



## Master's thesis

# Investigations on dust explosions of sewage sludge ash and carbon carriers

Thesis created by

### **Chair of Thermal Processing Technology**

**Submitted by:** Katja Hüttenbrenner, BSc 0635253 Supervisor: Dipl.-Ing. Dr.mont. Kerstin Held Dipl.-Ing. Dr.mont. Hannes Kern Univ.Prof. Dipl.-Ing. Dr.techn. Harald Raupenstrauch

Leoben, June 2014

### EIDESSTATTLICHE ERKLÄRUNG

Ich erkläre an Eides statt, dass ich diese Arbeit selbstständig verfasst, andere als die angegebenen Quellen und Hilfsmittel nicht benutzt und mich auch sonst keiner unerlaubten Hilfsmittel bedient habe.

#### AFFIDAVIT

I declare in lieu of oath, that I wrote this thesis and performed the associated research myself, using only literature cited in this volume.

Place/Date

Signature

### Acknowledgment

I am thankful to Kerstin Held and Hannes Kern for the supervision of this thesis, the support and the advice during the work and also during the writing. Thanks for correcting my master's thesis and thanks for your help!

Also, I would like to thank Harald Raupenstrauch for the opportunity to write this thesis at the Chair of Thermal Processing Technology.

Last but not least thanks to my parents and my brother for the support during my studies.

#### Kurzfassung:

Um den vorgeschriebenen Sicherheitsanforderungen zu genügen, muss bei einer bestehenden Anlage der im Prozess verwendete Staub auf das Explosionsverhalten hin untersucht werden. Dazu werden mit Hilfe der MIKE 3- Apparatur und der 20- Liter Kugel die Explosionskennzahlen, wie der maximale Explosionsdruckanstieg, der maximale Explosionsdruck und die Mindestzündenergie, bestimmt. Eine Mischung aus Klärschlammasche und einem Kohlenstoffträger wird bei einem bestehenden Prozess als Einsatzstoff verwendet. Verschiedene Kohlenstoffträger werden bei den Untersuchungen getestet um die sicherste Staubmischung herauszufinden. Des Weiteren wird die Kohlenstoffkonzentration in den Mischungen variiert. Ein Zusammenhang zwischen der Explosionsfähigkeit und der Konzentration von flüchtigen Bestandteilen wird ebenfalls in die Betrachtung miteinbezogen, um dadurch das sicherste Gemisch aus Klärschlammasche und Kohlenstoffträger zu erhalten.

#### Abstract:

To meet the safety requirements in an existing apparatus the used dusts have to be investigated on their explosive properties. Therefore the explosion indices maximum pressure rise, maximum explosion pressure and the minimum ignition energy are determined with the help of the MIKE 3- apparatus and the 20-I- apparatus. The used dust is a mixture of sewage sludge ash and a carbon carrier. To find out the safest alternative for this mixture, various carbon carriers are tested. In addition the ratio of carbon in the mixture alter to find the best mixture in terms of safety. Furthermore an impact of volatilities on explosibility can be seen. This parameter should also be regarded at the search for the best mixture between sewage sludge ash and carbon carrier.

## Contents

Contents	II
Letter symbols	VII
Acronyms	VIII
List of figures	IX
1 Introduction	1
2 Theoretical principles	2
2.1 Dust explosions	5
2.1.1 Mechanism of dust explosions	6
2.2 Legal Basis – Directive 1999/92/EG	6
2.3 Explosion indices	8
2.3.1 Maximum explosion overpressure and maximum rate of pressure rise	8
2.3.1.1 Influential parameters	11
2.3.1.2 Appraisal of the trials and definitions of the pressure curve	14
2.3.2 Minimum ignition energy	15
2.3.2.1 MIKE 3 - apparatus	17
2.3.2.2 Influential parameters	18
3 Test procedure and sample preparation	23
3.1 Preparation of the dust	23
3.2 Test procedure in the 20-I-apparatus	28
3.3 Test procedure in the MIKE 3-apparatus	30
4 Process description	33

4	.1 Prod	cess description for the 20-I-apparatus	.33
	4.1.1	Identification of the process	. 33
	4.1.2	Actual state of the process	34
	4.1.3	Analysis of the process	42
	4.1.3.1	Moments of truth	42
	4.1.3.2	Voice of the customer	42
	4.1.3.3	Interfaces	43
	4.1.3.4	Analysis of the treats	43
	4.1.4	Identification and classification of the potential improvement	44
	4.1.5	Future state of the process	45
	4.1.6	Determination of the process objectives and the used indicators	45
	4.1.7	Definition of the reporting for process performance	. 46
	4.1.8	Realization of the potential improvements	47
4	.2 Prod	cess description of MIE	.48
	4.2.1	Identification of the process	. 48
	4.2.2	Actual state of the process	. 49
	4.2.3	Analysis of the process	. 56
	4.2.3.1	Moments of truth	. 56
	4.2.3.2	Voice of the customer	. 57
	4.2.3.3	Interfaces	. 58
	4.2.3.4	Analysis of treats	58
	4.2.3.5	Identification and classification of the potential improvement	. 59
	4.2.4	Future state of the process	60
	4.2.5	Determination of the process objectives and the used indicators	60
	4.2.6	Definition of the reporting for process performance	. 61
	4.2.6.1	Realization of the potential improvements	. 61
5	Materia	I characteristics	63
5	.1 Coa	۱	63
	5.1.1	Brown coal	. 66
	5.1.1.1	Briquetting of brown coal	. 67
	5.1.1.2 maximum	n rate of pressure rise (dp/dt) <sub>MAX</sub> and K <sub>ST</sub> -value	MAX, 68
	5.1.1.3	Investigations on pure brown coal - minimum ignition energy (MIE)	. 69
	5.1.2	Charcoal	. 73
	5.1.2.1 rate of pro	Investigations on pure charcoal - maximum explosion overpressure $p_{MAX}$ , maxin essure rise (dp/dt) <sub>MAX</sub> and $K_{ST}$ -value	num 75

	5.1.2.2	Investigations on pure charcoal - minimum ignition energy (MIE)	76
	5.1.3 I	Hard coal	77
	5.1.3.1 rate of pre	Investigations on pure hard coal - maximum explosion overpressure $p_{MAX}$ , massure rise (dp/dt) <sub>MAX</sub> and $K_{ST}$ -value	aximum 78
	5.1.3.2	Investigations on pure hard coal - minimum ignition energy (MIE)	80
	5.1.4	Anthracite	81
	5.1.4.1 maximum	Investigations on pure anthracite - maximum explosion overpressure rate of pressure rise (dp/dt) <sub>MAX</sub> and K <sub>ST</sub> -value	эр <sub>мах,</sub> 81
	5.1.4.2	Investigations on pure anthracite - minimum ignition energy (MIE)	83
ł	5.2 Grap	hite	84
	5.2.1	Graphitization	86
	5.2.2 I	Investigations on graphite dust	86
	5.2.2.1 and Ks⊤-va	Maximum explosion overpressure p <sub>MAX</sub> , maximum rate of pressure rise (d	p/dt) <sub>мах</sub> 86
	5.2.2.2	Minimum ignition energy (MIE)	88
6	Investig	ations on dust mixtures	93
6	6.1 Minir	num ignition energy (MIE)	93
	6.1.1 I	Brown Coal	94
	6.1.1.1	Investigation of 40% brown coal and 60% sewage sludge ashes	94
	6.1.1.2	Investigation of 35% brown coal and 65% sewage sludge ashes	95
	6.1.1.3	Investigation of 30% brown coal and 70% sewage sludge ashes	96
	6.1.1.4	Investigation of 25% brown coal and 75% sewage sludge ashes	97
	6.1.1.5	Investigation of 20% brown coal and 80% sewage sludge ashes	98
	6.1.2	Charcoal	99
	6.1.2.1	Investigation of 40% charcoal and 60% sewage sludge ashes	99
	6.1.2.2	Investigation of 35% charcoal and 65% sewage sludge ashes	100
	6.1.2.3	Investigation of 30% charcoal and 70% sewage sludge ashes	101
	6.1.2.4	Investigation of 25% charcoal and 75% sewage sludge ashes	102
	6.1.2.5	Investigation of 20% charcoal and 80% sewage sludge ashes	103
	6.1.3 I	Hard coal	104
	6.1.3.1	Investigation of 40% hard coal and 60% sewage sludge ashes	104
	6.1.3.2	Investigation of 35% hard coal and 65% sewage sludge ashes	105
	6.1.3.3	Investigation of 30% hard coal and 70% sewage sludge ashes	106
	6.1.3.4	Investigation of 25% hard coal and 75% sewage sludge ashes	107
	6.1.3.5	Investigation of 20% hard coal and 80% sewage sludge ashes	108
	6.1.4	Anthracite	109

6.1.4.1	Investigation of 40% anthracite and 60% sewage sludge ashes	109
6.1.4.2	2 Investigation of 25% anthracite and 75% sewage sludge ashes	110
6.1.5	Graphite	111
6.1.5.1	Investigation of 40% graphite and 60% sewage sludge ashes	111
6.1.5.2	2 Investigation of 25% graphite and 75% sewage sludge ashes	112
6.2 Max and K <sub>ST</sub> -valu	kimum explosion overpressure p <sub>MAX</sub> , maximum rate of pressure r	ise (dp/dt) <sub>MAX</sub> 113
6.2.1	Brown Coal	113
6.2.1.1	Investigation of 40% brown coal and 60% sewage sludge ashes	113
6.2.1.2	2 Investigation of 35% brown coal and 65% sewage sludge ashes	115
6.2.1.3	Investigation of 30% brown coal and 70% sewage sludge ashes	116
6.2.1.4	Investigation of 25% brown coal and 75% sewage sludge ashes	117
6.2.1.5	Investigation of 20% brown coal and 80% sewage sludge ashes	119
6.2.2	Charcoal	120
6.2.2.1	Investigation of 40% charcoal and 60% sewage sludge ashes	120
6.2.2.2	Investigation of 35% charcoal and 65% sewage sludge ashes	121
6.2.2.3	Investigation of 30% charcoal and 70% sewage sludge ashes	123
6.2.2.4	Investigation of 25% charcoal and 75% sewage sludge ashes	123
6.2.2.5	Investigation of 20% charcoal and 80% sewage sludge ashes	124
6.2.3	Hard coal	125
6.2.3.1	Investigation of 40% hard coal and 60% sewage sludge ashes	125
6.2.3.2	Investigation of 35% hard coal and 65% sewage sludge ashes	126
6.2.3.3	Investigation of 30% hard coal and 70% sewage sludge ashes	127
6.2.3.4	Investigation of 25% hard coal and 75% sewage sludge ashes	129
6.2.3.5	Investigation of 20% hard coal and 80% sewage sludge ashes	130
6.2.4	Anthracite	132
6.2.4.1	Investigation of 40% anthracite and 60% sewage sludge ashes	132
6.2.5	Graphite	134
6.2.5.1	Investigation of 40% graphite and 60% sewage sludge ashes	134
7 Results	\$	136
7.1 Car	bon Sources	136
7.2 Bro	wn Coal and sewage sludge ash	140
7.2.1	Minimum ignition energy (MIE)	140
7.2.2	Maximum explosion overpressure and maximum rate of pressure rise	140
7.3 Cha	arcoal and sewage sludge ash	143

7	7.3.1	Minimum ignition energy (MIE)	143
7.3.2 Maximum explosion overpressure and n		Maximum explosion overpressure and maximum rate of pressure rise	143
7.4	4 Hai	rd coal and sewage sludge ash	146
7	7.4.1	Minimum ignition energy (MIE)	146
7	7.4.2	Maximum explosion overpressure and maximum rate of pressure rise	146
7.5	5 Ant	thracite and sewage sludge ash	149
7	7.5.1	Minimum ignition energy (MIE)	149
7	7.5.2	Maximum explosion overpressure and maximum rate of pressure rise	149
7.6	6 Gra	aphite and sewage sludge ash	150
7	7.6.1	Minimum ignition energy (MIE)	150
-	7.6.2	Maximum explosion overpressure and maximum rate of pressure rise	150
8 (	Conclu	usions 1	51
Refe	erence	s 1	55
Ann	ex I		57
Ann	ex II		58

## Letter symbols

bar	bar (unit for pressure)
н	inductance
K/min	kelvin per minute
Μ	median
m	metre
μm	micrometre
mbar	millibar
mg	milligram
mH	millihenry
ml	millilitre
mm	millimetre
ms	millisecond
t <sub>V</sub>	ignition delay time

## Acronyms

- K<sub>ST</sub> specific dust parameter for evaluation of potential hazards
- MIE minimum ignition energy
- p<sub>MAX</sub> maximum explosion overpressure
- (dp/dt)<sub>MAX</sub> maximum rate of pressure rise
- TOC total organic carbon
- TPT Chair of Thermal Processing Technology / Lehrstuhl für Thermoprozesstechnik

## List of figures

Figure 1: Total losses in the process industry worldwide [1]	2
Figure 2: Fire-triangle	3
Figure 3: Detonation and deflagration [1] (slightly modified)	4
Figure 4: Explosive atmosphere hazard warning label [6]	7
Figure 5: 20-I-apparatus [7]	9
Figure 6: Maximum explosion pressure as a function of the dust concentration	10
Figure 7: Maximum rate of pressure rise as a function of the dust concentration	10
Figure 8: Effect of the median on the explosion overpressure [4] [7]	12
Figure 9: Impact of the product humidity [4] [7] (slightly modified)	13
Figure 10: Impact of the temperature [4] [7] (slightly modified)	14
Figure 11: Pressure curve from the 20-I-apparatus [7]	15
Figure 12: The calculated statistic minimum ignition energy [12] (modified)	17
Figure 13: Minimum ignition energy tests [12] (modified)	17
Figure 14: MIKE 3–apparatus [12]	18
Figure 15: Impact of the turbulence [4] [12]	19
Figure 16: Impact of the particle size [12]	20
Figure 17: Impact of the product humidity [4] [12]	20
Figure 18: Impact of the temperature [4] [12]	21
Figure 19: Relation between the dust concentration and MIE [4] [12]	22
Figure 20: Impact of the O <sub>2</sub> -content [12]	22
Figure 21: Composition of sewage sludge ash	25
Figure 22: Sewage sludge ash	26
Figure 23: Curve of the test check [7]	29
Figure 24: Affixing of the igniters [7]	29
	Dege IV

Figure 25: Ignition in the 20-I-apparatus	30
Figure 26: Example for a dust tested in MIKE 3 [12]	31
Figure 27: MIKE3 ignition	32
Figure 28: Low hanging fruits	45
Figure 29: Low hanging fruits	60
Figure 30: Brown coal	67
Figure 31: Brown coal	69
Figure 32: MIE – brown coal	72
Figure 33: Results from the software	73
Figure 34: Comparison wood – charcoal [16] (slightly modified)	74
Figure 35: Charcoal	74
Figure 36: Curve Charcoal	76
Figure 37: MIE - charcoal	77
Figure 38: Hard coal	78
Figure 39: Curve hard coal	79
Figure 40: MIE – hard coal	80
Figure 41: Anthracite	81
Figure 42: Curve anthracite	83
Figure 43: MIE - anthracite	84
Figure 44: Graphite	85
Figure 45: Curve graphite	87
Figure 46: Electrostatic discharges	
Figure 47: MIE - 40% brown coal	94
Figure 48: MIE - 35% brown coal	95
Figure 49: MIE - 30% brown coal	96
Figure 50: MIE - 25% brown coal	97
Figure 51: MIE - 20% brown coal	98
Figure 52: MIE - 40% charcoal	99

Figure 53: MIE - 35% charcoal100
Figure 54: MIE - 30% charcoal
Figure 55: MIE - 25% charcoal
Figure 56: MIE - 20% charcoal
Figure 57: MIE - 40% hard coal104
Figure 58: MIE - 35% hard coal105
Figure 59: MIE - 30% hard coal106
Figure 60: MIE - 25% hard coal107
Figure 61: MIE - 20% hard coal108
Figure 62: MIE - 40% anthracite
Figure 63: MIE - 25% anthracite
Figure 64: MIE – 40% graphite111
Figure 65: MIE – 25% graphite
Figure 66: Explosion overpressure and rate of pressure rise – 40% brown coal
Figure 67: Explosion overpressure and rate of pressure rise – 35% brown coal
Figure 68: Explosion overpressure and rate of pressure rise – 30% brown coal
Figure 69: Explosion overpressure and rate of pressure rise – 25% brown coal
Figure 70: Explosion overpressure and rate of pressure rise – 40% charcoal
Figure 71: Explosion overpressure and rate of pressure rise – 35% charcoal
Figure 72: Explosion overpressure and rate of pressure rise – 40% hard coal
Figure 73: Explosion overpressure and rate of pressure rise – 35% hard coal
Figure 74: Explosion overpressure and rate of pressure rise – 30% hard coal129
Figure 75: Explosion overpressure and rate of pressure rise – 25% hard coal130
Figure 76: Explosion overpressure and rate of pressure rise – 40% anthracite
<b>Figure 77:</b> Explosion overpressure and rate of pressure rise – 40% graphite
Figure 78: Explosion pressure in relation to volatile components
Figure 79: Rate of pressure rise in relation to volatile components
Figure 80: Calorific value in relation to the volatile components

