would be somewhere between the concentrations for BF and PF.

Table 19. Summary of explosibility results of sieved sulfur samples

SAMPLE	PSD RANGE (μm)	MEC (g/m^3)	LEVEL 1 explosion (g/m^3)	LEVEL 2 explosion (g/m^3)	Comparable MEC from literature (g/m^3)
QP_Sulfur_FP	0-53	37	47	109	30 (Yanqiu et al., 2014) 100 (Cashdollar et al, 2007) 2.5 (Áñez et al, 2017)
QP_Sulfur_FP	63-75	48	201	NA	
QP_Sulfur_FP	90-106	57	822	No Explosion	
QP_Sulfur_FP	125-150	175	NA	NA	
QP_Sulfur_FP	150-180	578	No Explosion	No Explosion	
QP_Sulfur_FP	355-425	No Fire	No Explosion	No Explosion	150 (Yanqiu et al., 2014)

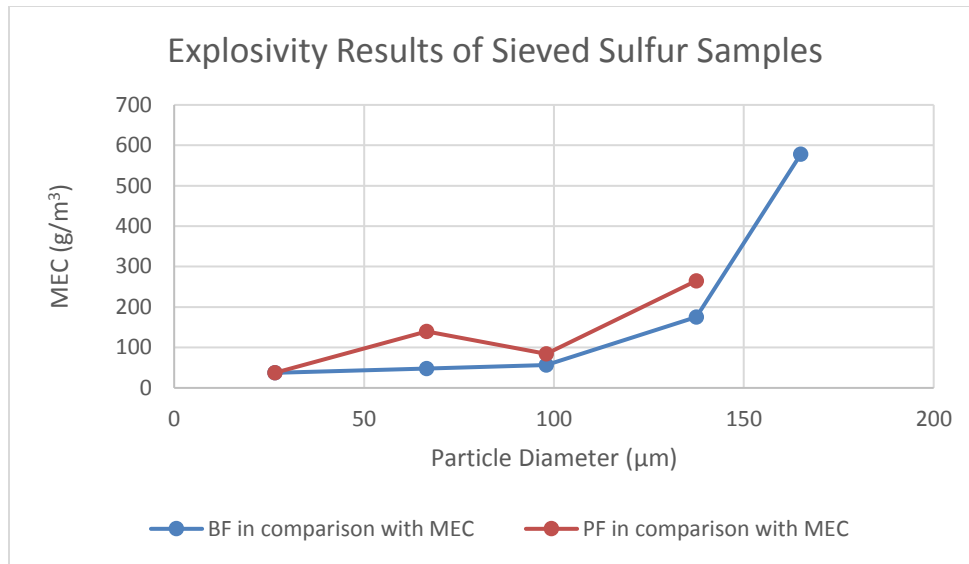


Figure 45. Graphical summary of explosibility results of sieved sulfur samples

5.1.3.1 Sample of 0-53 μm

The smallest sieved sample of 0-53 μm ignited and propagated a fire at 37.34 g/m³, gave a Level 1 explosion at 37.34 g/m³, and a Level 2 explosion at 108.9 g/m³. The MEC is therefore relatively low (37.34 g/m³). There was also a transition from a propagating fire to Level 1 explosion occurring over a small range of concentration. This experimental result agrees with Yanqiu et al. (2014), who found an MEC value of 30 g/m³ for a dust particle mean size of 35 μm²⁰ using a 20-liter sphere.

Cashdollar et al. found an MEC value of 100 g/m³ for a particle size range of 10 to 50 μm⁵⁶ using a 20-liter sphere, and Áñez et al. found an MEC value of 2.5 g/m³ for a particle size (d50) of 25.7 μm⁵² (equipment not mentioned), which are very different from our results.

In regards to the value found by Áñez et al., such a low value of MEC has not been found anywhere for any other dust. OSHA also states that for most dusts, the lower explosive limit is usually 15 g/m^3 ⁵². Thus, this data needs to be scrutinized for accuracy and applicability in regards to sulfur dust.

5.1.3.2 Sample of 63-70 μm

The sieved sample of 63-70 μm ignited a fire (BF) at 47.98 g/m^3 , propagated a fire (PF) at 140.00 g/m^3 , gave a Level 1 explosion at 200 g/m^3 , but no Level 2 explosion was recorded. The uncertainty on MEC is high for this sample.

5.1.3.3 Sample of 90-106 μm

The sieved sample of 90-106 μm ignited a fire (BF) at 56.95 g/m^3 , propagated a fire (PF) at 84.43 g/m^3 , gave a Level 1 explosion at 821.67 g/m^3 , but no Level 2 explosion was recorded. Here, while the uncertainty of MEC is less than the previous sample, we can observe that the MEC is quite low but the severity of the explosion seems to be decreasing at this particle size range.

5.1.3.4 Sample of 125-150 μm

The sieved sample of 125-150 μm ignited a fire (BF) at 175.00 g/m^3 , propagated a fire (PF) at 265 g/m^3 , but no significant overpressure for a Level 1 explosion was recorded even at concentration up to 1000 g/m^3 . At this particle size, the MEC clearly increases and the severity of potential explosion decreases.

5.1.3.5 Sample of 150-180 μm

The sieved sample of 150-180 μm ignited a fire without clear propagation (only BF) at 578.33 g/m^3 , but no significant overpressure for a Level 1 explosion was recorded even at concentration up to 1000 g/m^3 .

For all the samples with dust particle size ranging from 63 to 180 μm , there was no data in the literature for this particle size to compare the MEC results to.

5.1.3.6 Sample of 355-425 μm

For the largest particle size sample of 355-425 μm , it was found that sulfur dust is not ignitable in Hartmann tube at this particle size range. This experimental result disagreed with the results found by Yanqiu et al., which found an MEC of 150 g/m^3 for particle size ranging from 1400 to 1680 μm ²⁰. Even though the characterization of the dust particle sizes is different for both our research and Yanqiu et al., the results obtained by Yanqiu et al. have to be scrutinized as a particle stops being classified as dust after 425 μm , and even extremely explosible dusts like aluminum dust do not explode at this range.

As is shown in Figure 45, even if significant uncertainties are generated by the use of the Hartmann tube, the MEC of sulfur dust is dependent on the particles sizes. It can be noticed that dust particle size has very limited influence on MEC up to a dust particle size of around 100 μm . Above this particle size, the MEC increases with increasing particle size, indicating a strong effect of dust particle size on MEC values.

Hertzberg et al. (1982) finds a similar effect of particle size on MEC at lower particle sizes for both polyethylene and coal dust³³. Figure 45 is the major finding of this thesis.

5.2 Experimental results for polyethylene dust

5.2.1 Phase I: Analysis and physical characterization of polyethylene dusts found in the local industries of Qatar

5.2.1.1 Collection of Samples

Five samples of dust were collected by the representative of the company from the polyethylene process areas in the plant and provided to us directly (Table 20):

- Two of these were pure polyethylene samples (Qchem_PE_Rx Fluff and Qchem_PE_TR571 Fluff) of different grades taken from the storage facility.
- The other three samples were taken from the process units:
 - The feeder unit (QChem_PE_Fdr);
 - The blender unit (QChem_PE_Bldr);
 - The charging station unit (Qchem_PE_CS).

The company also provided a sample of additive (Qchem_SN1010FF) which is used in the process by the company to provide the final polyethylene product with given mechanical properties.

The last sample collected was a mixture of three components (Qchem_PE_Mixture):

- TR571 Fluff polyethylene final product;
- Songnox6280 additive;

- Songnox1010 additive.