Figure 81: K <sub>ST</sub> - value in relation to volatile components	139
Figure 82: Maximum overpressure in relation to brown coal	141
Figure 83: Pressure rise as a function of brown coal	142
Figure 84: Dependence of K <sub>ST</sub> -value	142
Figure 85: Explosion pressure as a function of charcoal content	144
Figure 86: Rate of pressure rise in relation to charcoal content	145
Figure 87: Dependence of K <sub>ST</sub> -value	145
Figure 88: Explosion pressure in relation to the ratio of hard coal	147
Figure 89: Rate of pressure rise in relation to hard coal	148
Figure 90: KST-value as a function of hard coal	148
Figure 91: Maximum explosion pressure in relation to the ratio of carbon carrier	152
Figure 92: Maximum rate of pressure rise in relation to ratio of carbon carrier	152

### **1** Introduction

Because of the scarcity of phosphorus, a European project, called "RecoPhos – Recovery of Phosphorus", is investigating the possibility of recovering this element from sewage sludge ash. The production of phosphorus is carried out in the thermo-reductive RecoPhos-reactor. where a carbon carrier is needed as reducing agent. Therefore a mixture of sewage sludge ash and reactive carbon is needed in the RecoPhos process which sets the danger of a possible dust explosion at the RecoPhos- apparatus. Therefore it is essential to find the explosive properties of the dust mixture which is used. Various possible carbon carriers will be tested on explosibility to find out the safest alternative for the RecoPhos-process. These carbon sources are four types of coal, as brown coal, charcoal, hard coal and anthracite on the one hand and graphite on the other. For evaluating the possibility of dust explosions, the critical parameters are the explosion indices maximum explosion overpressure (p<sub>MAX</sub>), maximum rate of pressure rise ((dp/dt)<sub>MAX</sub>) and the minimum ignition energy (MIE). To find these explosive properties of various dust mixtures of sewage sludge ash and a carbon carrier, the MIKE 3- apparatus and the 20-I-apparatus from the Kühner AG are used. It can be said that there is an impact of volatility on explosibility. The more volatile compounds the carbon carriers contain, the higher is the explosiveness. Therefore various potential carbon carriers with different volatilities will be tested in order to find the safest mixture for the RecoPhos process. Besides the variation of carbon carrier material, the ratio of carbon in the mixture will be varied to find out the concentration where no explosion occurs.

## **2** Theoretical principles

In the process industry, the main causes for major losses are fires and explosions, as shown in **Figure 1**. Even though fires and explosions are rare, the great danger hails from the possibility of loss of life and the damage to the environment. To the industry the loss of equipment and inventory, together with production interruption cause a high financial damage up to factory closure and therefore loss of many jobs. A long-lasting consequence for the plant operator is the loss of public confidence in the industry. A fire or explosion steams from the presence of combustible substances in the process or in the case of an accident. Every material which is capable of producing an exothermic oxidation reaction can be regarded as combustible. These substances which are combustible gases or gas-mixtures, such as natural gas, methane or other gases. Also combustible liquids, such as waste oil, solvents or other liquids belong to these substances. The last group are dusts of combustible solids, for example coal, metals, plastics or food, like grain [1].



Figure 1: Total losses in the process industry worldwide [1]

Crowl defines the difference between a fire and an explosion by the time frame: "Fires are typically much slower events involving the combustion of materials. Explosions appear due to the sudden release of energy over a very short period of time and may or may not involve combustion or other chemical reactions. AIChE/CCPS (AIChE, 1994) defines an explosion as 'a release of energy that causes a blast.' Crowl and Louvar (Crowl and Louvar, 2002) define an explosion as 'a rapid expansion of gases resulting in a rapidly moving pressure- or shock-wave'" [1]. Explosions cause various types of damage, based on the resulting blast wave, including flying fragments and objects and also thermal energy. Also the subsequent fire can breed more damage and harm. In the chemical industry another impact can result. The risk of a toxic release as a consequence of a fire or an explosion is possible. Besides the danger of a subsequent fire, an explosion can also lead to a secondary explosion, if combustible gases are involved. According to Crowl there are three essential characteristics of an explosion:

- "1. Sudden energy release
- 2. Rapidly moving blast or shock wave
- 3. Blast magnitude large enough to be potentially hazardous" [1]

The occurrence of explosions and fires is based on three requirements: fuel, oxidant and an ignition source. A fire or an explosion is only possible if all these three requirements are present. **Figure 2** shows the well-known triangle of the fire/explosion prerequisites. The difference between a fire and an explosion can also be seen in the fire-triangle. A fire just needs these three requirements, but for an explosion, the proportion between the oxygen and the fuel is also a very important point. If one of these three prerequisites is removed, the fire or explosion does not occur. Therefore fire protection is based on the removal of one component [1].



Figure 2: Fire-triangle

Basically explosions are divided into two types: detonations and deflagrations. The difference can be detectable through the sonic velocity and the reaction front. In a detonation the speed of the reaction front is greater than the sonic speed and the pressure is much greater than 10 bar. If the speed of the reaction front is less than the sonic velocity, the explosion is

called a deflagration and the pressure is around 10 bar or below. **Figure 3** shows this in a more graphic way [1].



Figure 3: Detonation and deflagration [1] (slightly modified)

The more diverse the material, the more diverse is the behaviour of the explosion. Mainly three properties of the material influence the behaviour: The physical state of the material, the physical properties and the reactivity. The physical state is described as solid, liquid or gas, as well as mist or powder. Apart from the behaviour the type of the explosion or fire also differs and depends on various factors. According to Crowl the four factors are "the material's initial conditions of use or storage, the way in which the material is released, how the material is dispersed and mixed with air" and "when and how the material is ignited" [1]. There is a classification for describing explosions. Basically explosions can be classified in two types: the physical and the chemical explosion. The great difference between these two types is the occurrence of a chemical reaction. In the first type, the physical explosion, a sudden release of mechanical energy leads to an explosion without a chemical reaction. Releasing compressed air can cause such an explosion, which can lead, among other damage to vessel ruptures. On the contrary chemical reactions, including combustion reactions and other exothermic reactions, lead to chemical explosions. These explosions have a very dangerous nature. If it is possible they propagate spatially through the reaction mass and damage the whole surrounding. Another classification of explosions knows three types of explosions: an explosive gas (vapour)-air-mixture, an explosive dust-air mixture and hybrid mixtures. A mixture of air and combustible substances in different aggregate states is called a hybrid mixture. An explosive dust-air mixture can lead to a so-called dust explosion [1].

This thesis is concerned with dust explosions. The principles of this type of explosions will be explained in the next chapter.

#### 2.1 Dust explosions

Caution is advised at the occurrence of dust in general. As known historically great explosions in mines or grain elevators were based on dust explosions. According to Crowl a finely ground solid material with 420 µm or less in diameter is defined as dust. These fine solids they can produce both fire and explosions. On the one hand the formation of dust layers on process equipment can be dangerous. These layers may become hot, start to glimmer and can even ignite. On the other hand the dust can be dispersed in air forming a combustible dust cloud, a potential source for an explosion [1]. "A dust explosion is initiated by the rapid combustion of flammable particulates suspended in air" [2]. Due to this rapid combustion special risks exist. 70% of dusts processed in industry require special alertness and even more are combustible and therefor a realistic source for explosions [2]. "The requirements for a dust explosion are: a combustible dust dispersed in air, a concentration above the flammable limit, the presence of a sufficiently energetic ignition source, and certain confinements" [3]. A great danger of dust explosions arises from the course in two or more stages. In the industry dust layers are present, caused by the production processes. The first stage of a dust explosion involves a suspension of the dust from these layers in the air. This can be caused by an air blast or a broken pipe and even by a small dust explosion or by another explosion which is not related to the dust. The dust forms a combustible cloud. Now the second stage is achieved. The dust-air mixture causes a dust explosion, which is frequently larger than the previous explosion. These special hazards cause greater damage. If there is more dust in the surrounding areas the same procedure happens again and again [1].

The production of dust is diverse. Even if the base product has no fines, handling produces finer particles. Solids become pulverized during industry processes caused by mechanical pressure. Pneumatic transfer form segregation and particle size classification. So these fine particles enrich in clouds and there is a potential explosion. Dust explosion tests required by the industry are performed on material of smaller size than used in industry. This realization involves the problem of grinding into a powder [1].

For evaluating the possibility of the occurrence of dust explosions, the critical parameters are the explosion indices maximum explosion overpressure ( $p_{MAX}$ ), maximum rate of pressure rise ((dp/dt)<sub>MAX</sub>) and the minimum ignition energy (MIE). A basic description of these explosion characteristics and the ways they are determined will be given in chapter 2.3.

#### 2.1.1 Mechanism of dust explosions

The course of the explosion is determined by the released volatilities and the solid part of the dust particle. Due to previous investigations it is known that the volatile part of a particle is just crucial in the beginning. The explosion mechanism is described for a single coal dust particle. According to Bartknecht there are three steps for the combustion. The first step involves the heating of the particle and the resulting pyrolysis. The volatile parts get released and the development of tar products starts. The ignition and combustion of the pyrolysis products are the next step in the mechanism. The last and third step involves the solid part of the dust particle. The ignition and combustion of the residual coal and coke part starts. The sequence of these three steps differs among the various types of dust. Particle size and heating rate influence the time sequence. For coal particles a heating rate of 10<sup>6</sup> to 10<sup>9</sup> K/min can be expected [4].

In opposite to this pyrolysis, the mechanism of metal dusts is a chemical surface reaction. Investigations showed that metal dusts had excessive explosion pressures compared to organic dusts. Thus this pressure is formed from the expansion of the residual gases, due to the high temperature of the combustion. According to Bartknecht the reaction of the combustion of solid parts takes place at the surface with the involvement of oxygen. The combustible parts react with flame propagation. After an ignition, the combustion process and the energy are spreading to the contiguous particles. Besides convection, light beam takes over a great part of the energy transport [4].

#### 2.2 Legal Basis – Directive 1999/92/EG

To establish a maximum of protection of workers the European Commission launched a Directive for the improvement of safety and health of workers, near or in potential spaces of explosions. Therefore the Directive 1999/92/EG of the European Parliament and the Council of 16 December 1999 on minimum requirements for improving the safety and health protection of workers potentially at risk from explosive atmospheres were adopted.

According to Article 2 of this Directive an "explosive atmosphere' means a mixture with air, under atmospheric conditions, of flammable substances in the form of gases, vapours, mists or dusts in which, after ignition has occurred, combustion spreads to the entire unburned mixture" [5].

To cope with the hazard of dust explosions, the Directive classifies zones in hazardous areas, depending on the duration and the frequency of the occurrence of an explosive atmosphere. In the context of dust explosions three zones are known:

"Zone 20

A place in which an explosive atmosphere in the form of a cloud of combustable dust in air is present continuously, or for long periods or frequently.

#### Zone 21

A place in which an explosive atmosphere in the form of a cloud of combustible dust in air is likely to occur in normal operation occasionally.

#### Zone 22

A place in which an explosive atmosphere in the form of a cloud of combustible dust in air is not likely to occur in normal operation but, if it does occur, will persist for a short period only" [5].

There are some comments to make for the understanding of these three zones: "Layers, deposits and heaps of combustible dust must be considered as any other source which can form an explosive atmosphere" and "'normal operation' means the situation when installations are used within their design parameters" [5].

So if there occurs an explosive atmosphere, special equipment has to be used. According to Directive 1999/92/EG, three categories of equipment are known, which are suitable for gases, vapours or mist and/or dust: In zone 20 equipment of the category 1 has to be used. In zone 21 equipment of the category 1 or 2 can be used and for the zone 22 equipment of category 1, 2 or 3 can be used. Pursuant to Article 7(3) there must be a warning sign at such a hazard zone. A triangular shape is binding, "with black letters on a yellow background with black edging (the yellow part to take up at least 50% of the area of the sign)" [5].



Figure 4: Explosive atmosphere hazard warning label [6]

The employer is required to make an assessment of the special explosion risks. The assessment is regulated in Article 4 of the Directive. This article says that the employer has to take into account "the likelihood that explosive atmospheres will occur and their persistence, the likelihood that ignition sources, including electrostatic discharges, will be present and become active and effective" and "the scale of the anticipated effects". Furthermore "explosion risks shall be assessed overall". In addition there must be an assessment to identify "places which are or can be connected via openings to places in which explosive atmospheres may occur" [5].

To meet the requirements of the Directive, special tests on potential explosion scenarios have to be done. There are various standardized tests to find out if the process brings out an explosive atmosphere, for example, by producing dust. So tests with this special concentration and with this special particle size have to be done. The description of standardized tests will be explicated in the following chapter.

#### 2.3 Explosion indices

#### 2.3.1 Maximum explosion overpressure and maximum rate of pressure rise

To get knowledge about the potential violence of the reactions of dust/air mixtures the explosion indices maximum explosion overpressure  $p_{MAX}$  and the maximum rate of pressure rise (dp/dt)<sub>MAX</sub> have to be determined. These two maximum values from explosion tests with dust are derived from systematic test series within large concentration range. At the ideal dust concentration for the tested dust, the two parameters maximum explosion overpressure  $p_{MAX}$ and maximum rate of pressure rise (dp/dt)<sub>MAX</sub> can be seen. It has to be taken into account that the ideal concentration varies between the different types of dusts. The two indices are determined in a standardized 20-liter-apparatus from the Kühner AG. "The test chamber is a hollow sphere made of stainless steel, with a volume of 20 liters. A water jacket serves to dissipate the heat of explosions or to maintain thermostatically controlled test temperatures. For testing, the dust is dispersed into the sphere from a pressurized storage chamber via the outlet valve and a nozzle. The ignition source is a chemical igniter and located in the centre of the sphere" [7]. At the start of a test trial there must be 600 ml in the container. To get as close as possible to working conditions, the tests have to be done at normal pressure, so a preevacuation to the absolute pressure of 400 mbar is essential. After this pre-evacuation the dust is pressed into the 20-liter vessel from the dust storage container with an overpressure of 20 bar. The testing procedure cores tests at different dust concentrations for every mixture.

For testing various dusts a source of sufficient energy has to be used. According to EN 14034-1 for industrial testing there is only one mode of ignition for reliable determination of the explosion indices: Two chemical igniters of 5000 J each are used, so the trials in the 20-l-apparatus were made with an ignition energy of 10 kJ. In industry such high ignition energy are extremely unlikely, but the worst case has to be taken into account. With such a high energy most industrial dusts can be tested. The turbulence at the moment of ignition is very relevant to the maximum explosion overpressure  $p_{MAX}$  and the maximum rate of pressure rise  $(dp/dt)_{MAX}$ , so for testing the dust mixtures a constant ignition delay time has to be standardized. The trials were made with an ignition delay time of 60 ms. If the ignition delay time does not comply with 60 ms, problems with the concentration can be the result. With a delay time over 60 ms the dust/air mixture is not full made up. On the other hand with a delay time over 60 ms the dust/air mixture begins to demix, caused by sedimentation [7] [4] [8] [9]. **Figure 5** shows the 20-l-apparatus.



Figure 5: 20-I-apparatus [7]

The maximum explosion overpressure is effectively volume-independent, because of the positioning of the ignition source in the middle of the chamber. "The maximum pressure for the most flammable gases and dusts is in the range of 7-10 bar(g)" [2]. **Figure 6** shows the alteration of the maximum explosion overpressure as a function of the dust concentration in the vessel.



Figure 6: Maximum explosion pressure as a function of the dust concentration

Another safety-relevant parameter, measured with the 20-I-apparatus, is the maximum rate of pressure rise  $(dp/dt)_{MAX}$ . In contrast to the explosion overpressure, the  $(dp/dt)_{MAX}$ -value depends on the volume. Based on the cubic law, the  $(dp/dt)_{MAX}$ -value shrinks with the rising vessel volume [7].



Figure 7: Maximum rate of pressure rise as a function of the dust concentration

The  $K_{ST}$ -value is a dust-specific parameter which is based on the (dp/dt)-value. This safety relevant parameter is used to make measurements from various apparatuses with different volumes comparable and it is based on the so called cubic law. The cubic law can be applied to a minimum volume of 20 liters. So this is the reason for using a testing apparatus of 20 liters. The maximum explosion overpressure is independent of the vessel volume, whereas the

maximum rate of pressure rise decreases with the rising vessel volume. Caused by this, the  $K_{ST}$ -value is volume independent [7] [4].

$$K_{ST} = \left(\frac{dp}{dt}\right)_{\max} * V^{\frac{1}{3}}$$
(2-1)

To categorize the explosion intensity the  $K_{ST}$ -value separates the reaction into dust explosion classes:

Dust explosion class	K <sub>sT</sub> -value [m*bar/s]
St 1	>0-200
St 2	201-300
St 3	> 300

Table I: Dust explosion classes [7] (slightly modified)

The declaration of the dust explosion class only defines the behaviour of the explosion, not the ignition behaviour. Therefore the probability of the explosion occurrence or the explosion impact cannot be predicted by the classification. Most dusts in the industry belong to the explosion class 1 [4] [7].

#### 2.3.1.1 Influential parameters

For the explosion tests a few influential parameters have to be taken in account. The effectivity and the impact of these values are described in the next paragraphs.

#### Turbulence

Turbulence within the dust cloud current is a very important parameter that influences the progress of the dust explosion. The ignition delay time can be set to adjust the turbulence level. The time between the onset of the dust dispersion and the start of the ignition source is called the ignition delay time  $t_V$  and for testing, a constant  $t_V$  has to be standardized. The standard ignition delay time for the 20-I-apparatus is 60 ms. If the ignition delay time does not comply with 60 ms problems with the concentration can be the result. With a delay time under 60 ms

the dust/air mixture is not fully mixed. On the other hand with a delay time over 60 ms the dust/air mixture begins to demix, caused by settling [4] [7].

#### Particle size

To characterize the particle size of dust the median M of the material has to be taken. Based on grading the dust, the median is the 50% value of the particle size distribution curve. The median M has to be taken into account for explosion tests, because of the considerable influence of the explosion indices. Finer dust has a much more severe reaction than coarsely ground material, but principally this can be seen in the violence of the explosion, in form of the K<sub>ST</sub>-value. **Figure 8** from Bartknecht shows that the effect of the median on the explosion overpressure is not as high as on the violence of the explosion (K<sub>ST</sub>-value) [4] [7].



Figure 8: Effect of the median on the explosion overpressure [4] [7]

Based on this influence of the median M on the explosibility, the testing practice for explosion indices has to be done only with a median lower than 63  $\mu$ m. This parameter has to be taken into account to get optimal values of the products. Pursuant to Bartknecht, particles with diameters of 400 to 500  $\mu$ m, even in presence of strong ignition sources, react non explosive. It must be kept in mind that coarse material can also react explosively, caused by particulate matter formed by abrasion. According to Bartknecht, a fine dust/coarse dust-mixture can allow an explosion with a content of 5 to 10% fine dust. Particular care has to be taken caused by the produced maximum explosion overpressure of these mixtures. An under limit for the content of fine dust to eliminate the requirement of safety equipment for explosions is not known [4].

#### **Product Humidity**

Another influential parameter for the violence of explosions is the product humidity, the ratio between water and dry matter. The view which used to prevail, that the content of a few % of water form inexplosive characteristics is refuted by **Figure 9** from Bartknecht. It can be seen that a minimization of the violence of the explosion arise at a relatively high water content. For explosion testing trials the product humidity should be clearly below 10% to eliminate the influence of this parameter and to get optimal values of the product [7].



Figure 9: Impact of the product humidity [4] [7] (slightly modified)

Products with a water content of more than 10% achieve a lower dispersion capacity, whereby the danger of forming an explosive dust/air-mixture is shrinking. Contrary to this behaviour, solvent-containing products show rising explosion indices with a high water content. The explosion indices of an ethanol-containing product falls first with the rising water content and later rises with a high product humidity. This behaviour is attributed to the losing of the dust character of the material. The material is no longer a dust, because the rising solvent-content caused agglomeration. The agglomerated product forms a solvent/air-mixture in the explosive space around the product. The occurrence of a spark ignites the solvent/air-mixture [4] [7].

#### **Initial pressure**

Explosions in vessels are influenced by the initial pressure, the pressure at the moment of ignition. The maximum explosion overpressure and the  $K_{ST}$ -value is directly proportional to the initial pressure. Also the optimal dust concentrations for the explosion indices rise linearly with the initial pressure, thus a doubling of the initial pressure leads to a doubling of the explosion

indices. Due to the fact that the curve does not cross the point of origin Bartknecht says that dust explosions are not possible under a pressure limit of 10 mbar [4] [7].

#### Temperature

In the industry dusts are often produced at high temperatures, so the temperature can be a very important parameter. As seen in **Figure 10**, the pressure of the explosion ( $p_{MAX}$ ) decreases linearly with the reciprocal temperature. This temperature dependency is caused by the falling oxygen content, which can be calculated by the ideal gas law [7].



Figure 10: Impact of the temperature [4] [7] (slightly modified)

The behaviour of the K<sub>ST</sub>-value also shows a linear dependency on the temperature, based on the oxygen content. Usually a reduction of the oxygen concentration at ambient temperature leads to a reduction of the maximal rate of pressure rise and so to a falling K<sub>ST</sub>-value. There are two types of reactions and so there are two forms of the dependency. If the dust reacts more violently, a high temperature leads to a linear reduction of the K<sub>ST</sub>-value. If the dust reacts slowly, the K<sub>ST</sub>-value increases. Taken as a whole the influence of an increasing temperature up to 300°C on the K<sub>ST</sub>-value is very low, so for industrial processes the temperature influence can be neglected if they are below 300°C [4] [7].

#### 2.3.1.2 Appraisal of the trials and definitions of the pressure curve

The software of the 20-I-apparatus (KSEP 6.0f) from Kühner AG produces a pressure curve for every dust tested. **Figure 11** shows such an ideal curve for one experiment. On the abscissa the time can be seen and on the ordinate the pressure in the vessel is plotted. The maximum explosion pressure is the highest measured pressure during the test and can be seen at the peak of the curve. This maximum explosion pressure has to be corrected, because

of cooling and pressure effects of the chemical igniters. To get the real maximum explosion overpressure  $p_{MAX}$  of a dust, a wide range of dust concentrations has to be tested. From these tests the maximum corrected overpressure is the maximum overpressure  $p_{MAX}$  for the product. The maximum rate of pressure rise can be calculated by laying a tangent on the pressure time curve. On the diagram the time  $t_1$  describes the duration of the combustion, the time between the ignition and the culmination point of the curve. The ignition delay time  $t_V$  can also be seen in **Figure 11**. The testing takes place as follows: To get an ambient pressure for the experiment, the test procedure starts with an evacuation to -0.6 bar. This can be seen in the curve at the beginning. Then the dust is released from the storage container, seen in **Figure 11** as the point between  $t_d$  and  $t_V$ . During the ignition delay time the pressure in the vessel reaches ambient pressure. The red line in the diagram stands for the ignition point. The pressure in the vessel rises up to the maximum explosion overpressure  $p_{EX}$  [7].



Figure 11: Pressure curve from the 20-I-apparatus [7]

#### 2.3.2 Minimum ignition energy

To get an idea of the combustibility, it is necessary to determine the minimum ignition energy (MIE) of the dust to be used. As described by Eckhoff the "MIE is defined as the lowest spark energy that gives at least one ignition in ten trials at the same spark energy" [10]. The control of the time-based behaviour is an essential point of the investigations for the MIE. To alter the

spark energy and the behaviour in time a circuit of capacitors, inductances and resistors is installed. "By definition, minimum ignition energy data refers to protracted capacitor discharges. These are generally clearly more incentive than purely capacitive discharges" [10]. "In standard testing for MIE optimal discharge times for ignition is achieved by introducing a 1-2 mH inductance in the discharge circuit, and this feature is compulsory in the current international (IEC) and European standard methods for MIE determination for dust clouds" [10] [11]. The effective minimum ignition energy of an inflammable dust lies within an energy range. The MIE lies between the lowest energy value (E2) at which ignition occurred at the tests and the energy (E1) at which no ignition occurred [12].

$$E1 < MIE < E2 \tag{2-2}$$

With these two values a comparison between different apparatuses is not possible, therefore one value instead of a range is used. It is called the statistic MIE value (Es) and can be calculated by the use of the probability of ignition: [12]

$$Es = 10^{\wedge} \left( \log E2 - \frac{I[E2]^* (\log E2 - \log E1)}{(NI + I)[E2] + 1} \right)$$
(2-3)

In this formula I[E2] stands for the number of tests with ignition at the energy E2 and (NI+I)[E2] for the total number of tests at the energy E2. For demonstration of the calculations the following table is taken from the tests with brown coal dust. In **Table II**, I stands for the ignition of dust and NI for no ignition of dust in 10 trials.

	IE \ mg	1200	1500	1800	2400	3000		Probability
E2 =	100 mJ	NI	Ι	Ι	Ι	NI	$\rightarrow$	3 of 5
E1 =	30 mJ		NI	NI	NI			

Table II: Example of a minimum ignition energy test

So this means that the minimum ignition energy MIE lies between 30 mJ and 100 mJ. The statistic MIE value for the minimum ignition energy is calculated with:

E1 = 30 mJ

E2 = 100 mJ

#### (NI+I)[E2] = 5

The calculated statistic MIE value ( $E_s$ ) with formula (2-3) is 54.77 mJ. **Figure 12** shows the calculated statistic MIE value ( $E_s$ ). The square stands for no ignition in 10 trials and the orange square for an ignition.



Figure 12: The calculated statistic minimum ignition energy [12] (modified)

Examples for other tests can be seen in **Figure 13**. The statistic MIE value ( $E_s$ ) is always calculated the same way. Only the occurrence of the ignition differs in the two trials.



Figure 13: Minimum ignition energy tests [12] (modified)

#### 2.3.2.1 MIKE 3 - apparatus

For testing dusts, the MIKE 3-apparatus from the Kühner AG was used. An image of the MIKE 3-apparatus can be seen in **Figure 14**. According to Kühner AG, for this MIKE 3 "a modified Hartmann tube made of glass with a volume of 1.2 liter is used as explosion vessel. The dust dispersion system at the base of the tube is of the 'mushroom-shaped'-type on which the sample is loosely scattered. A blast of compressed air at 7 bar is used to disperse the dust in the glass cylinder where it is ignited by a spark between two electrodes" [12]. For the determinations of the explosibility of dust, minimum conditions of the apparatus have to be

fulfilled. When an inductance in the discharge is used, the inductance H should be between 1 mH and 2 mH to produce a time protracted spark. The material of the two electrodes has to be made of tungsten or stainless steel with a diameter of 2 mm and the space between the two electrodes must be at least 6 mm [12].



Figure 14: MIKE 3-apparatus [12]

#### 2.3.2.2 Influential parameters

For the tests on the minimum ignition energy of dust a few influential parameters have to be taken in account. These parameters are described in the next paragraphs.

#### Inductance in the discharge circuit

To generate time protracted sparks, as they are required for MIE tests, inductances have to be used. These protracted sparks are more incendive than short sparks, formed without an inductance [12].

#### Turbulence

The turbulence of the dust current is a very important parameter, which influences the outcome of the experiments. To adjust the turbulence the ignition delay time can be set. The time between the actuation of the outlet valve and the sparkover is called the ignition delay

time  $t_v$ . The ignition delay time for the trials in the MIKE 3 is more or less 120 ms. A short delay time induces a high turbulence, and a long delay time leads to a very low turbulence. According to Bartknecht, in contrast to the explosion indices explosion overpressure and maximum rate of pressure rise ( $t_v = 60$  ms), minimum values for the MIE needs a longer delay time. Therefore the turbulence in the dust/air mixture has to be low and so more or less 120 ms are used [4] [12]. The influence of the turbulence can be seen in **Figure 15**.



turbulence

Figure 15: Impact of the turbulence [4] [12]

#### Particle size

The median M of the material is used to characterize the particle size of the dust. Based on grading the dust, the median is the 50% value of the particle size distribution curve. The median M has to be taken into account for MIE tests. The finer the dust, the more likely the dust can be ignited by a time protracted discharge of a capacitor. For characterising the ignition behaviour of a product the particulate matter has to be tested [4] [12]. **Figure 16** shows this correlation.



Figure 16: Impact of the particle size [12]

#### **Product humidity**

Another influential parameter of the minimum ignition energy is the product humidity, the ratio of water to dry matter. The influence of the humidity is more instinctive than the influence of the particle size. In general a water content below 5% has just a slight influence, whereas a product moisture above 10% influences the minimum ignition energy by increasing around one to the power of ten [12]. **Figure 17** shows that the impact of the humidity depends on the dust used.



Figure 17: Impact of the product humidity [4] [12]

#### Temperature

The temperature has also a great influence on the minimum ignition energy MIE. With increasing temperature the ignition energy of hard ignited dust is falling down much faster than the ignition energy of readily ignited dusts. In a log-log plot the straight lines meet at the point 1000°C and 0.088 mJ. It seems probable that the energy limit value related to a temperature of 1000°C for a wide range of dusts is 0.088 mJ. To get an estimated value of the temperature dependence of a product, just the minimum ignition energy at room temperature has to be investigated and this point has to be connected with the point 1000°C / 0.088 mJ [4] [12]. The impact of the temperature on the MIE can be seen in **Figure 18**.



Figure 18: Impact of the temperature [4] [12]

#### **Dust concentration**

Also the dust concentration has a negligible impact on the ignition energy of a product. The relation between these two values is a parabolic curve, as seen in **Figure 19**. Therefore ignition tests have to be made over a wide range of concentrations.


dust concentration

Figure 19: Relation between the dust concentration and MIE [4] [12]

## **Oxygen concentration**

Because oxygen is necessary for combustion, the oxygen content has an influence on the minimum ignition energy. This impact can be seen in **Figure 20**. Transforming this into a semilogarithmic plot, a linear rise of the MIE can be seen. The linear line is explained on the one hand by the rising nitrogen content, by the falling oxygen content and vise versa. A low oxygen content results in a high nitrogen content, which has an inerting effect. A high oxygen concentration provides enough  $O_2$  for combustion and leads to a low nitrogen content.



Figure 20: Impact of the O2-content [12]

# **3** Test procedure and sample preparation

# 3.1 Preparation of the dust

Because a mixture of sewage sludge ash and reactive carbon is needed in the RecoPhos process the requirements for a dust explosion may exist in RecoPhos-apparatus. Due to this it is essential to find out the explosive properties of the dust mixture to be used. Various possible carbon carriers will be tested for explosibility to determine the safest alternative for the RecoPhos-project. The composition of the used sewage sludge ash can be seen in **Table III** and in **Figure 21**. The results from the test of the ash blending behaviour are shown in **Table IV** and the diagram can be seen in the Annex I.

Oxides	SiO <sub>2</sub>	32.471%
	Fe <sub>2</sub> O <sub>3</sub>	11.613%
	Al <sub>2</sub> O <sub>3</sub>	9.080%
	MgO	3.157%
	K <sub>2</sub> O	1.720%
	TiO <sub>2</sub>	0.852%
	Na₂O	0.525%
	MnO <sub>2</sub>	0.145%

Table	III:	Reference	Ash	Analysis
-------	------	-----------	-----	----------

	SrO	0.114%
	BaO	0.103%
	CaO	0.006%
Phosphates	Ca <sub>3</sub> (PO <sub>4</sub> ) <sub>2</sub>	27.014%
	FePO <sub>4</sub>	6.980%
	AIPO <sub>4</sub>	3.540%
Sulphides	FeS	0.794%
	CaS	0.535%
	MgS	0.326%
	$AI_2S_3$	0.040%
Carbides	Fe₃C	0.353%
	TiC	0.039%
	Al <sub>4</sub> C <sub>3</sub>	0.025%
	SiC	0.018%
	CaC <sub>2</sub>	0.011%
Halogens	TiCl <sub>2</sub>	0.006%
	CaF <sub>2</sub>	0.004%
Inorganic	As, Cd, Co,	0.498%
components	Cr, Cu, Hg, Ni, Pb, Zn	
тос	$C_{14}H_{10}$ to $C_{22}H_{14}$	0.030%



Figure 21: Composition of sewage sludge ash

Sintering	980°C
point	
Softening	1090°C
temperature	
Flow	1230°C
temperature	
Softening	1090°C to
Softening range	1090°C to 1210°C
Softening range Yielding	1090°C to 1210°C 1210°C to
Softening range Yielding region	1090°C to 1210°C 1210°C to 1230°C

#### Table IV: Ash melding behaviour

The first step for the investigations was to find out the particle size distribution of the sewage sludge ash. A Helos Vario KF Magic from SYMPATEC was used to determine the particle size distribution contactlessly by laser diffraction. Three tests were made. The results of the tests can be taken from the Annex II and the three results from the median M can be seen in **Table V**. The sewage sludge ash has a median M of 73.2367  $\mu$ m, so the dust of the carbon sources has to be around this size, thus the coals and the graphite was ground to 50  $\mu$ m by a mill. The drying of the dusts and the sewage sludge ash was the next step.