Table 20. Location and identification of the dust samples from the polyethylene plant

ORIGIN	SAMPLE	DESCRIPTION	DUST SAMPLE ID
Storage	Polyethylene	Rx Fluff polyethylene final product	QChem_PE_Rx Fluff
Storage	Polyethylene	TR571 Fluff polyethylene final product	QChem_PE_TR571 Fluff
Feeder	Polyethylene	Polyethylene dust from feeder	QChem_PE_Fdr
Blender	Polyethylene	Polyethylene dust from blender	QChem_PE_Bldr
Charging station	Polyethylene	Polyethylene dust from charging station	QChem_PE_CS
NA	Additive	Additive used in making final polyethylene pellets	QChem_SN1010FF
NA	Mixture of polyethylene and additive	Mixture of TR571 polyethylene and Songnox6280, Songnox1010 additives	QChem_PE_Mixture

5.2.1.2 Characterization of Samples

A comprehensive summary of the particle size of these samples is given in Table 21. Most of the polyethylene dust samples exhibited a wide range of dust particle size, reaching up to more than 2000 μm . The two largest samples in this subset were those of the final products of QChem_PE_RxFluff and QChem_PE_TR571Fluff. These samples had a very broad particle size range of 10 to 2000 μm . Their d_{50} was 692 μm and 825 μm respectively, and these particle sizes lie outside the particle size of 420 μm at which the NFPA defines particles as dusts. While QChem_PE_RxFluff had a spherical shape, QChem_PE_TR571Fluff had an irregular shape. Irregular shapes present a greater

hazard for dust explosions than spherical shapes, because in the case of irregular shapes there is a higher surface area for the combustion reaction to take place.

The polyethylene dust sample from the feeder (QChem_PE_Fdr) had a very broad particle size range, but still contained a good percentage of some very fine particles.

While the d10 was very fine at 26 μm , the d50 was 461 μm , which is still above the NFPA standard of 425 μm . When looking at the particle size distribution in **Error!**

Reference source not found., QChem_PE_Fdr has two peaks. The second peak on the left seems to indicate that QChem_PE_Fdr was contaminated with a small quantity of sample having a mode diameter around 30 μm . This may affect the values of mean, mode, d10, d25, d50, d75, and d90 for this sample.

The two polyethylene samples from the charging station and the blender (QChem_PE_CS and QChem_PE_Bldr) had very fine particle sizes and this can be seen by their d50 of 11 μm and 164 μm respectively.

The additive sample (QChem_SN1010FF) contained particle sizes which were larger than 678 μm (median value), but also had some very fine particles of about 50 μm (d10) which indicated a very wide range of dust particle sizes. The sample which contained a mixture of two additives and polyethylene (QChem_PE_Mixture) had very fine particle size ranges with a d50 of just 49 μm . It can be noted in **Error! Reference source not found.** that this sample has three different peaks, confirming the mixture of one polyethylene dust and two additives.

Table 21. Summary of particle sizes for polyethylene and additive samples

PARTICLE SIZE DISTRIBUTION	Sample	Particle Size Range (μm)	D10 (μm)	D50 (μm)	D90 (μm)	Relative span	MEAN (μm)	MODE (μm)	Particle Shape
QChem_PE_RxFluff	Polyethylene	10- <2000	229.4	692.3	1336	1.59	743.0	751.1	Spherical
QChem_PE_TR571Fluff	Polyethylene	10- <2000	149.4	825.3	1671	1.84	870.1	1443	Irregular
QChem_PE_Fdr	Polyethylene	6- 1500	26.26	461.3	869.9	1.82	448.9	567.7	Spherical
QChem_PE_Bldr	Polyethylene	4- <2000	52.33	164.7	844.6	4.81	314.8	153.8	Spherical
QChem_PE_CS	Polyethylene	1-200	3.172	11.46	36.15	2.87	17.83	11.29	Irregular
QChem_SN1010FF	Additive	1- <2000	50.84	678.7	1575	2.24	748.9	1197	-
QChem_PE_Mixture	Mixture of polyethylene and additive	1- <2000	9.522	49.67	399.4	7.84	130.1	34.58	Irregular

The samples received were tested for moisture content using methodology similar to sulfur dust, and the polyethylene dust was found to have negligible amount of moisture, and hence were assumed dry.