	Median M
Test 1	79.12 µm
Test 2	67.32 µm
Test 3	73.27 µm

Table V: Helos-measurement



Figure 22: Sewage sludge ash

The conditions of the drying chamber were 24 hours at a temperature of 108°C. Some products were dried in parts and some completely as one piece, but for all materials, the conditions were equal. After drying the material had to cool down to room temperature. The reduction of weight after the drying can be seen in **Table VI**.

Material	Weight before the artificial drying	Drying conditions	Weight after the artificial drying	Weight loss [g]
	[g]		[g]	
Graphite	2777.4	108°C,	2775.9	1.5
		24 hours		
Hard coal	2118.6	108°C,	2113.0	5.6
		24 hours		
Sewage	2285.0	108°C,	2283.2	1.8
sludge ash		24 hours		
Sewage	2346.9	108°C,	2345.1	1.8
sludge ash		24 hours		
Charcoal	3039.5	108°C,	2962.0	77.5
		24 hours		
Sewage	2201.9	108°C,	2200.7	1.2
sludge ash		24 hours		
Anthracite	2206.6	108°C,	2155.2	51.4
		24 hours		
Sewage	2737.2	108°C,	2735.5	1.7
sludge ash		24 hours		
Sewage	3630.4	108°C,	3628.4	2.0
sludge ash		24 hours		
Brown	4226.7	108°C,	3687.2	539.5
coal		24 hours		
Anthracite	2547.2	108°C,	2479.3	67.9
		24 hours		

Table VI: Drying

To sum up the results, the average reduction of weight after drying is shown in **Table VII**. As can be seen the highest weight loss occurs to brown coal, so the volatile matter of brown coal is very high. In contrast, the weight loss of graphite is very low. This means that graphite has very few volatile components. Also sewage sludge ash has nearly no weight loss, but in this case the burning of the sewage sludge has eliminated the bulk of the volatile compounds.

Material	Water
	content [%]
Graphite	0.054
Sewage	0.0656
sludge ash	
Hard coal	0.264
Anthracite	2.4975
Charcoal	2.549
Brown coal	12.764

#### Table VII: Water content

After pretreatment the dust was ready for the experiments in the 20-I-apparatus and the MIKE 3–apparatus. For the testing the coal-dust was mixed with the sewage sludge ash in various mixture ratios: 20%, 25%, 30%, 35% and 40% of coal dust. The upper limit for the percentage of the carbon carrier in the mixture sewage sludge ash and carbon carrier for the RecoPhos reactor is 40%, therefore the highest ratio is 40%.

# 3.2 Test procedure in the 20-I-apparatus

At the start of a new trial the 20-I-apparatus has to switched on and the software "KSEP" has to be started. To get results, which will be as near as possible at working conditions, the operating temperature should correspond to room temperature. So a water cooling system is installed and with the help of two thermometers, the temperature of 20°C can be proved and held. Every series started with a check of the function of the 20-I-apparatus. It helps to check the system for the correct function in a simple way. The test sequence is made without dust and without chemical igniters. Before starting with a new trial, after a pause or at the beginning of a new day this test check has to be done. For this, all valves and the dust chamber have to be closed. With the software KSEP a new file will be created and so the test check can start. The pressure curve of a correct test check is shown in **Figure 23**.



Figure 23: Curve of the test check [7]

It can be seen that the curve begins below atmospheric pressure caused by the evacuation of the 20-I-apparatus. At around 40 ms the dust is blown in and the system reaches normal pressure. After the test check the first dust can be tested. Starting at a low concentration, with every test the concentration is raised until reaching the highest value for the explosion overpressure and the rate of pressure rise. According to Kühner AG the following steps should be used:

60 g/m<sup>3</sup>; 125 g/m<sup>3</sup>; 250 g/m<sup>3</sup>; 500 g/m<sup>3</sup>; 750 g/m<sup>3</sup>; 1000 g/m<sup>3</sup>; 1250 g/m<sup>3</sup>; 1500 g/m<sup>3</sup>

The next step is to check the observed maxima twice. This means that around the observed maximum of the explosion overpressure and the maximum rate of pressure rise, a new test serial has to be done. With these two series, the maximum explosion overpressure, the maximum rate of pressure rise and the  $K_{ST}$ -value for the dust can be calculated.

A special fitting of the igniters is required. The ignition source should be in the middle of the 20-I-apparatus and the explosion should spread spherically to the wall of the apparatus. One chemical igniter has the energy of 5 kJ, therefore two igniters have to be mounted on the electrodes. **Figure 24** from the Kühner AG shows the correct mounting of the igniters. With this fixture the igniters fire in opposite directions [7].



Figure 24: Affixing of the igniters [7]



Figure 25: Ignition in the 20-I-apparatus

# 3.3 Test procedure in the MIKE 3-apparatus

For the determination of the minimum ignition energy the MIKE 3-apparatus from the Kühner AG was used. After starting the apparatus and the software, the sparkover can be tested. Therefore various spark energies can be used, but without dust. If a spark can be seen, the system works. After every cleaning the electrode spacing has to be checked with a spacer of 6 mm. For the first series a low dust concentration was used. The test started with a high energy of 1000 mJ and an ignition delay time of 120 ms. The start was initiated by clicking "OK" or the "flash"-button. The test had to be observed, because it had to be noted if the dust ignited or not. The next test started by pressing the button "NO" for no ignition or "YES" if an ignition occurred. Because dust adheres to the tube wall and the electrodes, with every test a small amount of dust can escape and therefore the dust concentration decreases. According to the Kühner AG experiments have shown that it is only possible to do four tests with the same filling [12]. Kühner AG recommends the following procedure [12]:

3 tests Cleaning the tube Fresh dust sample 3 tests Cleaning the tube Fresh dust sample 4 tests

If an ignition occurred (**Figure 27**) the spark energy could be lower for the next series. If there has been no ignition, the concentration can be altered. For the spark energy the following steps are possible: 1000 mJ, 300 mJ, 100 mJ, 30 mJ, 10 mJ, 3 mJ and 1 mJ. For the concentration there are also fixed steps: 300 mg, 600 mg, 900 mg, 1200 mg and 1500 mg. The results of a tested dust can be seen in **Figure 26**.



Figure 26: Example for a dust tested in MIKE 3 [12]

The solid squares belong to tests with an ignition and the circles to the series without an ignition. So this dust was tested first with a concentration of 900 mg and a spark energy of 100 mJ. After an ignition the spark energy was reduced. At the third series the ignition did not occur at the first test (shown by the T-shaped). The next trial (number 4) was made with a reduced spark energy of 3 mJ where no ignition occurred. As a result the next step was to get back to higher energy (10 mJ) and make tests with various concentrations. At test number 5 an ignition occurred, thus another test with lower energy and a concentration of 1200 mg had

to be made. The results show that the lowest energy value where ignition occurred (E1) was 10 mJ and the energy where no ignition occurred (E2) was 3 mJ. Therefore the minimum ignition energy lay between these two energy levels.



Figure 27: MIKE3 ignition

# **4** Process description

# 4.1 Process description for the 20-I-apparatus

# 4.1.1 Identification of the process

The first step of process-management is the identification and distinction of the processes. Therefore a distinct name for the process is needed. The fist and the last step of the process, as well as the input and output define the process [13].

Name of the process	Investigation on the maximum explosion overpressure and the maximum rate of pressure rise
Purpose	The purpose of the process is the determination of the maximum explosion pressure and the maximum rate of pressure rise for dust explosions. This comports to the turnover of the organisation.
Main customer	Industry partner Chair of Thermal Processing Technology
Prospect of the	
customer	Results with constant quality
Output	Maximum explosion overpressure p <sub>MAX</sub> Maximum rate of pressure rise (dp/dt) <sub>MAX</sub> Report
Input	Special concentration of dust Igniters of 10 kJ
First step of the process	Start-up operation

Table VIII: Identification of the process

Last step of the process	
· ·	Print report
Interface input	Industry partner
	Chair of Thermal Processing Technology
Interface output	Industry partner
	Archive (reports)
	Accounting
Required resources	Lab personnel
	Head of laboratory
	Project manager
Information, know-how	Enrolment
	Submission of report
Working area	
	Laboratory
Equipment	Electricity
	Vacuum pump
	Dust
	20-I-apparatus
	Igniters for the apparatus
	Synthetic air
	Intranet
	Personal computer
	Scale
	Water
	Personal protective equipment (PPE)
Factor of success	Clear procedure
	Communication
	Documentation takeover
Applicable documents	Standard EN 14034-1
	Standard EN 14034-2
	Manual
	Data base Gestis StaubEx
	Know-how
	Technical appendix

# 4.1.2 Actual state of the process

The actual state is the description of the present state of the process. Therefore the process is described like the lab personnel conducts the working steps. The following figure shows the sequence of the tasks of the process along the vertical axes. The description of the process has to be made in a way that every person who does not know the process will be able to perform it. It has to be obvious and understandable [13].



Master's thesis Katja Hüttenbrenner



Master's thesis Katja Hüttenbrenner







# Table IX: Actual state of the process

Nr	Element	Explanation
1	Instruction	The needed information are delivered to the lab personnel
	delivered	If some information is not available, the head of laboratory has to be
2	Consultation	consulted
3	Defined	The lab personnel has to decide if the instruction she/he got is
	Enrolment for	enough of it she/he needs more information
4	20-I-apparatus done?	The head of the laboratory has to prove if the lab personnel has had an enrolment
5	Enrolment of the personnel	An enrolment for the personnel for the MIKE 3-apparatus has to be done
6	Particle size analyses	A particle size analyses has to be done
7	Dust particle <63µm	With the help of a particle size analysis it has to be decided if the dust particles are smaller than $63\mu m$
8	Dust milling	The material has to be milled to reach a size of less than $63\mu m$
9	Dust drying	The dust has to be dried with the help of the compartment drier
10	Cool down the dust	After the artificial drying the dust has to be cooled down
11	Determine dry matter	The dust has to be weighed to find out the dry matter
12	Switch on 20-l- apparatus	The 20-Iapparatus has to be switched on
13	Turn on water	The water has to be turned on for the jacket cooling (around 25°C)
14	Switch on air supply	The air supply has to be switched on
15	Start KSEP 6.0f	The software KSEP 6.0f has to be started
16	Create worksheet	A new worksheet has to be created with the help of the software
17	Switch on exhauster	The exhauster has to be switched on
18	Weigh out needed dust quality	The concentration of dust for the trail has to be weighed out
19	Insert dust in chamber	The needed dust has to be brought up in the storage container
20	Mount igniters	The igniters have to be mounted as described in the manual
21	Connection of the igniters with the power supply	The igniters have to be connected with the power supply
22	Close levers	The two (red) levers have to be closed

	Adjust	
23	concentration of dust	The concentration of the dust has to be adjusted in the program KSEP 6.0f
24	Evacuate apparatus	The 20-I-apparatus has to be evacuated with the help of the installed vacuum pump
25	Start trial	The trial has to be started with the start-button in the program
26	Evaluate the data	The trial has to be evaluated with the help of the diagram
27	Open connection of the igniters	The connection between the igniters and the power supply has to be unconnected
28	Open levers	The levers have to be opened
29	Flush with air	The 20-I-apparatus has to flushed with air
30	Open storage chamber	The storage chamber has to be opened
31	Open cap	The cap of the apparatus has to be opened and the igniters have to be removed
32	Clean apparatus	The 20-I-apparatus has to be cleaned: hoover it, clean it with a damp cloth and dry it
33	P <sub>MAX</sub> is falling or zero?	With the help of the diagram it can be said if the curve is falling or if $p_{MAX} = 0$
34	Are all needed concentrations tested?	With the help of the manual and the European standard all needed dust concentrations can be found out
35	Switch off exhauster	The exhauster has to be switched off
36	Print and save report	The report has to be saved and also printed
37	Turn off water	The water has to be turned off for the jacket cooling
38	Switch off air supply	The air supply has to be switched on and the conductions have to be depleted from air
39	Close KSEP 6.0f	The software KSEP 6.0f has to be closed
40	Switch of the 20-I-apparatus	The 20-I-apparatus has to be switched off

# 4.1.3 Analysis of the process

# 4.1.3.1 Moments of truth

According to Wagner the moments of truth are at any time when the customer gets in contact with the organization or with the product. For the customer the process is a serial of meetings with the performance of the organization. These moments of truth are important, because they form the impression of the customer for the product or the organization and therefore they are crucial to the organization [13].

## What happens at the first contact of the customer with the organization?

Normally the first contact is a phone call where the customer describes his problem and gets information form the organization about the price of the testings, the needed time and the needed dust quantity.

## What happens when the service/product gets used the first time?

The customer gets a quotation from the organization. Also a detail time frame for the investigations and the exactly needed dust quantity will be discussed.

## Which information gets the customer from the organization?

Information about the used procedure, the time frame of the investigation and the price of the service. This information is committed clearly und adequately.

## Which impression gets the customer from the employee?

The employees of the organization make a good and competent impression to the customer

# In which way experiences the customer the organization in case of complaining?

Those responsible take the complaining serious and deal with it in the right way to improve the process

## 4.1.3.2 Voice of the customer

The expectations of the customer and the actually fulfilled expectations have to be found out. Based on the identification of the process the customers of the organization are known and so the expectations can be identified. The measurement can be made by operation numbers or by interviewing the customer. Both qualitative and quantitative statements can furnish clues [13].

Voice of the customer	Problems in the	Requirements of the
	process	process
Investigations are not	Too less human	Sufficient human
done on schedule	resources	resources
Documentation is	The submission of the	A model report is
inadequate	report is not changeable	available
I am not used to the	Inadequate enrolment	Periodic enrolment
procedure		

#### Table X: Voice of the customer

## 4.1.3.3 Interfaces

In the context of interfaces, processes with interfaces have to be found out. Therefore it is essential to know what (data and information) and in which way (by letter, oral, electronically) it is delivered [13].

#### Table XI: Interfaces

Name of the process: Investigation on the maximum explosion overpressure and the maximum rate of pressure rise				
Point of interface of the process and placements	What is committed on such points of interface	How does the committal occur		
Industrial partner	Dust	By letter or/and oral		
Chair of Thermal Processing Technology	Dust, records	By letter or/and oral		
Students	Dust, records	By letter or/and oral		
Archives (for the reports)	Report	By letter		
Industrial partner	Report	By letter or/and oral		
Account department	Report	By letter		

## 4.1.3.4 Analysis of the treats

Seven catchwords give food for thoughts about potential improvements, which are not known at the present. All of the seven catchwords stand for a word in the cause-effect diagram from Ishikawa. To remember these words, all begin with an "M" [13].

Management/	Cause:	No plan of use
Money Power	Effect:	Calendar of the present month to write in
Machine	Cause:	No annotation of the axis
	Effect:	Automatic annotation
Material	Cause:	The artificial drying is not documented
(includes raw		
material)	Effect:	Documentation
Man power	Cause:	No frequent meeting
		One meeting per year for all persons, who are
	Effect:	involved in the process
Measurement	Cause:	The apparatus is moistly after the cleaning
(inspection)	Effect:	Double check if the apparatus is really dry
Method	Cause:	The igniter is installed incorrectly
(process)	Effect:	Take care by the instalment
Mother nature	Cause:	Local exhausted ventilation
(Environment)	Effect:	Complete removal by suction

#### Table XII: Analysis of the treats

# 4.1.4 Identification and classification of the potential improvement

The potential improvements have to be identified from the viewpoint of the process to detect the so-called "low hanging fruits" (easily identifying potential improvements, which can be realized quickly). Improvements which belong to the whole process have to be discussed with the process leader [13]. The potential improvements have to be classified as follows:

importance of the process	1 (low) $\rightarrow$ 4 (high)
effort of the implementation	1 (low) $\rightarrow$ 4 (high)

Table XIII: Classification of the improvements

Name of the ma	ne process: Investigation of aximum rate of pressure r	on the maximum explo ise	sion overpressure
Number	Room for improvement	Importance for the process	Effort for the implementation
1	Use-plan for apparatus	2	1
2	Software update (annotation of the axes)	1	3
3	Exhaust hoot over the whole apparatus	2	4
4	Feedback for the lab personnel	3	1

5	Improvements for the use of the apparatus for gas investigations	4	4
	tor guo inveoligationo		

The classification of the improvements can be seen in the following figure, showing a way to identify the so-called "low hanging fruits". These easy improvements are in the bottom right corner.



Figure 28: Low hanging fruits

# 4.1.5 Future state of the process

Actual sate of the process = future state of the process

# 4.1.6 Determination of the process objectives and the used indicators

A clear definition of the process objectives helps to control and navigate a process. The goals should be deduced on the one hand from the strategic objectives of the organization ("top down") and on the other hand from the experience und the know-how of the staff ("bottom up") [13].

Objective	Indicator	Target value	Measurement method´s	Measurement frequency	Responsibility
High delivery reliability	Amount of the punctual deliveries	>98%	Count of all deliveries	Once a year	Head of laboratory
High customer satisfaction	Count of a good feedback	>95%	Conversation with customer	Once a year	Head of laboratory
Better effectiveness of the enrolment	Amount of the employees who pass the exam	>98%	Exam	After an enrolment	Head of laboratory

Table XIV: Determination of the process objectives

# 4.1.7 Definition of the reporting for process performance

For the reporting of the process performance it has to be identified which information regarding the process objectives have to pass on and when. It has to be taken into account that the way in which the reporting happens has to be clearly and easily [13].

# Table XV: Reporting of the performance

Which information?	By whom? To whom? In which way?	At which moment?
High delivery reliability: The investigations are done in the given time frame	Negative feedback regarding the delivery reliability from the customer is point in the next meeting of the lab personnel and the head of laboratory	If required
High customer satisfaction	Negative feedback from the customer has to be discussed in the next meeting of the lab personnel and the head of laboratory	If required
Effectiveness of the enrolment, shown by good marks at the exams	The examiner has to talk to the person which enrol the new employees	After exam

# 4.1.8 Realization of the potential improvements

With this step the way of implementation to reach the future process is defined [13].

|--|

Activity	Responsibility	Appointment	State
Use-plan for	Lab personnel	From now on	Open
apparatus			
Software	Producer of the	If possible	Open
update	machine		
(annotation of			
the axes)			
Exhausted hoot	Head of	If possible	Open
over the hole	laboratory		
apparatus			
Feedback for	Personnel	From now on	Done
the lab			
personnel			
Improvements	Head of	If possible	In the works
for the possible	laboratory,		
use of the	head of the		
apparatus for	organisation		
gas			
investigations			

# 4.2 Process description of MIE

# 4.2.1 Identification of the process

The first step of process-management is the identification and distinction of the processes. Therefore a distinct name for the process is needed. The fist and the last step of the process, as well as the input and output define the process [13].

Name of the process	Investigation on minimum ignition energy
Purpose	The purpose of the process is the
Fulpose	determination of the minimum ignition
	energy. This comports to the turnover of
	the organisation
Main customor	Industry partner
Main customer	Chair of Thermal Processing Technology
Prospect of the	Chair of Therman Tocessing Technology
customer	Results with constant quality
Output	Statistic minimum ignition energy value
	Report
Input	Special concentration of dust
	Energy level of the ignition spark
First step of the process	
	Start-up operation
Last step of the process	Drint roport
Interface input	
interface input	Chair of Thormal Processing Technology
Interface output	Industry partner
	Archive (reports)
	Accounting
Required resources	
Required resources	Head of Jaboratory
	Project manager
Information know-bow	Enrolment
	Submission of report
Working area	
	Laboratory
Equipment	Electricity
	Vacuum pump
	Dust
	MIKE 3
	Synthetic air
	Intranet
	Personal computer
	Scale
	Personal protective equipment (PPE)

Table XVII: Identification of the process

Factor of success	Clear procedure Communication Documentation takeover
Applicable documents	Standard EN 13821 Manual Data base Gestis StaubEx Know-how Technical appendix

# 4.2.2 Actual state of the process

The actual state is the description of the present state of the process. Therefore the process is described like the lab personnel conducts the working steps. The following figure shows the sequence of the tasks of the process along the vertical axes. The description of the process has to be made in a way that every person who does not know the process will be able to perform it. This should be obvious and understandable [13].





Master's thesis Katja Hüttenbrenner





Master's thesis Katja Hüttenbrenner



Master's thesis Katja Hüttenbrenner

# Table XVIII: Actual state of the process

Nr	Element	Explanation
1	Instruction	
	delivered	The needed information are delivered to the lab personnel
2	Consultation	consulted
	Defined	The lab personnel has to decide if the instruction she/he got is
3	enough?	enough or if she/he needs more information
4	Enrolment for	The head of the laboratory has to prove if the lab personnel has had
	MIKE 3 done?	an enrolment
5	the personnel	done
6	Particle size	
0	analyses	A particle size analyses has to be done
7	Dust particle	With the help of a particle size analysis it has to be decided if the dust
	<63µm	particles are smaller than 63µm
8	Dust milling	The material has to be milled to reach a size of less than 63µm
9	Dust drying	The dust has to be dried with the help of the compartment drier
10	Cool down the	
	dust	After the artificial drying the dust has to be cooled down
11	matter	The dust has to be weighed to find out the dry matter
40	Switch on	
12	MIKE 3	The MIKE 3-apparatus has to be switched on
13	Switch on air	<u>-</u>
	Supply	The air supply has to be switched on
14	worksheet	A new worksheet has to be created with the help of the software
45	Switch on	
15	exhauster	The exhauster has to be switched on
	Weigh out	
16	needed dust	The concentration for the trail has to be weighed out
	Insert the dust	
17	in MIKE 3	
	apparatus	The needed dust has to be brought in the storage chamber
18	Close fasteners	The chamber of the dust has to be closed
10	Measure	The gap between the two electrodes has to be measured with the
19	electrode gap	help of the limit gauge
20	Insert high	<u>-</u> ,,,,,,
	Voltage plug	The high voltage plug (the red one) has to be plugged in
21	door	The safety door has to be closed
	Adjust	
22	concentration	The concentration of the dust has to be edited in the areas
	or the dust	The concentration of the dust has to be adjusted in the program

23	Adjust energy level of the spark	The energy level of the spark has to be adjusted in the program	
24	Start trial	The trial has to be started with the start-button in the program	
25	Observe trial	The trial has to be observed to find out if the dust ignites	
26	Ignition occurred?	The lab personnel has to say if the dust has ignited	
27	Open safety door	n safety The safety door has to be opened	
28	Test for no ignition	The trial has to went on if no ignition occurred	
29	Unplug the high voltage plug	The high voltage plug (the red one) has to be unplugged	
30	Ignition occurred?	The lab personnel has to say if the dust has ignited	
31	Clean MIKE 3 apparatus	The MIKE 3-apparatus has to be cleaned	
32	Trialled ten times?	To reach the European standard for testing the MIE tests have to be made until ten times no ignition occurred	
33	Are all needed concentrations and energy levels tested?	With the help of the European standard and the manual of MIKE 3 all needed concentrations of the dust and all needed energy levels can be found out	
34	Open safety door	The safety door has to be opened	
35	Unplug the high voltage plug	The high voltage plug (the red one) has to be unplugged	
36	Clean MIKE 3	The MIKE 3-apparatus has to be cleaned	
37	Switch off exhauster	The exhauster has to be switched off	
38	Switch off air supply	The air supply has to be switched off and the conductions have to be depleted from air	
39	Print and save report	The report has to be saved in the program and also printed	
40	Switch off MIKE 3	The MIKE 3-apparatus has to be switched off	

# 4.2.3 Analysis of the process

# 4.2.3.1 Moments of truth

According to Wagner the moments of truth are at any time when the customer gets in contact with the organization or with the product. For the customer the process is a serial of meetings with the performance of the organization. These moments of truth are important, because they form the impression of the customer for the product or the organization and therefore they are crucial to the organization [13].

## What happens at the first contact of the customer with the organization?

Normally the first contact is a phone call where the customer describes his problem and gets information form the organization about the price of the testings, the needed time and the needed dust quantity.

## What happens when the service/product gets used the first time?

The customer gets a quotation from the organization. Also a detail time frame for the investigations and the exactly needed dust quantity will be discussed.

## Which information gets the customer from the organization?

Information about the used procedure, the time frame of the investigation and the price of the service. This information is committed clearly und adequately.

#### Which impression gets the customer from the employee?

The employees of the organization make a good and competent impression to the customer

# In which way experiences the customer the organization in case of complaining?

Those responsible take the complaining serious and deal with it in the right way to improve the process

## 4.2.3.2 Voice of the customer

The expectations of the customer and the actually fulfilled expectations have to be found out. Based on the identification of the process the customers of the organization are known and so the expectations can be identified. The measurement can be made by operation numbers or by interviewing the customer. Both qualitative and quantitative statements can furnish clues [13].

Voice of the customer	Problems in the process	Requirements of the process
Investigations are not done on schedule	Too less human resources	Sufficient human resources
Documentation is inadequate	The submission of the report is not changeable	A model report is available
Documentation is inadequate	The submission of the report is not changeable	A model report is available

## Table XIX: Voice of the customer
Voice of the customer	Problems in the process	Requirements of the process
I am not used to the procedure	Inadequate enrolment	Periodic enrolment

#### 4.2.3.3 Interfaces

In the context of interfaces, processes with interfaces have to be found out. Therefore it is essential to know what (data and information) and in which way (by letter, oral, electronically) it is delivered [13].

#### Table XX: Interfaces

Name of the process: Investigation on minimum ignition energy					
Point of interface to processes and placements	What is committed on such points of interface	How does the committal occur			
Industrial partner	Dust	By letter or/and oral			
Chair of Thermal	Dust, records	By letter or/and oral			
Processing Technology					
Students	Dust, records	By letter or/and oral			
Archives (for the	Report	By letter			
reports)					
Industrial partner	Report	By letter or/and oral			
Account department	Report	By letter			

#### 4.2.3.4 Analysis of treats

Seven catchwords give food for thoughts about potential improvements, which are not known at the present. All of the seven catchwords stand for a word in the cause-effect diagram from Ishikawa. To remember these words, all begin with an "M" [13].

Management/	Cause:	No plan of use
Money Power	Effect:	Calendar of the present month to write in
Machine	Cause:	No annotation of the axis
	Effect:	Automatic annotation
Material	Cause:	The artificial drying is not documented
(includes raw material)	Effect:	Documentation

Man power	Cause:	: No frequent meeting				
		One meeting per year for all persons, who are				
	Effect:	involved in the process				
Measurement	Cause:	Not cleaned				
(inspection)		Double check if the apparatus is really cleaned				
	Effect:	enough				
Method	Cause:	Distance between the two electrodes is inaccurate				
(process)	Effect:	Check after every cleaning				
Mother nature	Cause:	Local exhausted ventilation				
(Environment)	Effect:	Complete removal by suction				

#### 4.2.3.5 Identification and classification of the potential improvement

The potential improvements have to be identified from the viewpoint of the process to detect the so-called "low hanging fruits" (easily identifying potential improvements, which can be realized quickly). Improvements which belong to the whole process have to be discussed with the process leader [13]. The potential improvements have to be classified as follows:

importance of the process	1 (low) $\rightarrow$ 4 (high)
effort of the implementation	1 (low) $\rightarrow$ 4 (high)

Name of the process: Investigation on minimum ignition energy					
Number	Room for improvement	Importance for the process	Effort for the implementation		
1	Use-plan for the apparatus	2	1		
2	Software update (annotation of the axes)	1	3		
3	Automatic exhausted hoot	2	4		
4	Feedback for the lab personnel	3	1		
5	Permanent spark	2	4		
6	Optical recognition of the ignition	4	4		

Table XXII: Classification of the improvements

The classification of the improvements can be seen in the following figure, showing a way to identify the so-called "low hanging fruits". These easy improvements are in the bottom right corner.



Figure 29: Low hanging fruits

#### 4.2.4 Future state of the process

Actual state of the process = future state of the process

#### 4.2.5 Determination of the process objectives and the used indicators

A clear definition of the process objectives helps to control and navigate a process. The goals should be deduced on the one hand from the strategic objectives of the organization ("top down") and on the other hand from the experience und the know-how of the staff ("bottom up") [13].

Objective	Indicator	Target value	Measurement method´s	Measurement frequency	Responsibility
High delivery reliability	Amount of the punctual deliveries	>98%	Count of all deliveries	Once a year	Head of laboratory
High customer satisfaction	Count of a good feedback	>95%	Conversation with customer	Once a year	Head of laboratory

Table XXIII: Determination of the process objectives

Objective	Indicator	Target value	Measurement method´s	Measurement frequency	Responsibility
Better effectiveness of the enrolment	Amount of the employees who pass the exam	>98%	Exam	After an exam	Head of laboratory

#### 4.2.6 Definition of the reporting for process performance

For the reporting of the process performance it has to be identified which information regarding the process objectives have to pass on and when. It has to be taken into account that the way in which the reporting happens has to be clearly and easily [13].

Which information?	By whom? To whom? In which way?	At which moment?
High delivery reliability: The investigations are done in the given time frame	Negative feedback regarding the delivery reliability from the customer is point in the next meeting of the lab personnel and the head of laboratory	If required
High customer satisfaction	Negative feedback from the customer has to be discussed in the next meeting of the lab personnel and the head of laboratory	If required
Effectiveness of the enrolment, shown by good marks at the exams	The examiner has to talk to the person which enrol the new employees	After exam

#### Table XXIV: Reporting of performance

#### 4.2.6.1 Realization of the potential improvements

With this step the way of implementation to reach the future process is defined [13].

Activity	Responsibility	Appointment	State
Use-plan for apparatus	Lab personnel	From now on	Open
Software update (annotation of the axes)	Producer of the machine	If possible	Open
Automatic exhausted hoot	Head of laboratory	If possible	Open
Feedback	Personnel	From now on	Done
Permanent spark	Head of laboratory, head of the organisation	If possible	In the works
Optical recognition of the ignition	Head of laboratory, head of the organisation	If possible	Open

#### Table XXV: Realization of the improvements

### **5** Material characteristics

Various possible carbon carriers were tested for explosibility to find out the safest alternative for the RecoPhos-project. There is an impact of volatility on explosibility - the more volatile compounds the carbon carriers contain, the higher is the explosiveness. Therefore various potential carbon carriers were tested to find the best fitting mixture for the RecoPhos process. The carbon sources tested will be described in this chapter. Also the results of the tests of the explosibility will be discussed.

#### 5.1 Coal

Coal is an organic rock, which is mainly used as a fossil fuel for combustion. It is sedimentary and the main collocations are carbon, hydrogen and oxygen. Tectonic movements were the reasons for the formation of coal. Vegetation had been consolidated between rocks and with the burial of this compound the plant material underwent high temperatures and high pressures. These extreme conditions caused changes in the structure and the physical and chemical properties of the material and so a transformation to peat and later to coal began. The temperature, the pressure and also the length of the time in formation (the so called "organic maturity") are attributes for the quality of the coal. After a short period of formation time, peat converts into lignite or "brown coal". This coal is characterized by a low organic maturity and are quite soft. The range of colours moves from dark black to various shades of brown. More time of transformation and the continuous presence of temperature and pressure leads to an increase of organic maturity. This state of the transforming produces "subbituminous" coals. Persistent temperature and pressure cause further transformations and so the coal becomes harder and blacker. "Bituminous" or "hard coal" is formed. If the transformation process continues and the right conditions are present, the organic maturity reaches the maximum and anthracite is formed. The physical and chemical properties categorise the coal into various ranks. A low rank means that these coals are softer and friable and the appearance is dull and earthy. The high moisture level and the low carbon content leads to a low energy content. An example of these coals are lignite or sub-bituminous coal. A hard and strong structure means that the rank is much higher. A high rank coal contains more carbon and has a black, vitreous lustre. The energy content is high because the level of moisture is low and the carbon content is high. Hard coal is an example and anthracite is at the top of the scale [14]. **Table XXVI** shows the results of a chemical analysis of dry and ash-free coals.