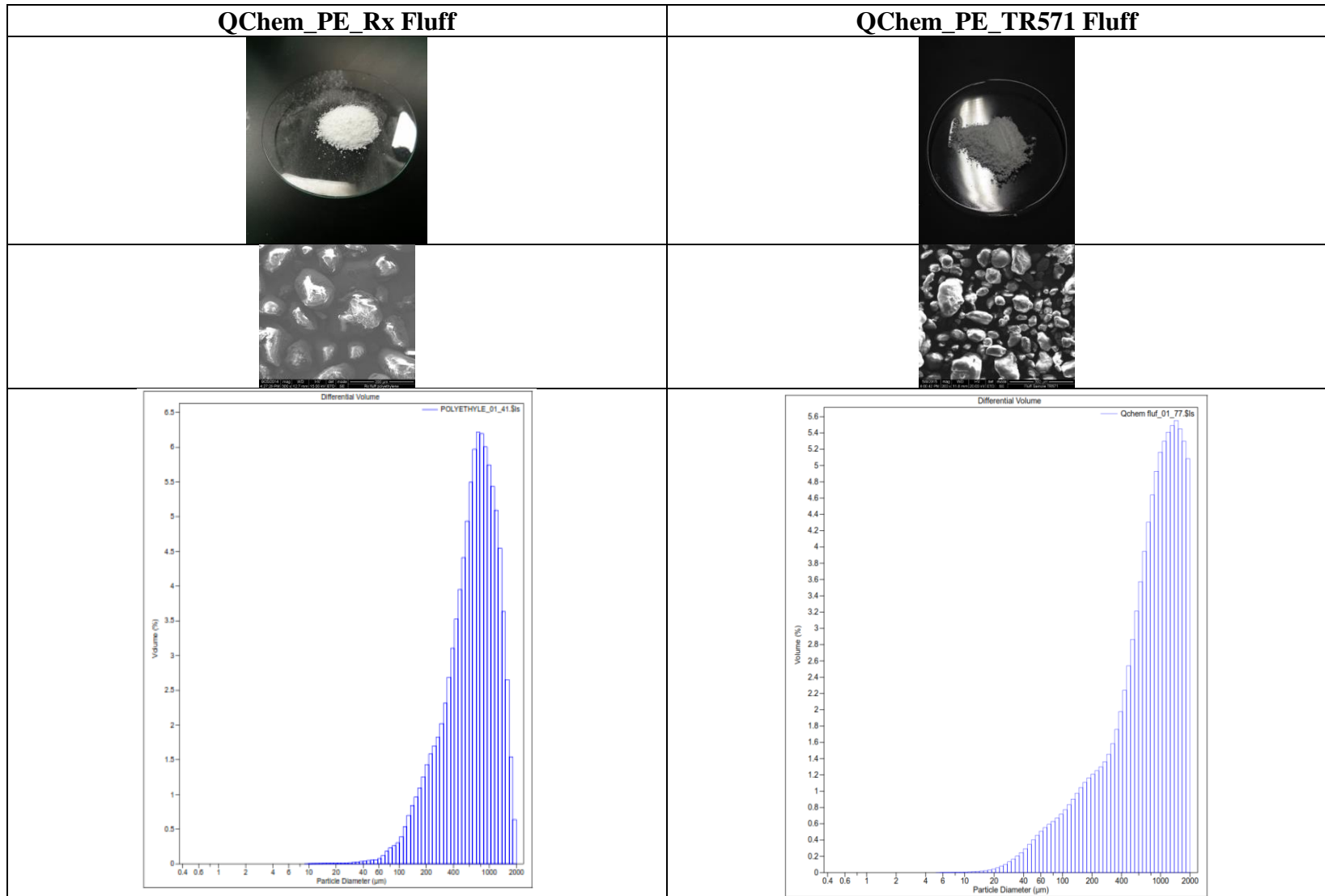


Figure 46. Particle size characterization of polyethylene dust collected on-site

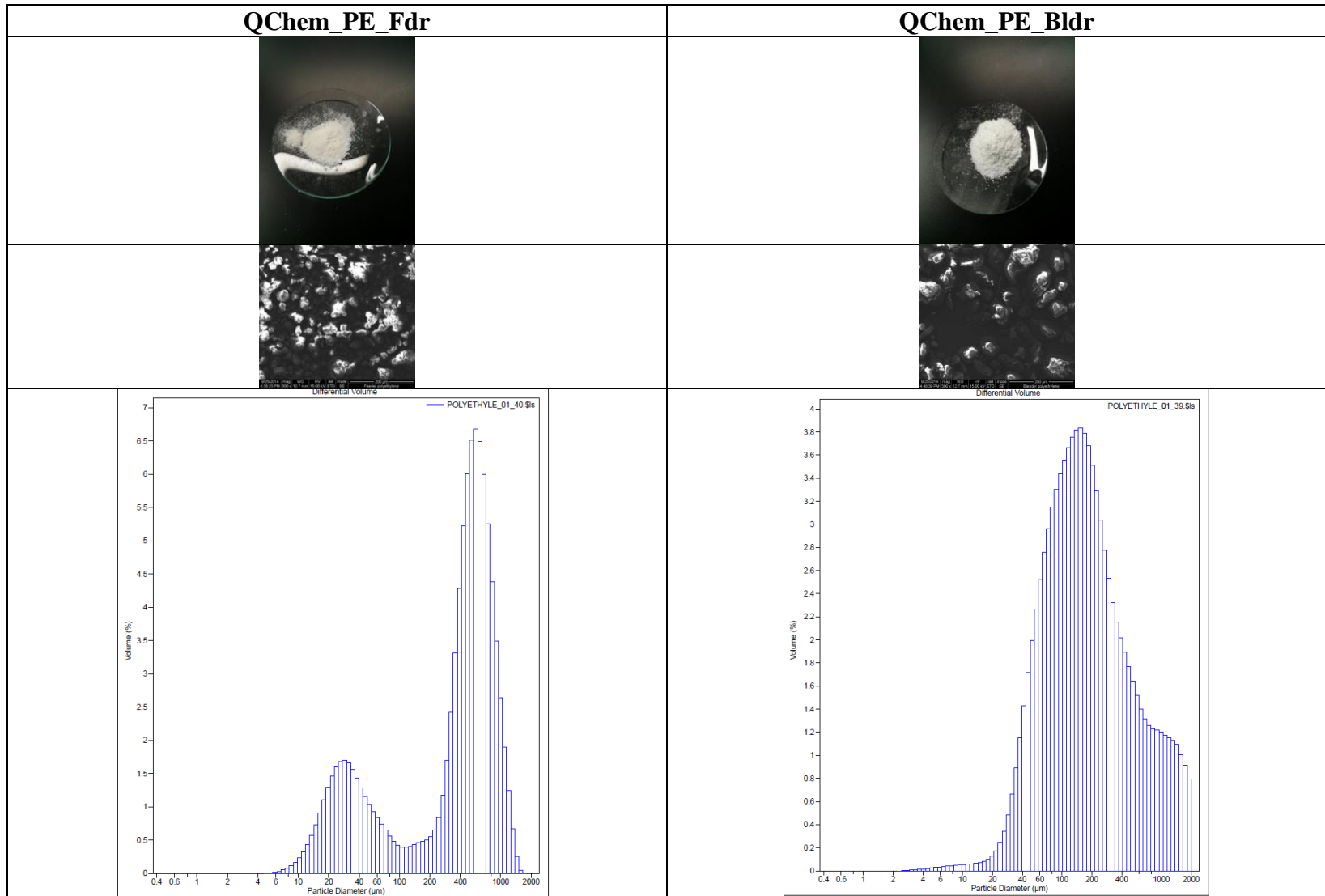


Figure 46. continued.

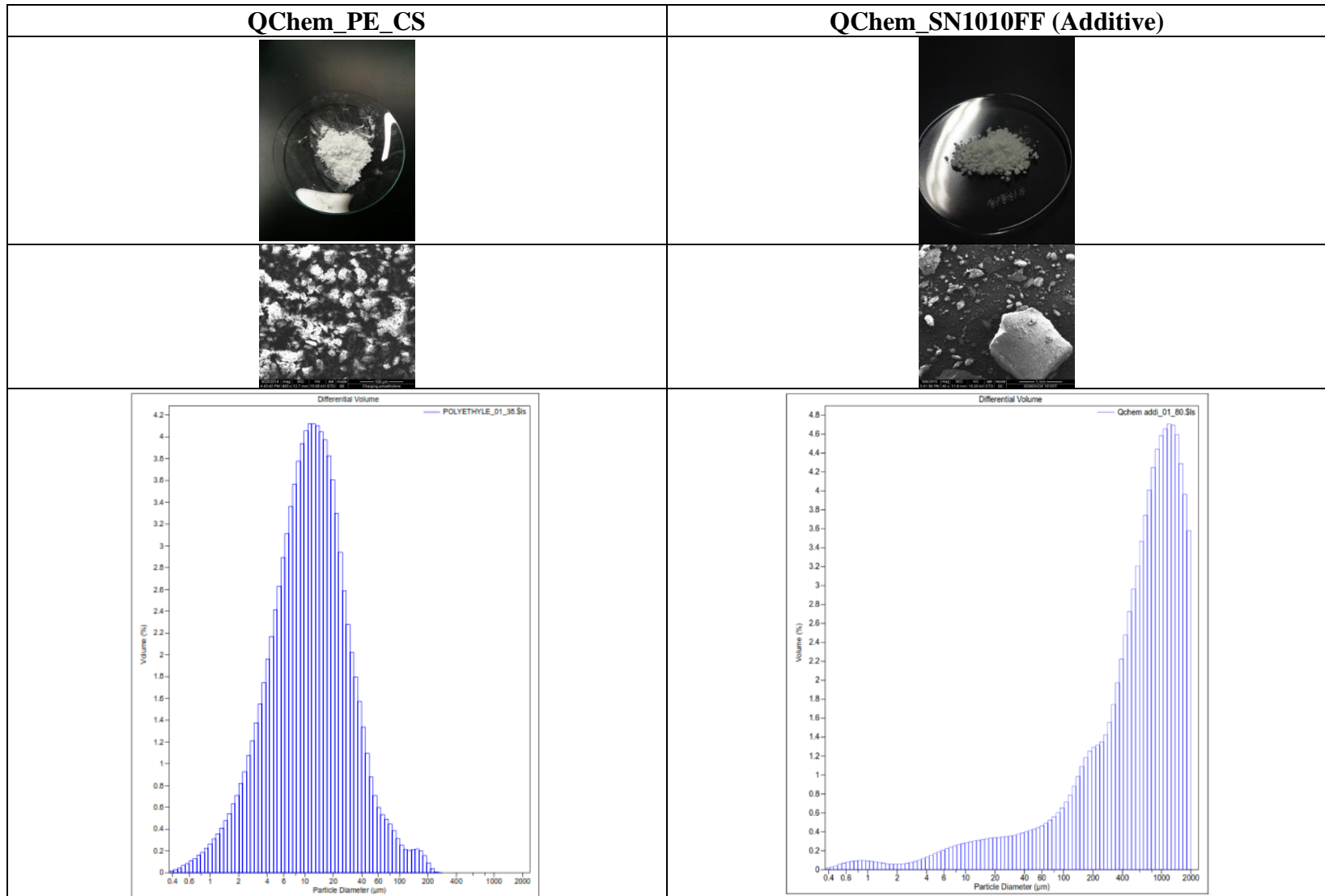


Figure 46. continued.