Ranks & Localities	Humidity	Chemical						
of analysed coal	[%]	analysis on						
		dry, ash-free						
		basis						
		Proximate	Ultimate					
		analysis	analyses					
		[%]	[%]	-			-	-
		Volatile	Fixed	С	Н	N	S	0
		matter	carbon (calculated)	[%]	[%]	[%]	[%]	[%]
Peat (Ireland)	90	65	35	58.2	5.2	1.5	0.5	34.6
Brown Lignite	40	55	45	66.6	5.0	1.6	3.2	23.6
(Sile, Istanbul,								
Türkiye)								
Lignite (Seyitomer,	35	42	58	69.8	5.0	1.0	2.0	22.2
Central Anatolia,								
Türkiye)								
Black Lignite	20	40	60	70	5.3	2.3	1.9	20.5
(Soma, Western								
Anatolia, Türkiye)								
Black Bituminous	15	38	62	79	5.4	0.7	3.5	11.4
Coal (Gediz,								
Western Anatolia,								
Türkiye)								
Black Bituminous	10	30	70	84	5.2	1.2	0.6	9.0
Coal (Zonguldak,								
Northern Anatolia,								
Türkiye)								
Anthracite (South	2	7	93	92.4	3.0	0.7	0.5	3.4
Wales)								

 Table XXVI: Chemical analyses on dry and ash-free coals [14]

#### 5.1.1 Brown coal

A large range of rocks are known under the term brown coal. Both the brownish hard and soft coals and the black and earthy-brown bituminous coals belong to the group of brown coals. Thanks to the evolution of fossil plants, remaining pieces of plants can be identified in such coals. Petrographic investigations show that these coals have no homogeneous substance, so categorical attributes are very difficult to define. Calorific values and therefore the heating value are lower than the values of hard coals. Heating values of the average coal can be seen in **Table XXVII**, every value referred to an ash and water free substance. Based on the composition shown in **Table XXVII** it can be seen that the concentration of oxygen falls with the grade of coalification, while the concentration of carbon increases [15].

	Occurrence	C [%]	H [%]	O, N, S [%]	Higher calorific value [kcal/kg]
Wood		50	6	44	4600
Peat	North Germany	59	6	35	5600
Bituminous coal	Hungary	75.5	6	18.5	7800
Pitch coal	West Germany	73	5.5	21.5	7550
	Czechoslovakia	77	6	17	8000
Bright coal	Austria	75	5	20	7600
	Czechoslovakia	78	5.5	16.5	7750

Table XXVII: Brown coal [15] (slightly modified)

Besides water, brown coals have a great part of ash-forming mineral matter. This matter does not count as part of the mineral components, even if the content of ash varies between 1 and 30% and up to 40% for low-quality coals. The ash of these coals are lime-based and sulphate-based, in contrast to hard coal, based on the high rate of lime in brown coals. The content of bitumen, soluble humic acid, lignite and cellulose characterises this sort of coal. To determine the composition of brown coals, the bituminous part can be identified by an extraction with benzene-alcohol. The bituminous part is made up of resin and cere and ranges between 7 and 31%, but brown coals with a bituminous content as low as 3.5% are known. It

is important to know the percentage of bitumin, because tar formation is caused by bitumen. The higher the content of bitumen, the higher the tar formation. This rule only applies for brown coals, not for hard coals, because of the thermal decomposition, starting at a temperature under 200°C for brown coal. Even the humic part can produce some tar. The content of the humic acid range between 3 and 93% and it can be said that coals with a content of more than 10% are brown coals. The minerals Al-, Si-, and Fe- compounds are known, but also Mg, K, Na and even Mg occur in coals [15].



Figure 30: Brown coal

#### 5.1.1.1 Briquetting of brown coal

Briquetting was developed to combine various characteristics of different coals; the briquettes can be formed with or without binder. "The binder helps in agglomeration and also gives cohesive strength to briquettes" [14]. Another positive side effect is the improvement of properties, for example to maintain of ignitability, the lowering of volatility, the reduction of smoke and of sulphur emissions. Because of the high content of moisture and a very low calorific value, brown coal has a very low commercial value. To produce a more attractive commercial product, brown coal has to be briquetted. As described, it is possible to form briquettes without a binder, a popular way to form briquettes out of brown coal. To achieve success, high pressure and the preparation of the coal are important. Therefore a press, originally designed by Exter for the manufacturing of peat briquettes in Ireland, is used [14]. For the investigations of brown coal, described in this thesis, briquettes without a binder were used.

#### 5.1.1.2 Investigations on pure brown coal - maximum explosion overpressure $p_{MAX}$ , maximum rate of pressure rise (dp/dt)<sub>MAX</sub> and K<sub>ST</sub>-value

The concentration of the brown coal dust varied from 30 g/m<sup>3</sup> to 2250 g/m<sup>3</sup>, with maximum values of the explosion between 500 g/m<sup>3</sup> and 1000 g/m<sup>3</sup>. The maximum explosion overpressure  $p_{MAX}$  of the tested brown coal dust is 7.6 bar and a maximum rate of pressure rise (dp/dt)<sub>MAX</sub> is 475 bar/s. So the K<sub>ST</sub>-value is 129 m\*bar/s, thus brown coal dust has a dust explosion class 1. The trend of the explosion can be seen in **Figure 31** and the results of the tests can be taken from **Table XXVIII**.

Series	Concentration [g/m³]	Maximum explosion overpressure	Maximum rate of	Ignition delay time [ms]	Chemical igniter energy
		Вмах	rise	[mo]	chergy
		[bar]	(dp/dt) <sub>MAX</sub>		
1	30	0	0	60	10kJ
1	60	0.7	13	60	10kJ
1	125	5.2	102	60	10kJ
1	250	6.9	374	60	10kJ
1	500	7.6	444	60	10kJ
1	750	7.4	465	60	10kJ
1	1000	6.7	391	60	10kJ
1	1250	6.3	240	60	10kJ
1	1500	5.8	283	60	10kJ
1	1750	5.4	217	60	10kJ
1	2000	4.4	230	60	10kJ
1	2250	4.4	224	60	10kJ
2	125	4.7	84	60	10kJ
2	250	6.1	238	60	10kJ
2	500	7.5	409	60	10kJ
2	750	7.1	462	60	10kJ

Table XXVIII: Results: brown coal

2	1000	6.7	485	60	10kJ
2	1250	5.9	277	60	10kJ
2	1500	5.4	207	60	10kJ



Figure 31: Brown coal

#### 5.1.1.3 Investigations on pure brown coal - minimum ignition energy (MIE)

The next step to get an idea of the explosibility of brown coal dust, the minimum ignition energy (MIE), investigated with the MIKE 3-appartus from Kühner AG, is required. The ignition delay time was adjusted to 120 ms and the inductance to 1 mH. Because the dust ignited with an ignition energy of 1000 mJ, also 300 mJ, 100 mJ and also 30 mJ were tested. The dust ignited at the energy steps of 1000 mJ, 300 mJ and 100 mJ with a concentration range of 300 mg to 3600 mg. With an ignition energy of 30 mJ the tests were made with concentrations of 1500 mg, 1800 mg and 2400 mg and there was no ignition, so an ignition energy of 30 mJ is too low to ignite a brown coal dust. The course of the explosion is shown in **Figure 32** and the results can be seen in **Table XXIX**.

Series	Concentration [mg]	Ignition energy	lgnition delay time set [ms]	Effective ignition delay time [ms]	Induction	Ignition	Number of the test, when the ignition occurred
1	300	1000 mJ	120	122	1 mH	NO	
2	600	1000 mJ	120	122	1 mH	YES	1
3	900	1000 mJ	120	122	1 mH	YES	1
4	1200	1000 mJ	120	122	1 mH	YES	1
5	1500	1000 mJ	120	122	1 mH	YES	1
6	1800	1000 mJ	120	123	1 mH	YES	1
7	2400	1000 mJ	120	122	1 mH	YES	2
8	3000	1000 mJ	120	122	1 mH	YES	2
9	3600	1000 mJ	120	123	1 mH	YES	1
	600	200 m l	100	101	1	NO	
10	600	300 mJ	120	121	1 mH	NU	
11	900	300 mJ	120	121	1 mH	YES	1
12	1200	300 mJ	120	121	1 mH	YES	4

Table XXIX: Results MIE – brown coal

MATERIAL CHARACTERISTICS

Series	Concentration [mg]	Ignition energy	Ignition delay time set [ms]	Effective ignition delay time [ms]	Induction	Ignition	Number of the test, when the ignition occurred
 13	1500	300 mJ	120	121	1 mH	YES	1
 14	1800	300 mJ	120	121	1 mH	YES	4
 15	2400	300 mJ	120	122	1 mH	YES	1
 16	3000	300 mJ	120	122	1 mH	YES	2
 17	3600	300 mJ	120	121	1 mH	YES	3
 18	900	100 mJ	120	121	1 mH	NO	<u> </u>
 19	1200	100 mJ	120	121	1 mH	NO	
 20	1500	100 mJ	120	121	1 mH	YES	4
 21	1800	100 mJ	120	121	1 mH	YES	4
 22	2400	100 mJ	120	121	1 mH	YES	6
 23	3000	100 mJ	120	121	1 mH	NO	
 24	1500	30 mJ	120	121	1 mH	NO	
 25	1800	30 mJ	120	121	1 mH	NO	
 26	2400	30 mJ	120	121	1 mH	NO	



Figure 32: MIE - brown coal

"The minimum ignition energy MIE lies between the lowest energy value (E2) at which ignition occurred and the energy (E1) at which in at least 10 successive experiments no ignition was observed" [12]. To find out this energy **Figure 32** has to be consulted. The unfilled circle means that there was no ignition in at least ten successive experiments. In contrast, the filled square means that there has been an ignition. The red "T" above some squares show that the ignition did not occurred at the first test.

"For the purpose of comparison between different apparatuses, only one statistic MIE value  $(E_s)$  instead of the energy range (E1, E2) should be used. This single value  $(E_s)$  can be estimated by the use of the probability of ignition as follows:

$$Es = 10^{\wedge} \left( \log E2 - \frac{I[E2]^* (\log E2 - \log E1)}{(NI + I)[E2] + 1} \right)$$
(4-1)

Where: I[E2] = number of tests with ignition at the energy E2.

(NI+I)[E2] = total number of tests at the energy E2" [12]

	IE \ mg	900	1200	1500	1800	2400	3000		Probability
E2 =	100 mJ	NI	NI	I	Ι	I	NI	$\rightarrow$	3 of 6
E1 =	30 mJ			NI	NI	NI			

I = ignition of dust

NI = no ignition of dust in 10 trials

This means that the minimum ignition energy MIE lies between 30 mJ and 100 mJ. The statistic MIE value for the minimum ignition energy is calculated with the formula (4-1).

E1 = 30 mJ E2 = 100 mJ I[E2] = 3 (NI+I)[E2] = 6

$$Es = 10^{\wedge} \left( \log 100 - \frac{3 * (\log 100 - \log 30)}{6 + 1} \right)$$
(4-2)

So the calculated statistic MIE value ( $E_s$ ) is 59.69 mJ. The results from the software of MIKE 3 can be seen in **Figure 33**.



Figure 33: Results from the software

#### 5.1.2 Charcoal

For the most part the porous material charcoal contains fixed carbon, ash and volatile components. These are oxygen and hydrogen, which are chemically bounded. Also some tar is possible. If there is an increase of temperature, these volatile components can escape as hydrogen, carbon monoxide, carbon dioxide and higher-valence hydrocarbons. One characteristic of charcoal is the high possibility of the water content. So the "European Standard DIN EN 1860-2:2005 Appliances, solid fuels and firelighters for barbecuing – Part 2: Barbecue charcoal and barbecue charcoal briquettes – Requirements and test methods" says that the moisture content has to be lower than 8% for barbecue charcoal and barbecue charcoal briquettes. An important quality criterion of charcoal is the fixed carbon, which differs for the various purposes. The source of the material affects the specific gravity and the ash content. It is difficult to say anything about the energetic efficiency, because this value has a high fluctuation, because of different base materials. The energetic efficiency lies around "one

third to two fifths" related to the lower heating value of the utilised solid fuel. The characteristics of charcoal in comparison to wood can be seen in **Figure 34** [16] [17].



Figure 34: Comparison wood - charcoal [16] (slightly modified)

To produce charcoal wood has to be heated under exclusion of air. The most common way to generate charcoal goes back to the Middle Ages: the coal piles. For this charcoal burning a so-called charcoal kiln has to be built. To form these kilns, big logs of wood have to be set up to form conical piles (kilns). In the middle of these kilns there are three pickets, providing the base on which the logs are arranged. Grass, earth and waste of coals are used to cover the outside of these kilns, so the air can be regulated to control the burning of the wood. Essentially just the gases which form from the process should be burned and the temperature should rise to reach to temperature of carbonising [16].



Figure 35: Charcoal

#### 5.1.2.1 Investigations on pure charcoal - maximum explosion overpressure $p_{MAX}$ , maximum rate of pressure rise (dp/dt)<sub>MAX</sub> and K<sub>ST</sub>-value

Dust from pure charcoal was tested. For the investigations on the maximum explosion overpressure and the maximum rate of pressure rise a concentration range of 30 g/m<sup>3</sup> to 2250 g/m<sup>3</sup> was used. The maximum of the charcoal dust was between 500 g/m<sup>3</sup> and 1000 g/m<sup>3</sup>. So the maximum explosion overpressure  $p_{MAX}$  of the tested charcoal dust is 7.6 bar and a maximum rate of pressure rise (dp/dt)<sub>MAX</sub> is 475 bar/s. The K<sub>ST</sub>–value, calculated by the (dp/dt)<sub>MAX</sub>-value, is 129 m\*bar/s. From this it can be seen that charcoal dust has a dust explosion class 1. The course of the explosion can be seen in **Figure 36** and the results of the tests can be taken out from **Table XXX**.

Series	Concentration [g/m³]	Maximum explosion overpressure р <sub>мах</sub> [bar]	Maximum rate of pressure rise (dp/dt) <sub>MAX</sub> [bar/s]	lgnition delay time [ms]	Chemical igniter energy
1	60	0	0	60	10kJ
1	125	0	0	60	10kJ
1	250	7.2	268	60	10kJ
1	500	7.3	377	60	10kJ
1	750	6.9	453	60	10kJ
1	1000	6.1	249	60	10kJ
1	1250	5.6	149	60	10kJ
2	125	0	0	60	10kJ
2	250	6.7	169	60	10kJ
2	500	7.4	384	60	10kJ
2	750	6.9	509	60	10kJ
2	1000	6.2	246	60	10kJ

Table XXX: Results: charcoal





#### 5.1.2.2 Investigations on pure charcoal - minimum ignition energy (MIE)

Investigations in the MIKE 3-apparatus for detecting the minimum ignition energy were made with pure charcoal dust. The tests were made with a concentration range of 300 mg up to 1500 mg. Because no concentration ignited with a spark energy of 1000 mJ, tests with a lower spark energy were not necessary. The testing result was that charcoal dust of 50  $\mu$ m has a MIE above 1000 mJ. The results from the MIKE 3 tests can be taken from **Table XXXI** and the course can be seen in **Figure 37**.

Table XXXI:	MIE -	charcoal
-------------	-------	----------

Concentration [mg]	Ignition energy	Ignition delay time set [ms]	Effective ignition delay time [ms]	Induction	Ignition
300	1000 mJ	120	121	1 mH	NO
600	1000 mJ	120	120	1 mH	NO
900	1000 mJ	120	120	1 mH	NO
1200	1000 mJ	120	120	1 mH	NO
1500	1000 mJ	120	121	1 mH	NO



Figure 37: MIE - charcoal

#### 5.1.3 Hard coal

Hard coals were produced from land plants and marsh plants at the flood zone. Caused by the exclusion of air, mouldering of the dead plants was not possible and so the coalification started. This process has two steps. The first step is characterised by the decomposition processes and transformation processes by the microorganisms. Peat and brown coal is the result. The next step proceeds under more temperature and more pressure with the result of a tighter coal, the hard coal. A mixture of shiny and matt stripes is a typical sign of hard coals because these types of coal are not homogenous. Hard coals consist of various constituents. These depends on the coalification process. In general it can be said that the shiny stripes have a structure of wood cells and the matt parts of the coals have unlignified tissues, for example spores. The calorific value of hard coal rises with the coalification grade. As a consequence various hard coals exist. To describe the coalification grade the ratio between the carbon atoms and the hydrogen atoms is used. The chemical structure is not yet resolved. Most coals consist of ring-shaped hydrocarbons (aromatics) and, for a small part, of aliphates (linear hydrocarbon compounds) [18].



Figure 38: Hard coal

# 5.1.3.1 Investigations on pure hard coal - maximum explosion overpressure $p_{MAX}$ , maximum rate of pressure rise (dp/dt)<sub>MAX</sub> and K<sub>ST</sub>-value

Pure hard coal was the first coal dust investigated. The concentration ranged from 60 g/m<sup>3</sup> to 1250 g/m<sup>3</sup>, with a maximum pressure between 250 g/m<sup>3</sup> and 500 g/m<sup>3</sup>. The tests showed that hard coal has a maximum explosion overpressure  $p_{MAX}$  of 7.6 bar and a maximum rate of pressure rise (dp/dt)<sub>MAX</sub> of 510 bar/s. The K<sub>ST</sub>–value can be calculated to be 139 m\*bar/s, thus hard coal belongs to the dust explosion class 1. The course of the explosion is shown in **Figure 39** and the results can be seen in **Table XXXII**.

Series	Concentration [g/m <sup>3</sup> ]	Maximum explosion overpressure	Maximum rate of pressure	Ignition delay time [ms]	Chemical igniter energy
		p <sub>MAX</sub> [bar]	rise (dp/dt) <sub>MAX</sub> [bar/s]		
1	60	1	29	60	10kJ
1	125	6.4	159	60	10kJ
1	250	7.7	323	60	10kJ
1	500	7.4	550	60	10kJ
1	750	6.7	371	60	10kJ
1	1000	5.6	235	60	10kJ
1	1250	5.6	331	60	10kJ
2	125	6.2	170	60	10kJ
2	250	7.5	272	60	10kJ
2	500	7.1	470	60	10kJ
2	750	6.2	367	60	10kJ
2	1000	4.9	166	60	10kJ

Table XXXII: Results: hard coal





#### 5.1.3.2 Investigations on pure hard coal - minimum ignition energy (MIE)

The next step to determine the explosibility of the hard coal dust, the minimum ignition energy (MIE), was investigated with the MIKE 3-appartus from Kühner AG. The test was made with an ignition delay time of 120 ms and ignition energy of 1000 mJ. All tests were made with an inductance of 1 mH. Pure hard coal dust did not ignite for ten times. Because it is not possible for the MIKE 3-apparatus to provide ignition energies above 1000 mJ the testing result is, that a dust of pure hard coal has a MIE above 1000 mJ. The course of the explosion is shown in **Figure 40** and the results can be seen in **Table XXXIII**.

Concentration [mg]	Ignition energy	Ignition delay time set [ms]	Effective ignition delay time [ms]	Induction	Ignition
300	1000 mJ	120	122	1 mH	NO
600	1000 mJ	120	122	1 mH	NO
900	1000 mJ	120	122	1 mH	NO
1200	1000 mJ	120	122	1 mH	NO
1500	1000 mJ	120	122	1 mH	NO

Table XXXIII: Results MIE - hard coal





#### 5.1.4 Anthracite

Anthracite is a lustrous hard black coal. In the ranking of coal, anthracite belongs to the highest rank, because of its high carbon content of more than 93%. The volatile matter is lower than 8% and it is not possible to carbonize anthracite coal. A special characteristic is that it emits no smoke when it burns. Therefore anthracite is the most preferable coal for domestic stoves and central heating installations. The caloric values range from 8000 Kcal/kg [14].

Characteristics of anthracites are a high porosity and a highly developed orientation, because the crystalline layers are parallel to the bedding plane. Anthracites are like non graphitable carbons in heat treatments up to 1600°C, becaused of the high porosity and the high number of interconnections. Heat-treatment at a temperature of 2500°C breaks these interconnections and so anthracites are graphitable [19].



Figure 41: Anthracite

# 5.1.4.1 Investigations on pure anthracite - maximum explosion overpressure $p_{MAX}$ , maximum rate of pressure rise (dp/dt)<sub>MAX</sub> and K<sub>ST</sub>-value

For the investigations of anthracite on the maximum explosion overpressure and the maximum rate of pressure rise, a concentration range of 500 g/m<sup>3</sup> to 2250 g/m<sup>3</sup> was used. Some problems happened at the first series. Tests with the concentration of 1500 g/m<sup>3</sup> and 1700 g/m<sup>3</sup> had to be done again because no explosion occurred the first time. The maximum explosion overpressure  $p_{MAX}$  of the tested anthracite dust is 4.5 bar. It has to be taken in

account that the divergence between the first and the second trial is 10.7%. The acceptable divergence is 10%. The maximum rate of pressure rise  $(dp/dt)_{MAX}$  from investigations in the 20-I-apparatus is 29 bar/s. The K<sub>ST</sub>-value is 8 m\*bar/s. As a result anthracite dust has a dust explosion class 1. The course of the explosion can be seen in **Figure 42** and the results of the tests can be seen in **Table XXXIV**.

Series	Concentration	Maximum	Maximum	Ignition	Chemical
	[g/m³]	explosion	rate of	delay time	igniter
		overpressure	pressure	[ms]	energy
		<b>P</b> MAX	rise	_	
		[bar]	(dp/dt) <sub>MAX</sub>		
			[bar/s]		
1	500	0	0	60	10kJ
				-	-
1	750	0	0	60	10kJ
				-	-
1	1000	0	0	60	10kJ
1	1250	3.6	22	60	10kJ
1	1500	4.0	10	60	10kJ
1	1750	3.3	28	60	10kJ
1	2000	2.2	18	60	10kJ
1	2250	0	0	60	10kJ
2	750	0	0	60	10kJ
2	1000	4.9	30	60	10kJ
2	1250	4.6	30	60	10kJ
2	1500	3.8	28	60	10kJ
2	1750	3.8	30	60	10kJ
2	2000	3.1	17	60	10kJ
2	2250	2.2	18	60	10kJ

#### Table XXXIV: Results: anthracite





#### 5.1.4.2 Investigations on pure anthracite - minimum ignition energy (MIE)

The next test with pure anthracite dust was made with the MIKE 3-apparatus from Kühner AG. The tests were made with an inductance of 1 mH to produce a time protracted spark. The anthracite dust did not ignite for ten times, so the result is that anthracite dust with a median of 50  $\mu$ m has a minimum ignition energy above 1000 mJ. The results of the software of MIKE 3-apparatus can be seen in **Figure 43** and the values of the tests are shown in **Table XXXV**.

Concentration [mg]	Ignition energy	Ignition delay time set [ms]	Effective ignition delay time [ms]	Induction	Ignition
300	1000 mJ	120	122	1 mH	NO
600	1000 mJ	120	122	1 mH	NO
900	1000 mJ	120	122	1 mH	NO
1200	1000 mJ	120	122	1 mH	NO
1500	1000 mJ	120	122	1 mH	NO

Table XXXV: Results MIE - anthracite



Figure 43: MIE - anthracite

#### 5.2 Graphite

Graphite is a mineral with a colour of iron black to steel grey and in transmitted light graphite appears deep blue. The chemical formula is "C" and the crystal-system is a hexagonal system. According to the Mohs-scale graphite has a hardness of 1 to 2. Graphite occurs in metamorphosed rocks, caused by the sedimentary carbonaceous material, and by the reduction of carbon compounds [20].

The excellent electrical and thermal conductivity is a well-known characteristic of graphite. Furthermore, this mineral has a variety of other properties, such as chemical influence resistance. Moreover graphite has a good resistance to high temperatures and also a moderate resistance to temperature changes. Therefore this material is used in industry for various processes. Under normal conditions graphite is not meltable and a sintering of the single particles is also not possible [19].



Figure 44: Graphite

There are three forms of graphite known: The nearly ideal graphite, the artificial graphite and the pyrolytic graphite.

Natural graphite and heat-threated synthetic carbons (more than 3000°C) are called nearly ideal graphite. Heat-threated synthetic carbons need a very high temperature (more than 3000°C) and the raw materials must be very easily graphited [19].

The second form, the artificial graphite, includes all polycrystalline industrially formed graphites with a middle graphitization grad. They are a mixture of fine carbon material (anthracite powder, carbon black and so on) and organic binding material, such as bitumen or tar. Artificial graphites are high-purity graphites for metallurgically processes, reactor graphites and special electro graphites. These special graphites include impermeable graphite and hot-formed graphite for electrodes and carbon brushes [19].

Pyrolytic graphites, or pyrographites, are developed by the thermal decomposition of gaseous hydrocarbons onto a substrate surface. The heat treatment with temperatures over 2500°C graphitizes this material [19].

A high thermal conductivity is a well-known characteristic of graphite. Original material, conditions of the production, the gravity, the size of the crystallites and the orientation influence the thermal conductivity. Tests show that the heat flow like lines to the graphite layers [19].

#### 5.2.1 Graphitization

Graphitization is a process, which starts with a carbon-containing material and after a heattreatment graphite will be the result. The final state of this graphitization is a state of equilibrium [19].

The graphitizing process has 3 temperature steps: the first step is under 1600°C, the next step between 1600 and 2000°C and the last step takes place at temperatures higher than 2000°C. The first step – the pyrolysis – creates an aromatic ring structure, formed out of the carbon-containing material. The higher the temperature, the more hydrocarbons will go away and so a structure of parallel ring structures will form. This material is called "turbostratic crystallites" and is characterized by hexagonal arrangement of the C-atoms in the planes, but with no ordered sequence of the layers. The temperature between 1700 and 2500°C is the real temperature to provide a graphitization, but so-called "cross-linked structure" (graphite with the occurrence of high bonding between the single layers) will retard the graphitization up to 3000°C [19].

Basically the best material for graphitization will be fine granules with optimally thickening material. Carbon dust can be disturb the graphitization because of the collided character [19].

#### 5.2.2 Investigations on graphite dust

# 5.2.2.1 Maximum explosion overpressure $p_{MAX}$ , maximum rate of pressure rise $(dp/dt)_{MAX}$ and $K_{ST}$ -value

The investigation on graphite dust was made with the 20-I-apparatus to determine the maximum explosion overpressure and the maximum rate of pressure rise from a 50  $\mu$ m graphite dust. The concentration of the dust ranged from 500 g/m<sup>3</sup> to 2000 g/m<sup>3</sup>. There was a large fluctuation range between the first two series and because this fluctuation was too large, two new series had been done. The fluctuation between the next tests was smaller, so the results of the last two series count for this dust. The maximum explosion overpressure  $p_{MAX}$  is 1.4 bar, the maximum rate of pressure rise (dp/dt)<sub>MAX</sub> was 3 bar/s and the K<sub>ST</sub>-value was 1 bar\*m/s, thus the dust explosion class was 1. The results of the explosion tests can be seen in **Table XXXVI** and the course of the explosion is shown in **Figure 45**.

Series	Concentration [g/m³]	Maximum explosion overpressure р <sub>мах</sub> [bar]	Maximum rate of pressure rise (dp/dt) <sub>MAX</sub> [bar/s]	Ignition delay time [ms]	Chemical igniter energy
1	500	0	0	60	10 kJ
1	750	0	0	60	10 kJ
1	1000	0	0	60	10 kJ
1	1250	0	0	60	10 kJ
1	1500	1.5	2	60	10 kJ
1	1750	0	0	60	10 kJ
1	2000	0	0	60	10 kJ
2	1000	0.2	4	60	10 kJ
2	1250	1.3	1	60	10 kJ
2	1500	0	0	60	10 kJ

Table XXXVI: Results: graphite dust





#### 5.2.2.2 Minimum ignition energy (MIE)

The next parameter to define the explosibility of graphite dust, the minimum ignition energy (MIE), was investigated in the MIKE 3-appatatus from the Kühner AG. The concentration of the dust for the tests ranged from 300 mg to 1500 mg. The tests were made like the previous tests of the coal, but with graphite dust a special phenomenon occurred. During the tests electrostatic discharges were presents around of the electrodes. **Figure 46** shows three such discharges in the MIKE 3-apparatus. They can be seen in the green circle in the pictures near the right electrode.

Although it has been known, static electricity is still a frequent ignition source for powders and dusts. In the process industry, fires and explosions caused by electrostatic charges are a existing problem and such electrostatic charges represent a source of ignition which can be hazardous. Therefore the characterisation of the incendivity of discharges is an important field of study. According to Glor "about one dust explosion occurs each day in Germany and every tenth explosion is caused by static electricity" [21]. Electrostatic discharges do not necessarily ignite a dust cloud, the fatal combination is when a process produces both, dangerous high charge potentials and an explosive atmosphere. Special care should be taken when feeding a powder into silos, because the dust gets dispersed in the air. The dispersed dust accumulates varying degrees of electrostatic charge due to triboelectrification against the walls. High spacecharge densities can be the result and so an ignition can occur [22] [23] [24].

**Table XXXVII** to **Table XLI** show the testing procedure. It should be noted that these electrostatic discharges only occurred after the first ignition test. One explanation can be that the dust has to be charged up with the first test. After this charging the electrostatically charged dust congregated close to the surrounding of the electrodes and then the discharges occurred. Because of this phenomenon the investigations on graphite were not conclusive. The electrical conductivity is a well-known characteristic of graphite and therefore the electrostatic discharge was to be expected.



Figure 46: Electrostatic discharges

Concentration [mg]	Test number	Ignition energy	Ignition delay time set [ms]	Induction	Result
300	1	1000 mJ	120	1 mH	NO ignition
300	2	1000 mJ	120	1 mH	electrostatic discharge
300	3	1000 mJ	120	1 mH	NO ignition
300	4	1000 mJ	120	1 mH	NO ignition
300	5	1000 mJ	120	1 mH	electrostatic discharge
300	6	1000 mJ	120	1 mH	NO ignition
300	7	1000 mJ	120	1 mH	NO ignition
300	8	1000 mJ	120	1 mH	NO ignition
300	9	1000 mJ	120	1 mH	electrostatic discharge
300	10	1000 mJ	120	1 mH	NO ignition

Concentration [mg]	Test number	Ignition energy	Ignition delay time set	Induction	Result
			[ms]		
600	1	1000 mJ	120	1 mH	NO ignition
600	2	1000 mJ	120	1 mH	NO ignition
600	3	1000 mJ	120	1 mH	NO ignition
600	4	1000 mJ	120	1 mH	NO ignition
600	5	1000 mJ	120	1 mH	NO ignition
600	6	1000 mJ	120	1 mH	NO ignition
600	7	1000 mJ	120	1 mH	NO ignition
600	8	1000 mJ	120	1 mH	NO ignition
600	9	1000 mJ	120	1 mH	NO ignition
600	10	1000 mJ	120	1 mH	NO ignition

#### Table XXXVIII: Graphite 600 mg

#### Table XXXIX: Graphite 900 mg

Concentration [mg]	Test number	Ignition energy	Ignition delay time set	Induction	Result
900	1	1000 mJ	[ms] 120	1 mH	NO ignition
					0
900	2	1000 mJ	120	1 mH	NO ignition
900	3	1000 mJ	120	1 mH	NO ignition
900	4	1000 mJ	120	1 mH	NO ignition
900	5	1000 mJ	120	1 mH	NO ignition
900	6	1000 mJ	120	1 mH	NO ignition
900	7	1000 mJ	120	1 mH	NO ignition
900	8	1000 mJ	120	1 mH	NO ignition
900	9	1000 mJ	120	1 mH	NO ignition
900	10	1000 mJ	120	1 mH	electrostatic discharge

Concentration [mg]	Test number	Ignition energy	lgnition delay time	Induction	Result
			set [ms]		
1200	1	1000 mJ	120	1 mH	NO ignition
1200	2	1000 mJ	120	1 mH	electrostatic discharge
1200	error	1000 mJ	120	1 mH	electrostatic discharge, error
	Cleaning				
1200	3	1000 mJ	120	1 mH	NO ignition
1200	error	1000 mJ	120	1 mH	error
1200	error	1000 mJ	120	1 mH	electrostatic discharge, error
	Cleaning				
1200	4	1000 mJ	120	1 mH	NO ignition
1200	5	1000 mJ	120	1 mH	NO ignition
1200	error	1000 mJ	120	1 mH	electrostatic discharge, error
1200	6	1000 mJ	120	1 mH	NO ignition
	Cleaning				
1200	7	1000 mJ	120	1 mH	NO ignition
1200	error	1000 mJ	120	1 mH	error
	Cleaning				
1200	8	1000 mJ	120	1 mH	NO ignition
1200	9	1000 mJ	120	1 mH	NO ignition
1200	10	1000 mJ	120	1 mH	NO ignition

#### Table XL: Graphite 1200 mg

Concentration	Test number	Ignition	Ignition	Induction	Result
[mg]		energy	set		
			[ms]		
1500	1	1000 mJ	120	1 mH	NO ignition
1500	error	1000 mJ	120	1 mH	electrostatic
1500	error	1000 mJ	120	1 mH	error
	cleaning				
1500	2	1000 mJ	120	1 mH	NO ignition
1500	error	1000 mJ	120	1 mH	electrostatic
					discharge
	cleaning				
1500	3	1000 mJ	120	1 mH	NO ignition
1500	error	1000 mJ	120	1 mH	electrostatic discharge
	cleaning				<u> </u>
1500	4	1000 mJ	120	1 mH	NO ignition
1500	5	1000 mJ	120	1 mH	NO ignition
1500	error	1000 mJ	120	1 mH	electrostatic discharge
	cleaning				
1500	error	1000 mJ	120	1 mH	electrostatic discharge
	cleaning				
1500	6	1000 mJ	120	1 mH	NO ignition
1500	error	1000 mJ	120	1 mH	electrostatic discharge
	cleaning				diconargo
1500	7	1000 mJ	120	1 mH	NO ignition
1500	error	1000 mJ	120	1 mH	electrostatic
1500	8	1000 mJ	120	1 mH	NO ignition
1500	error	1000 mJ	120	1 mH	electrostatic
1500	9	1000 mJ	120	1 mH	NO ignition
1500	10	1000 mJ	120	1 mH	NO ignition

Table XLI: Graphite 1500 mg

### 6 Investigations on dust mixtures

### 6.1 Minimum ignition energy (MIE)

The first tested parameter was the minimum ignition energy (MIE), investigated with the MIKE 3-appartus from Kühner AG. The tests were made with an ignition delay time of 120 ms and ignition energy of 1000 mJ. An inductance of 1 mH was used to produce time protracted sparks.
# 6.1.1 Brown Coal

## 6.1.1.1 Investigation of 40% brown coal and 60% sewage sludge ashes

With the MIKE 3-apparatus the first mixture of brown coal and sewage sludge was 40% brown coal dust and 60% sewage ash. The concentration ranged from 300 mg to 1500 mg. The mixture of these dusts did not ignite for ten times. Because it is not possible for the MIKE 3-apparatus to provide ignition energies above 1000 mJ the testing result is that mixtures of 40% coal and 60% sewage sludge ash have a MIE above 1000 mJ. The course of the explosion is shown in **Figure 47** and the results can be seen in **Table XLII**.