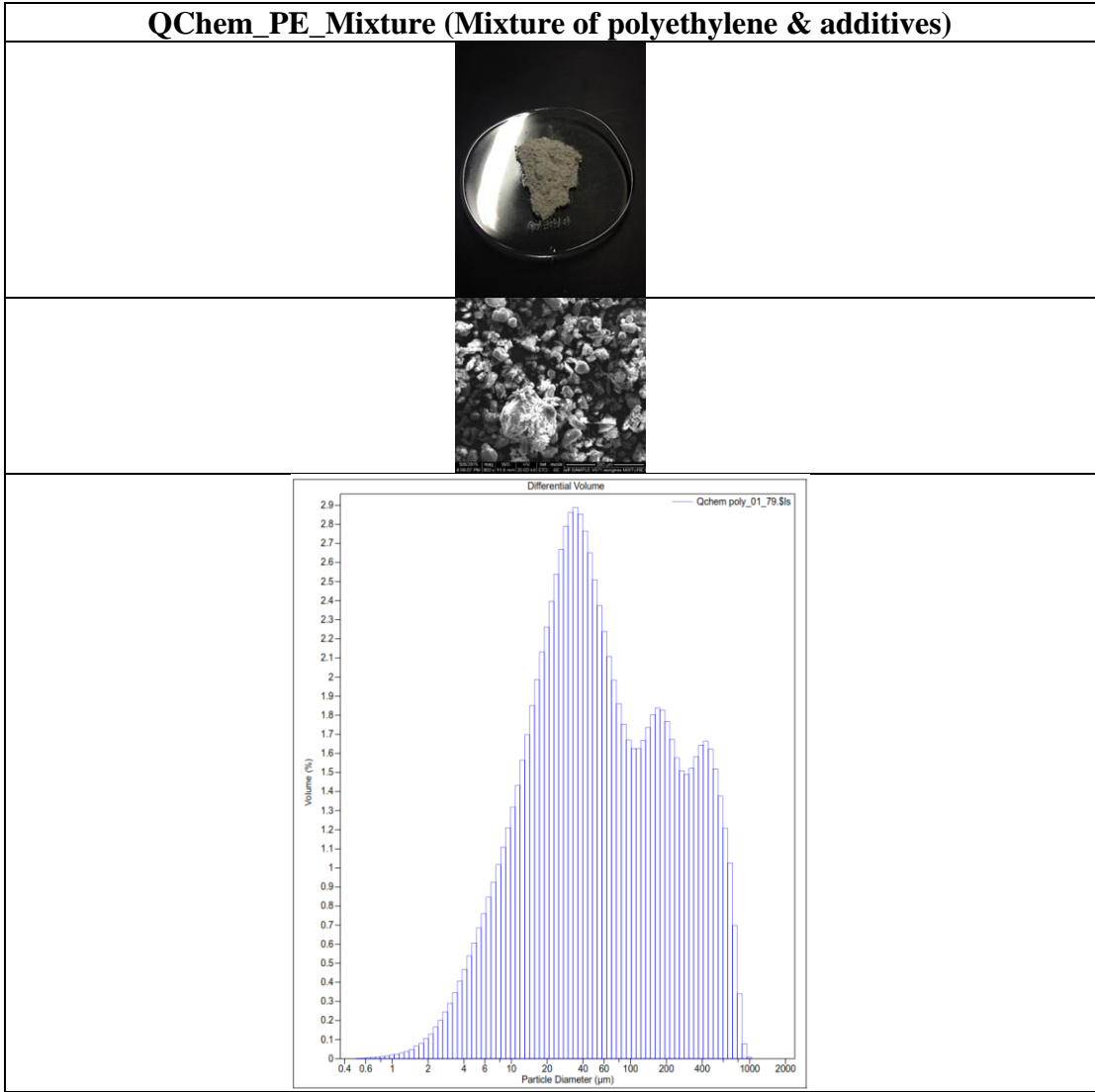


Figure 46. continued

5.2.2 Phase II: Experimental determination of the minimum explosible concentration (MEC) for polyethylene dust collected on-site

5.2.2.1 MEC of on-site samples

Table 22 and Figure 47 gives a summary of all the results of the explosibility tests for all on-site polyethylene samples and additives tested with the modified Hartmann tube. All the polyethylene samples either recorded a fire or explosion in the Hartmann tube.

Table 22. Summary of explosibility results for on-site polyethylene dust and additive samples

SAMPLE	Sample type	PSD MEAN (µm)	PSD MODE (µm)	MEC FIRE (g/m ³)	LEVEL 1 explosion (g/m ³)	LEVEL 2 explosion (g/m ³)	Comparable MEC from literature (g/m ³)
QChem_PE_Rx Fluff	Polyethylene	743.0	751.1	325 (-31) ^h	No Explosion	No Explosion	-
QChem_PE_TR571 Fluff	Polyethylene	870.1	1443	184.1 (-70)	545.8 (-128)	-	-
Qchem_PE_Fdr	Polyethylene	448.9	567.7	60.83 (-24)	69.16 (-8)	81.66 (-6)	-
Qchem_PE_Bldr	Polyethylene	314.8	153.8	46.66 (-23)	141.66 (-16)	-	60(Mittal et al.,1996) 65(Hertzberg et al, 1982)
QChem_PE_CS	Polyethylene	17.83	11.46	33.33 E-5	-	41.66 (-7)	50(Hertzberg et al, 1982)
QChem_SN1010FF	Additive	748.9	1197	No Fire	No Fire	No Fire	-
QChem_PE_Mixture	Mixture of polyethylene and additive	130.1	34.58	-	78.3 (-50)	145.0 (-46)	-

^h The numbers in the brackets indicate the possible error in measuring these values using the modified Hartmann tube.

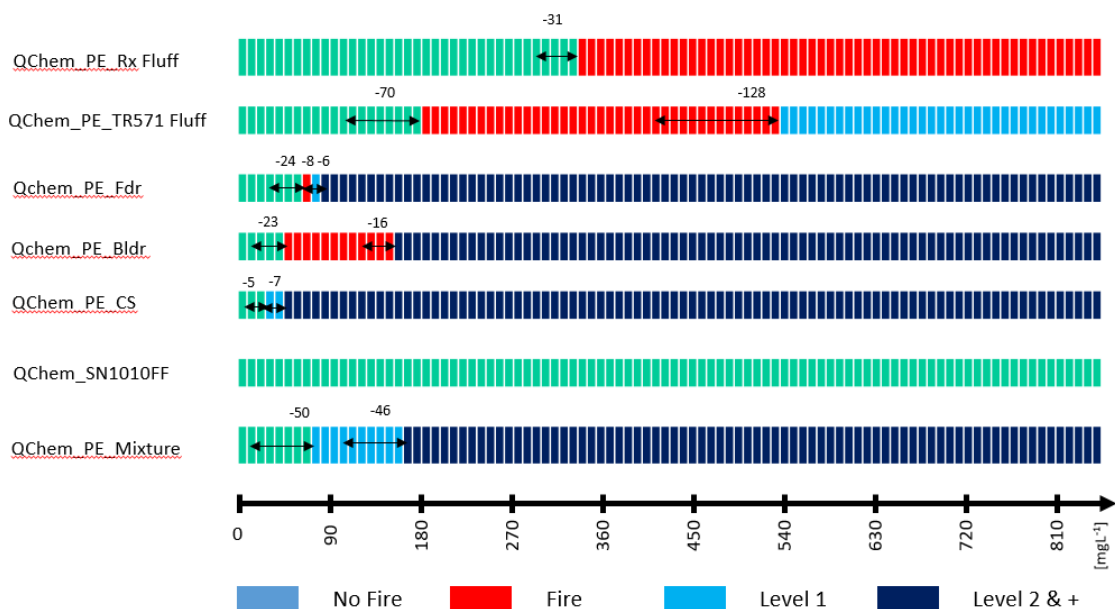


Figure 47. Explosibility results for on-site polyethylene dust samples

For one of the final products (QChem_PE_Rx Fluff), it was recorded that while this sample ignited and propagated a fire at 325 g/m^3 , there were no Level 1 or Level 2 explosions. On the other hand, the other final product (QChem_PE_TR571 Fluff) propagated fire at 184.1 g/m^3 , and the sample had a Level 1 explosion at the concentration of 545.8 g/m^3 . The dust concentration at which this sample exhibited fire was very wide. There is no literature data for these samples which have a particle size median value of $692 \text{ }\mu\text{m}$ and $825 \text{ }\mu\text{m}$ respectively. It can be seen in **Error! Reference source not found.** that the QChem_PE_TR571 Fluff sample has a higher concentration of finer particle sizes compared to QChem_PE_Rx Fluff. This might explain why the QChem_PE_TR571 had a Level 1 explosion, whereas the QChem_PE_Rx Fluff did not.

These explosibility results generate a very interesting conclusion. Both of these samples have a mean and d50, where the particle size is bigger than the NFPA standard particle definition of 420 μm , but their d10 is 229 μm and 149 μm respectively. Thus, it might be that the explosive behavior of these samples is being driven by the lower sized dust particles in the sample. Thus, in an industrial setting people might classify this dust sample to be not explosible based on the mean and d50, but a sample with such a large particle distribution could still pose a dust explosion hazard based on the presence of the finer particles in the dust sample.

When the dust sample from the feeder (QChem_PE_Fdr) was tested, it was shown that while this sample propagated a fire at 60.83 g/m^3 , the sample had a Level 1 explosion at the concentration of 69.16 g/m^3 , and had a Level 2 explosion at 81.66 g/m^3 . All of these dust concentration values are very close to each other, and as such, it is extremely difficult to differentiate at what concentration the sample propagated a fire and at what concentration the Level 1 explosion occurred. There is no literature data for this particle size median value of 461 μm , but the dust exploding at such low concentrations for a comparatively medium sized dust particle size range does not agree with the other results captured using the sieved samples of polyethylene. If the particle size graph is seen in **Error! Reference source not found.**, it can be seen that there are two peaks in regards to the particle size. It might be that the contamination of the dust samples at the peak of 30 μm is responsible for giving such extremely sensitive MEC values. The results above clearly show that the dusts generated at the feeder are very

prone to dust explosion and we can qualitatively say that the explosion may be very severe.

For the sample taken from the blender (QChem_PE_Bldr), the tests showed that while this sample propagated a fire at 46.66 g/m^3 , the sample had a Level 1 explosion at the concentration of 141.66 g/m^3 . In the literature, we find MEC values close to this dust particle size. Mittal et al. give an MEC value of 60 g/m^3 for a dust particle size range of $150 - 180 \mu\text{m}$ ³⁵. Hertzberg et al. also give an MEC value of 65 g/m^3 for a dust particle size range of $35 - 150 \mu\text{m}$ ³³. Even though the MEC values are comparable, the dust particle size range is not comparable as Hertzberg et al. (1982), Mittal et al. (1996), and this research, all use different ways to characterize the particle size. Once again, the results above clearly show that the dusts generated at the blender are prone to dust explosion and we can qualitatively say that the explosion may be less severe than dust generated at the feeder (Figure 48).

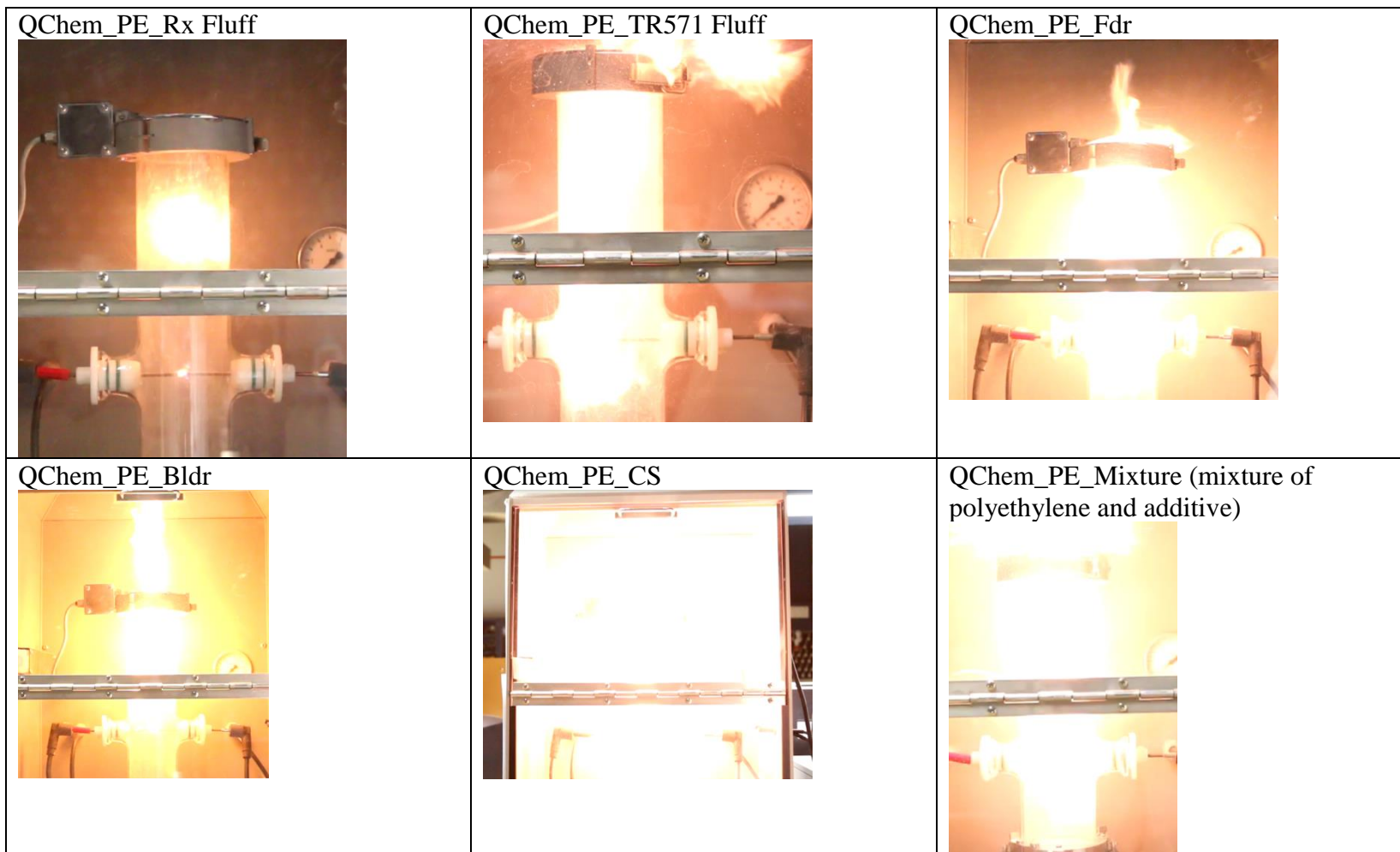


Figure 48. Pictures of the fire / explosion of polyethylene and additives dust (collected on-site) in the modified Hartmann tube

The tests with the sample from the charging station (QChem_PE_CS) confirmed that the particles were so fine that the sample directly had a Level 2 explosion at a very low concentration of 41.66 g/m^3 . The visual capture of this dust explosion shown in Figure 48 lend doubt to whether this sample is purely polyethylene sample. The extreme sensitivity of the MEC to the particle size and the nature of the explosion seem to suggest that this is a polyethylene additive, rather than a pure polyethylene sample. Hertzberg et al. gives an MEC value of 50 g/m^3 for a dust particle size range (polyethylene) of 8-60 μm (Hertzberg et al., 1982). As before, even though the dust particle size range sample is not comparable, as Hertzberg et al. uses the surface weighted area mean diameter to characterize the dust particle size, our research used the mean volume diameter to characterize the dust particles. The results above clearly show that the dusts generated at the charging station are very prone to dust explosion and we can qualitatively say that the explosion would be extremely severe.

The additive sample of QChem_SN1010FF contained quite large dust particles. This sample purely contained of additives used in the process of making polyethylene and did not contain any polyethylene. This additive sample did not explode and with its large particle size, this was an expected result. It is important to note that this sample was given to us in its final form, and as such, this sample does not represent the particle size characterization of the additive being used in the process. Therefore, no conclusions can be made regarding the explosibility of this additive for a particle size range that would contain just finer particle sizes.

The QChem_PE_Mixture sample contained a mixture of both pure polyethylene and two other additives used in the plant. The dust sample had a Level 1 explosion at 78.3 g/m^3 and a Level 2 explosion at 145 g/m^3 . This sample contains the polyethylene dust sample (QChem_PE_TR571 Fluff) and the MEC value for that sample was found to be 184 g/m^3 . The MEC value of this mixture is much lower at 78 g/m^3 , and this can be explained by both the addition of additives in the sample and the presence of much finer particle sizes in the sample. Since this is not a pure polyethylene sample, there is no data from the literature to compare against. It can be concluded though that as the particle size is quite small, it is expected that the dust particle would explode at lower dust concentrations.

5.2.2.2 Conclusions

As a general conclusion, the on-site samples with the lower particle sizes exploded at a lower MEC value than the larger particle size samples. In the literature, the MEC of polyethylene dusts ranged from 10 to 500 g/m^3 . The experimental results with the on-site samples gave the value of the MEC from 46.66 to 325 g/m^3 which lies within the range found in the literature. The key drawback regarding this data was the same as that of on-site sample regarding sulfur dust. As the particle size range of the samples was so wide, it was very difficult to accurately correlate the effect of particle size on MEC values.

It is important to note that the experimental results are for HDPE dust, and these results are being compared to results in the literature which mostly used LDPE dust.

While for most of the samples there was no data present in the literature to compare to, for the select few samples where the particle size range could be compared, the MEC data of HDPE dust could not be compared to the MEC data of the LDPE dust because of the difference in characterization of samples.

In regards to the quantification of error for polyethylene dust, on an average, the error for the polyethylene dust samples range between 20 to 30 g/m³.

Similar to the tests conducted with sulfur dust, the tests with polyethylene dust provide a qualitative indication of the severity of an explosion to the industry. This data gives a good indication of which areas of the plant face a higher risk from potential dust explosion, and the MEC values using a modified Hartmann tube still provide a close enough value to address the risks of these potential dust explosions.

5.2.3 Phase III: Effect of particle size distribution on the MEC values of polyethylene dust

The sample QChem_PE_TR571 Fluff was chosen to be sieved mechanically. This sample was chosen for two reasons; one is that it was a final product, thus the purity of the sample could be guaranteed; and the second was that there was a huge quantity of this sample available to grind and sieve into various cuts. The samples were sieved into five different particle size ranges. This research was only involved in sieving the polyethylene dust samples and the modified Hartmann tube results of these samples have been carried out by other members at the Qatar branch of the Mary Kay O' Connor Process Safety Center (Dr Walid Khalfaoui, Atif Ashraf, and Jack Altwal). Their

approval was taken in order to compare the results of this research to the data they measured for sieved polyethylene samples. Table 23 and Figure 49 give an overview of these results generated for the sieved polyethylene samples.

Table 23. Summary of explosibility results for sieved polyethylene samples

PSD RANGE (µm)	PARTICLE DIAMETER (µm)	MEC FIRE (g/m ³)	LEVEL 1 explosion (g/m ³)	LEVEL 2 explosion (g/m ³)	Comparable MEC from literature (g/m ³)
0-53	53	18.04	27.36	84.52	50 (Hertzberg et al.,1982)
53-63	63	16.67	NA	NA	10 (Amyotte et al,2012) 15 (HSE, 2003)
125-150	150	30.83	NA	NA	50 (Mittal et al.,1996)
150-180	180	50	NA	NA	60 (Mittal et al.,1996)
355-425	425	No Fire	No Explosion	No Explosion	-

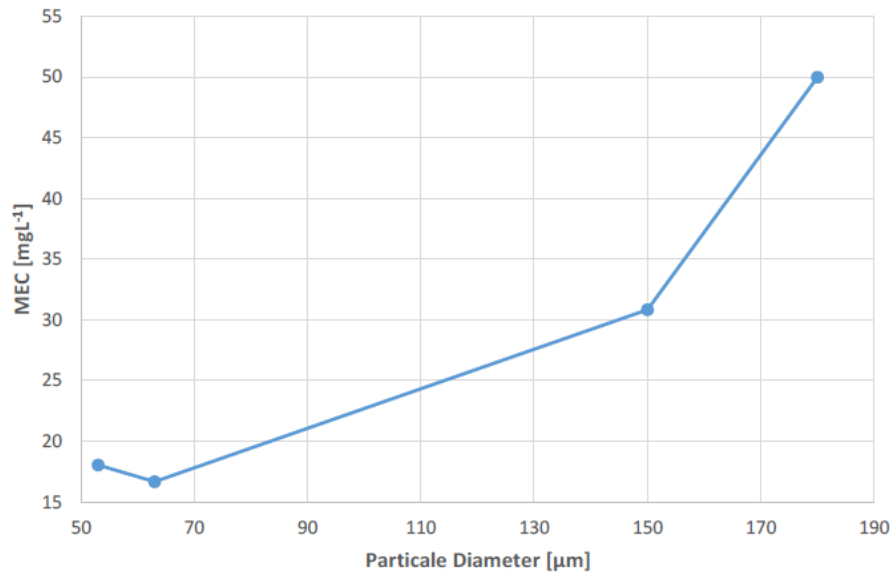


Figure 49. Graphical summary of explosibility results of sieved polyethylene samples

For the dust particle size with range of 0-53 µm, the MEC value was found to be 18.04 g/m³. As this is a very fine particle size range, the MEC of this dust sample is also very low. However, Hertzberg et al. gave an MEC value of 50 g/m³ for a dust particle size range of 8 to 60 µm³³.

The sample which contained the dust particles with range of 53-63 µm, the MEC value was found to be 16.67 g/m³. This result seems to agree with the MEC values found in two studies in the literature. Amyotte et al. found an MEC value of 10 g/m³ for a median volume diameter size of 49 µm³⁸, whereas the health and safety executive states an MEC value of 15 g/m³ for a median dust particle size of 62 µm³⁶.

In regards to the sample which had a range of 125-150 µm, the MEC was found to be 31 g/m³, and this value is comparable to the value found by Mittal et al., where for

the same particle size range, they recorded an MEC value of 50 g/m³. Similarly for the next sample of 150 to 180 μm, the MEC was found to be 50 g/m³ and for the same range, Mittal et al. found an MEC value of 60 g/m³³⁵.

The largest sample of 355-425 μm did not explode and while this is expected because of its large particle size, there is no data in the literature to compare our results.

The conclusion from the figure and table is that the MEC of polyethylene dust is dependent on the particles size. It can be seen that at lower dust particle sizes, the effect of particle size on MEC is not as pronounced, when comparing the very same effect at larger dust particles sizes. This finding is in agreement to the conclusions generated by experiments conducted by Hertzberg et al. (1982). For larger dust particle sizes, the MEC increases with increasing particle size. Mittal et al. (1996) had measured the effect of dust particle size on MEC using a 20-liter sphere and Hertzberg et al. (1982) had measured the same correlation using an 8-liter chamber. Comparing Figure 12 and Figure 13 to the graph generated in Figure 49, it can be seen that all the graphs follow the same trend of increasing MEC values with increasing dust concentrations at larger dust particle sizes. Thus, the applicability of a Hartmann tube can be validated, when its results and trends agree with other data found in the literature^{35,33}.

5.3 Discussion on manual sieving vs. mechanical sieving

As has been discussed above, the dusts were sieved into finer particle sizes to have a better understanding of the effect of particle size on MEC. The first sieving method was manual and then later the method used was of mechanical sieving. It was

found that inconsistencies occurred in the results because of the manual sieving process. The manual sieving would be done till there was enough mass for a sample dust particle size range to conduct tests. For example, the grinded dust would be sieved for a few hours between the 300 μm and 250 μm sieves. If about 1000 grams of dust was found on the 250 μm sieve, this was then deemed enough and experiments were conducted on it, and this dust sample was labelled to have a range of 300 to 250 μm . It is very likely though that this dust sample would have many particle sizes which are lower than 250 μm , and through the process of manual sieving, all of them would not have sieved down to the lower sized sieve. This would then undoubtedly affect the MEC results as the lower particle sizes explode at lower concentrations. Even though the general results using manually sieved samples agree with the MEC and dust particle size correlation, the individual MEC results of each particle size range might not be accurate and thus have not been compared to the values in the literature. When dusts were mechanically sieved, the mass of each sieve with the dust samples was tested after every hour and only if the mass of each sieve with the dust samples remained unchanged, then the mechanical sieving process was marked to be complete. Even with the high oscillation of the mechanical sieves, it would take around 3 hours to get about 300 grams of dust. It can be speculated that to get the same amount of dust through manual sieving would take days, which is not practical at all. Hence, this was a lesson learnt in the research that to get accurate results, manual sieving is not the right methodology to sieve dust into finer particle sizes.

5.3.1 *Experimental results with manually sieved samples of polyethylene dust*

The manual sieving was done for five ranges of dust particle size using calibrated sieves. The five ranges of dust particle size were 300-425 μm , 250-300 μm , 150-250 μm , 125-150 μm , 75-125 μm , and 20-75 μm . Table 24 states the overall explosibility results of all these different particle size ranges. From these results, it can be concluded that there is a correlation between the particle size and the MEC for polyethylene dust, and that for a lower particle size, a lower dust concentration is needed for the dust to be explosible. However, some data inconsistencies were also found out using the manually sieved samples. As an example for the dust particle size range of 150 to 125 μm , a Level 1 explosion was recorded for 105 g/m^3 , whereas for a smaller sample of particle size range of 125 to 75 μm , a Level 1 explosion was recorded at 247 g/m^3 . This does not agree with the general principle that a lower dust particle size will have a lower MEC. This also does not agree with the conclusions found specifically for polyethylene dust in regards to the effect of particle size on MEC, as found by both Mittal et al. and Hertzberg in their papers^{35,33}. As such, even though the overall results agree with MEC and dust particle size correlation for polyethylene, the individual MEC results of each particle size range might not be accurate, and thus have not been compared to the values in the literature.

Table 24. Explosibility results of manually sieved polyethylene dusts

PSD Range[μm]	MEC Fire [g/m^3]	Level 1 explosion [g/m^3]	Level 2 explosion [g/m^3]
300-425	NF	NE	NE
250-300	235	353	NE
150-250	116	412	NE
125-150	45	105	NE
75-125	138	247	682
20-75	40	40	

5.3.2 *Experimental results with manually sieved samples of sulfur dust*

The sample chosen to be manually sieved was the prilled sulfur sample from its storage silos. This involved the same time-consuming process of grinding the dust and sieving them into five different particle size ranges of 500-600 μm , 425-500 μm , 355-425 μm , 300-355 μm , and 250-300 μm . After conducting the full range of tests in the Hartmann tube, it was found out that none of the dust samples exploded at concentration of up to 1000 g/m^3 . This was a surprising result, even though there was no data in the literature to compare our results. These results led the research team to question whether prilled sulfur and granulated dust have different explosible properties for the same particle size. Upon checking the finer sizes of both the prilled size and granulated sulfur under the scanning electron microscope, it was found that the particle shape was only different at large particle sizes, and for fine dust, both of them looked similar and had similar crystallographic properties. The team decided to confirm the validity of the manual sieving methodology and it was found that the same errors were prevalent in the range of particle sizes collected as was encountered during the manual sieving of the

polyethylene dust. Thus, it was decided to mechanically sieve the sulfur dust sample to find the effect of the dust particle size on the MEC for sulfur dust. The tests using the mechanically sieved samples confirmed the data of the tests using the manually sieved samples, but since the manual methodology had errors in it, it was decided not to report these results. What can be concluded with a high certainty from these results though is that for the range of 250-600 μm , the sulfur dust does not meet the minimum MEC requirements for the dust to explode.

5.4 Comparison of results using a modified Hartmann tube vs 20-liter sphere for a sample of sulfur dust

The 20-liter sphere is the standard equipment to measure many dust explosibility properties including MEC, P_{max} , K_{St} , and LOC (see section 2.5.3.1). Through the thesis work, we used the modified Hartmann tube to evaluate the MEC of sulfur and polyethylene samples. Sections 5.1.2, 5.1.3, and 5.2.2 presented results of MEC with uncertainties associated with the value (Section 4.3).

At the time of the thesis, one of my tasks was to progress the installation of the 20-liter sphere in the laboratory. A major significant part of my time was dedicated to this task. The sphere was successfully installed only toward the end of my research and I unfortunately was not able to experiment with the 20-liter sphere myself.

One series of 4 MEC determination tests using the 20-liter sphere for one sulfur sample, namely QG_Sulfur_WC (see Table 16, Table 17, and **Error! Reference source not found.** for the description of the sample) is shown in this thesis in order to be

compared with our modified Hartmann tube. These 4 tests were performed by other members of the Qatar branch of the Mary Kay O' Connor Process Safety Center (Dr Walid Khalfaoui, Atif Ashraf, and Jack Altwal). Their approval was taken prior to using these data in this manuscript.

The ASTM E1515 *Standard Test Method for Minimum Explosible Concentration of Combustible Dusts* was used to determine the MEC (Table 25).

Figure 50 shows the results of these tests. An MEC of **60 g/m³** was measured for the QG_Sulfur_WC sample with the 20-liter sphere. The value of MEC for the same sample we measured with the modified Hartmann tube was between 50 and **75 g/m³**. For this sample, the modified Hartmann tube released to provide a good indication of the MEC.

A full study would be necessary for different types of dust to conclude the quality of MEC determination using the modified Hartmann tube, at least as a screening equipment before confirmation of the results in a 20-liter sphere, but the results we obtained with sulfur is quite promising.

Table 25. Determination of MEC in 20-liter sphere (ASTM E1515)

Vacuum pressure	0.4 bar
Initial pressure	1 bar
Dispersion Pressure	20 bar
Time Delay	60 ms
Ignition Energy	2.5 kJ
Explosion Criteria	Pm ≥1 barg
Definition of MEC	Lowest concentration that satisfies Explosion Criteria

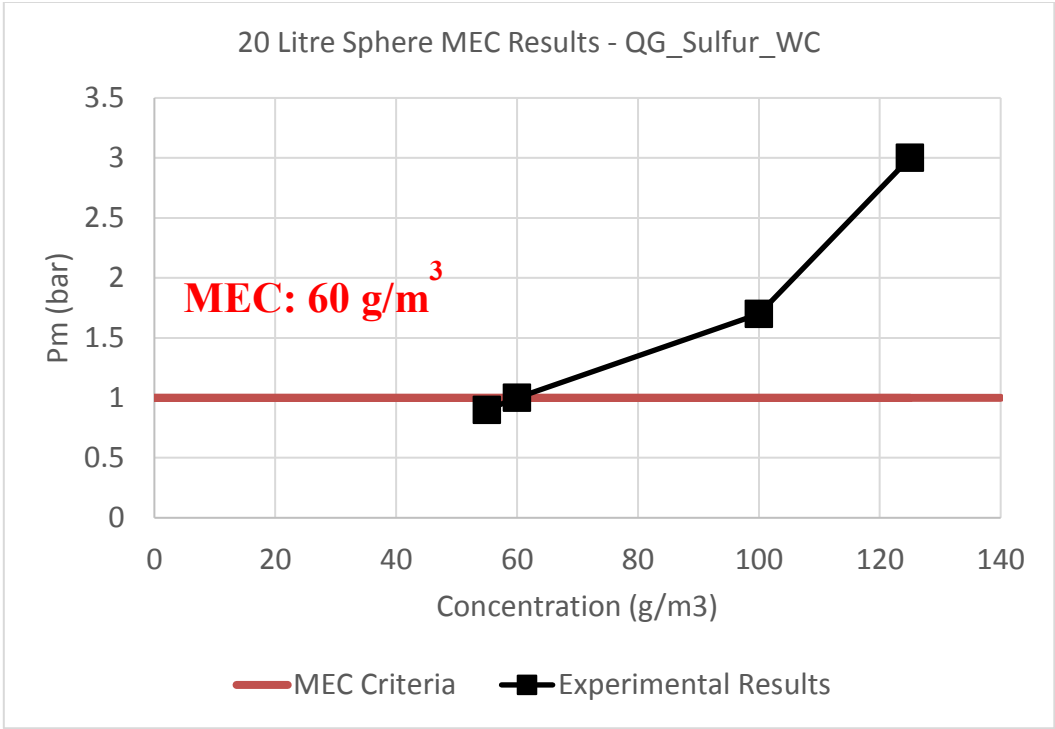


Figure 50. MEC measurement for QG_Sulfur_WC using a 20-liter sphere

6 CONCLUSIONS

The objective of the thesis was to perform the experimental study of the minimum explosion concentration (MEC) of polyethylene and sulfur dust in the context of the Qatar industry.

Typical dust samples collected on-site at polyethylene and sulfur production facilities in Qatar were collected and characterized to have the associated composition, particle size distribution and particle shape.

The MEC of these samples were then measured using a Modified Hartmann Tube. While the Modified Hartmann Tube is not the standard piece of equipment for the determination of MEC. It was used as a screening tool for MEC determination. The MEC measured by the modified Hartmann tube was compared to the data obtained from the 20-liter sphere for one sulfur sample and the values were comparable. Finally, the influence of the particle size distribution of the polyethylene and sulfur dust samples on the MEC was studied. It was showed that for both sulfur and polyethylene, at smaller particle sizes ($<100\ \mu\text{m}$), the particle size does not have a strong influence on the MEC values. For larger particle size MEC will clearly increase with the particle size.

6.1 Sulfur dust

The results clearly showed that dust collected on-site at the vibrating screen can be involved in a dust explosion, but we cannot say that this is entirely due to sulfur since we found that this sample was contaminated with carbon. The dusts generated at the wall

on chute and conveyor to structure are very prone to dust explosion, and we can qualitatively say that the explosion may be more severe than dust generated at the vibrating screen.

Our sulfur dust sample was grounded and mechanically sieved into 6 particle size ranges (0-53 μm , 63-70 μm , 90-106 μm , 125-150 μm , 150-180 μm , and 355-425 μm). The MEC for these cuts were analyzed and we were able to plot the MEC as a function of particle size, which constitutes the major finding of the thesis.

The literature provides very few data for MEC of sulfur dust and as such the data generated by this research contribute to fill this gap.

6.2 Polyethylene dust

The results with the collected polyethylene dusts clearly show that the dusts generated at the feeder, the blender and charging station are prone to dust explosion and we can qualitatively compare the relative severity of these explosions.

Polyethylene sample was mechanically sieved into 6 particle size ranges (0-53 μm , 53-63 μm , 125-150 μm , 150 - 180 μm) and the MEC as a function of particle size was determined. The 355-425 μm sample did not explode,

The literature provided quite a sufficient amount of data in regards to the MEC of polyethylene dust. In general, these data are not comparable to each other, as most times the complete dust characterization details are not mentioned and nor is the type of polyethylene dust used mentioned. There is no data present in the literature for HDPE dust except for one paper which lists just one P_{max} and K_{St} value for this type of dust.

This research generates some new MEC data in regards to HDPE dust using a modified Hartmann tube.

6.3 Future research

This research only measures the effect of particle size on MEC for polyethylene and sulfur dust. Many other factors have an effect on the MEC values such as water content, turbulence, ignition methods, and dispersion methods) and deserve to be investigated thoroughly.

These can be used as a starting point to build on the data generated by this research, especially for sulfur dust where little to no data exists. Since this research attempts to compare the values obtained by a modified Hartmann tube and a 20-liter sphere, other aspects such as particle breakage, uniformity of dispersion in the different equipment could also serve as topics to be explored in future research.

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APPENDIX

Incidents involving polyethylene dusts

West Pharmaceuticals - January 29, 2003

One of the most well-known incidents involving polyethylene dust explosion is that which occurred at the West Pharmaceuticals in Kinston, North Carolina. It occurred on January 29, 2003, killing 6 workers and injuring 36 people. According to the final CSB report, the explosion was caused by the deflagration of fine polyethylene powder, with less than 63 microns in diameter³. The explosion which ended up destroying the whole facility, involved a part of the building which was used to compound rubber. Some of the main products of the facility were rubber syringe plungers and various pharmaceutical devices. One of the processes involved coating of the products and this was done by running rubber strips through a tank. This tank contained a slurry of polyethylene powder in water and this was used to cool the rubber and also act as an anti-tack coating⁵⁷. The next stage required that the fans evaporated the water from the slurry which would only leave the powder coating on the rubber strips. As such, when the slurry was dyed, the airborne polyethylene dust was generated¹⁴. Over time, while the rubber dried, fine polyethylene powder started to accumulate on the suspended ceilings above, reaching up to a thickness of 1 cm. This accumulated dust was the primary source of fuel for the multiple secondary explosions which caused the main damage to the facility. One of the root causes of this incident was also related to poor

housekeeping. While the facility had a very good housekeeping program for the visible production areas which kept these areas extremely clean, the dust accumulated above the suspended ceiling was kept hidden, and most employees were not even aware that dust was accumulating in such a remote part of the plant. Furthermore, the Material Safety Data Sheet (MSDS) for the polyethylene slurry included no dust explosion warning, as it never occurred to the employees that the slurry after drying would give rise to fine airborne combustible dust. Lastly, even for the select employees, who did notice the dust accumulation above ceiling, were neither aware nor trained regarding the hazards of combustible dust³.

The key lesson learned from this incident is that aqueous solution of a combustible dust can dry, and thus present a combustible dust hazard. As mentioned above, the polyethylene slurry was not considered hazardous as it was a liquid/paste. Thus, the manufacturer had not included any dust explosion hazard warning on the MSDS, even though the manufacturer was aware that the slurry would dry to form a powder during normal operations³. In monetary fines, West Pharmaceutical Services was fined \$10,000 for its safety violations⁵⁸.

Incidents that Occurred in China

In their paper, Tan et al. report about the different incidents which have occurred in China involving polyethylene. In different petrochemical industries in China, 13 explosions in a powder silo of Low Density Polyethylene (LDPE) have occurred over an 11-year period. Furthermore, 14 explosions of a powder silo of polypropylene (PP) have

occurred over a 5-year period and 12 explosions of a powder silo of High Density Polyethylene (HDPE) have occurred over a 3-year period⁵⁹. Yan et al. collected the instances of the number of dust explosions in China during the period of 1981 - 2011. In this 15-year period, 9 accidents occurred involving polyethylene dust. The incidents were collected from literature, books, reports, and the internet⁶⁰.

Incidents involving sulfur dusts

While sulfur is a combustible dust, there is not much data that exists on sulfur dust explosion incidents. Some examples have been discussed by Eckhoff in his book, 'Dust explosion in the Process Industries'. An example of a sulfur dust incident is the one that occurred in Germany in the year 2000 during the loading of bulk sulfur. The ignition was probably caused by an electrostatic discharge and this explosion gave rise to a fire which caused significant damage to the plant. Fortunately, no one was injured⁷. Another incident occurred in 1973 at Dyno Industries in Norway. The incident occurred in a batch mixer which contained sulfur and aluminum flakes being mixed. This initial explosion in the mixer ignited a larger dust cloud which gave rise to a secondary explosion. This secondary explosion ended up killing 5 workers and seriously injuring 2 more. Moreover, a significant part of the plant was destroyed. The cause was determined to be an electrostatic discharge, and the preventive measure of nitrogen purging was found to be inadequate to maintain the lower levels of oxygen for an explosion not to occur¹. In recent times, an incident discussed was the series of the sulfur dust explosions and fires which took place over a period of years in the Shanghai Sulfur Factory in

China. Similar to the instance above, electrostatic discharge was found to be the most probable ignition source. It was found that the potential voltage inside the sulfur silo was from between 25 - 60 kV, and as such, during the transportation of sulfur in the plant, this would act as an ignition source⁶¹.