Concentration [mg]	Ignition enerav	Ignition delav time	Effective ignition	Induction	Ignition
[	5115135	set	delay time		
		[ms]	[ms]		
300	1000 mJ	120	122	1 mH	NO
600	1000 mJ	120	122	1 mH	NO
900	1000 mJ	120	122	1 mH	NO
1200	1000 mJ	120	122	1 mH	NO
1500	1000 mJ	120	122	1 mH	NO





## 6.1.1.2 Investigation of 35% brown coal and 65% sewage sludge ashes

The next test with the MIKE 3-apparatus was made with 35% brown coal dust and 65% sewage ash, in a concentration range of 300 mg to 1500 mg. The mixture 35% coal dust with 65% sewage sludge ash did not ignite for ten times, so this means that these mixtures have a MIE above 1000 mJ. The results can be seen in **Table XLIII** and the course of the test is shown in **Figure 48**.

Concentration [mg]	Ignition energy	Ignition delay time set [ms]	Effective ignition delay time [ms]	Induction	Ignition
300	1000 mJ	120	122	1 mH	NO
600	1000 mJ	120	122	1 mH	NO
900	1000 mJ	120	122	1 mH	NO
1200	1000 mJ	120	122	1 mH	NO
1500	1000 mJ	120	122	1 mH	NO

Table XLIII:	MIE	- 35%	brown	coal
--------------	-----	-------	-------	------



Figure 48: MIE - 35% brown coal

#### 6.1.1.3 Investigation of 30% brown coal and 70% sewage sludge ashes

With a concentration of 30% brown coal dust and 70% sewage ash the third trial for the MIE was made. The dust concentration varied from 300 mg to 1500 mg. During the tests no ignition occurred with an ignition energy of 1000 mJ. Because it is not possible for the MIKE 3-apparatus to provide ignition energies above 1000 mJ the testing result is, that mixtures of 30% coal and 70% sewage sludge ash have a MIE above 1000 mJ. The course of the explosion is shown in **Figure 49** and the results can be seen in **Table XLIV**.

Concentration [mg]	Ignition energy	Ignition delay time set [ms]	Effective ignition delay time [ms]	Induction	Ignition
300	1000 mJ	120	122	1 mH	NO
600	1000 mJ	120	122	1 mH	NO
900	1000 mJ	120	122	1 mH	NO
1200	1000 mJ	120	122	1 mH	NO
1500	1000 mJ	120	122	1 mH	NO



Figure 49: MIE - 30% brown coal

#### 6.1.1.4 Investigation of 25% brown coal and 75% sewage sludge ashes

The next trial was made with 25% brown coal dust and 75% sewage ash, in a concentration range of 300 mg to 1500 mg. Twenty-five percent of coal dust with 75% sewage sludge ash did not ignite for ten times at an ignition energy of 1000 mJ. So the result is, that mixtures of 25% coal and 75% sewage sludge ash have a MIE above 1000 mJ. The results of the tests can be seen in **Table XLV** and the course of the explosion is shown in **Figure 50**.

Concentration [mg]	Ignition energy	Ignition delay time set [ms]	Effective ignition delay time [ms]	Induction	Ignition
300	1000 mJ	120	122	1 mH	NO
600	1000 mJ	120	122	1 mH	NO
900	1000 mJ	120	122	1 mH	NO
1200	1000 mJ	120	122	1 mH	NO
1500	1000 mJ	120	122	1 mH	NO





#### 6.1.1.5 Investigation of 20% brown coal and 80% sewage sludge ashes

The mixture of 20% brown coal dust and 80% sewage ash was used for the last trial. The dust concentration ranged was from 300 mg to 1500 mg. Even with an ignition energy of 1000 mJ and an ignition delay time of 120 ms, 20% coal dust with 80% sewage sludge ash did not ignite for ten times. This means that mixtures of 20% coal and 80% sewage sludge ash have a MIE above 1000 mJ. The results of the tests can be seen in **Table XLVI** and the course of the explosion is shown in **Figure 51**.

Concentration	Ignition	Ignition	Effective	Induction	Ignition
[mg]	energy	delay time	ignition		
		set	delay time		
		[ms]	[ms]		
300	1000 mJ	120	121	1 mH	NO
600	1000 mJ	120	122	1 mH	NO
900	1000 mJ	120	122	1 mH	NO
1200	1000 mJ	120	122	1 mH	NO
1500	1000 mJ	120	122	1 mH	NO



Figure 51: MIE - 20% brown coal

# 6.1.2 Charcoal

## 6.1.2.1 Investigation of 40% charcoal and 60% sewage sludge ashes

The first mixture for the MIKE 3-apparatus was 40% charcoal and 60% sewage sludge ash. The concentration ranged from 300 mg to 1500 mg. The mixture 40% coal dust with 60% sewage sludge ash did not ignite for ten times. Because it is not possible for the MIKE 3-apparatus to provide ignition energies above 1000 mJ the testing result is, that mixtures of 40% coal and 60% sewage sludge ash have a MIE above 1000 mJ. The course of the explosion is shown in **Figure 52** and the results can be seen in **Table XLVII**.

Concentration [mg]	Ignition energy	Ignition delay time	Effective ignition	Induction	Ignition
1 01	0,	set	delay time		
		[ms]	[ms]		
300	1000 mJ	120	122	1 mH	NO
600	1000 mJ	120	122	1 mH	NO
900	1000 mJ	120	122	1 mH	NO
1200	1000 mJ	120	122	1 mH	NO
1500	1000 mJ	120	122	1 mH	NO

#### Table XLVII: MIE - 40% charcoal





#### 6.1.2.2 Investigation of 35% charcoal and 65% sewage sludge ashes

The next trial was made with a lower coal content. The concentration for 35% charcoal and 65% sewage ash varied from 300 mg to 1500 mg. This mixture did not ignite for ten times, so the testing result is, that mixtures of 35% charcoal and 65% sewage sludge ash have a MIE above 1000 mJ. The course of the explosion can be seen in **Figure 53** and the results can be taken from **Table XLVIII**.

Concentration [mg]	Ignition energy	Ignition delay time set [ms]	Effective ignition delay time [ms]	Induction	Ignition
300	1000 mJ	120	122	1 mH	NO
600	1000 mJ	120	122	1 mH	NO
900	1000 mJ	120	121	1 mH	NO
1200	1000 mJ	120	122	1 mH	NO
1500	1000 mJ	120	122	1 mH	NO

Table 2	XLVIII:	MIE -	35%	charcoal
---------	---------	-------	-----	----------



Figure 53: MIE - 35% charcoal

#### 6.1.2.3 Investigation of 30% charcoal and 70% sewage sludge ashes

A mixture of 30% charcoal and 70% sewage sludge ash was the next to be tested. Like all other tests, the concentration ranged from 300 mg to 1500 mg. This mixture did not ignite for ten times. Because it is not possible for the MIKE 3-apparatus to provide ignition energies above 1000 mJ the testing result is, that mixtures of 30% coal and 70% sewage sludge ash have a MIE above 1000 mJ. The course of the explosion is shown in **Figure 54** and the results can be seen in **Table XLIX**.

Concentration [mg]	Ignition energy	Ignition delay time set [ms]	Effective ignition delay time [ms]	Induction	Ignition
300	1000 mJ	120	122	1 mH	NO
600	1000 mJ	120	122	1 mH	NO
900	1000 mJ	120	122	1 mH	NO
1200	1000 mJ	120	122	1 mH	NO
1500	1000 mJ	120	122	1 mH	NO

	Table >	KLIX:	MIE -	30%	charcoal
--	---------	-------	-------	-----	----------



Figure 54: MIE - 30% charcoal

#### 6.1.2.4 Investigation of 25% charcoal and 75% sewage sludge ashes

The next to the last investigation for charcoal with the MIKE 3-apparatus was made with 25% coal dust and 75% sewage ash. The concentration ranged from 300 mg to 1500 mg. The mixture did not ignite for ten times. Because it is not possible for the MIKE 3-apparatus to provide ignition energies above 1000 mJ the testing result is, that mixtures of 25% coal and 75% sewage sludge ash have a MIE above 1000 mJ. The course of the explosion is shown in **Figure 55** and the results can be seen in **Table L**.

Concentration [mg]	Ignition energy	Ignition delay time set [ms]	Effective ignition delay time [ms]	Induction	Ignition
300	1000 mJ	120	122	1 mH	NO
600	1000 mJ	120	122	1 mH	NO
900	1000 mJ	120	122	1 mH	NO
1200	1000 mJ	120	121	1 mH	NO
1500	1000 mJ	120	122	1 mH	NO

	Table	L:	MIE	- 25%	charcoa
--	-------	----	-----	-------	---------



Figure 55: MIE - 25% charcoal

## 6.1.2.5 Investigation of 20% charcoal and 80% sewage sludge ashes

The last trial was made with 20% brown coal dust and 80% sewage ash. The mixture of 20% coal dust with 80% sewage sludge ash did not ignite for ten times, so the testing result is, that mixtures of 20% coal and 80% sewage sludge ash have a MIE above 1000 mJ. The course of the explosion is shown in **Figure 56** and the results can be seen in **Table LI**.

Concentration [mg]	Ignition energy	Ignition delay time set [ms]	Effective ignition delay time [ms]	Induction	Ignition
300	1000 mJ	120	122	1 mH	NO
600	1000 mJ	120	122	1 mH	NO
900	1000 mJ	120	122	1 mH	NO
1200	1000 mJ	120	122	1 mH	NO
1500	1000 mJ	120	122	1 mH	NO

#### Table LI: MIE - 20% charcoal



Figure 56: MIE - 20% charcoal

# 6.1.3 Hard coal

## 6.1.3.1 Investigation of 40% hard coal and 60% sewage sludge ashes

The mixture of 40% hard coal dust and 60% sewage ash was the first investigated mixture of the hard coal dust in the MIKE 3-apparatus. The concentration varied from 300 mg to 1500 mg. 40% coal dust and 60% sewage sludge ash did not ignite for ten times. Because it is not possible for the MIKE 3-apparatus to provide ignition energies above 1000 mJ the testing result is, that mixtures of 40% coal and 60% sewage sludge ash have a MIE above 1000 mJ. The course of the explosion is shown in **Figure 57** and the results can be seen in **Table LII**.

Concentration [mg]	Ignition energy	Ignition delay time	Effective ignition	Induction	Ignition
1 01	0,	set	delay time		
		[ms]	[ms]		
300	1000 mJ	120	121	1 mH	NO
600	1000 mJ	120	121	1 mH	NO
900	1000 mJ	120	121	1 mH	NO
1200	1000 mJ	120	120	1 mH	NO
1500	1000 mJ	120	120	1 mH	NO

Table LII: MIE - 40% hard coal



Figure 57: MIE - 40% hard coal

#### 6.1.3.2 Investigation of 35% hard coal and 65% sewage sludge ashes

The next test was made with 35% hard coal dust and 65% sewage ash, in a concentration changed of 300 mg to 1500 mg. 35% coal dust with 65% sewage sludge ash did not ignite for ten times, so this means that these mixtures have a MIE above 1000 mJ. The results can be seen in **Table LIII** and the course of the test is shown in **Figure 58**.

Concentration [mg]	Ignition energy	Ignition delay time set [ms]	Effective ignition delay time [ms]	Induction	Ignition
300	1000 mJ	120	122	1 mH	NO
600	1000 mJ	120	122	1 mH	NO
900	1000 mJ	120	122	1 mH	NO
1200	1000 mJ	120	122	1 mH	NO
1500	1000 mJ	120	122	1 mH	NO

Table LIII: MIE - 35% hard coal



Figure 58: MIE - 35% hard coal

#### 6.1.3.3 Investigation of 30% hard coal and 70% sewage sludge ashes

With a concentration of 30% hard coal dust and 70% sewage ash the third trial was made. The dust concentration ranged from 300 mg to 1500 mg. Because it is not possible for the MIKE 3-apparatus to provide ignition energies above 1000 mJ the testing result is, that mixtures of 30% coal and 70% sewage sludge ash have a MIE above 1000 mJ. The course of the explosion is shown in **Figure 59** and the results can be seen in **Table LIV**.

Concentration [mg]	Ignition energy	Ignition delay time set [ms]	Effective ignition delay time [ms]	Induction	Ignition
300	1000 mJ	120	122	1 mH	NO
600	1000 mJ	120	122	1 mH	NO
900	1000 mJ	120	122	1 mH	NO
1200	1000 mJ	120	122	1 mH	NO
1500	1000 mJ	120	122	1 mH	NO

T	able	LIV:	MIE	- 30%	hard	coal
	unic			00/0	nuiu	ooui





## 6.1.3.4 Investigation of 25% hard coal and 75% sewage sludge ashes

The next trial was 25% hard coal dust and 75% sewage ash, in a concentration range of 300 mg to 1500 mg. The mixture of 25% coal dust with 75% sewage sludge ash did not ignite for ten times. So the result is that mixtures of 25% coal and 75% sewage sludge ash have a MIE above 1000 mJ. The results of the tests can be seen in **Table LV** and the course of the explosion is shown in **Figure 60**.

Concentration [mg]	Ignition energy	Ignition delay time set [ms]	Effective ignition delay time [ms]	Induction	Ignition
300	1000 mJ	120	120	1 mH	NO
600	1000 mJ	120	120	1 mH	NO
900	1000 mJ	120	120	1 mH	NO
1200	1000 mJ	120	120	1 mH	NO
1500	1000 mJ	120	120	1 mH	NO

Table LV: MIE - 25% hard	l coal
--------------------------	--------



Figure 60: MIE - 25% hard coal

## 6.1.3.5 Investigation of 20% hard coal and 80% sewage sludge ashes

With a concentration of 20% hard coal dust and 80% sewage ash the last trial was made. The dust concentration varied was from 300 mg to 1500 mg. Even with an ignition energy of 1000 mJ and an ignition delay time of 120 ms, 20% coal dust with 80% sewage sludge ash did not ignite for ten times. This means that mixtures of 20% coal and 80% sewage sludge ash have a MIE above 1000 mJ. The results of the tests can be seen in **Table LVI** and the course of the explosion is shown in **Figure 61**.

Concentration [mg]	Ignition energy	Ignition delay time set [ms]	Effective ignition delay time [ms]	Induction	Ignition
300	1000 mJ	120	122	1 mH	NO
600	1000 mJ	120	122	1 mH	NO
900	1000 mJ	120	122	1 mH	NO
1200	1000 mJ	120	122	1 mH	NO
1500	1000 mJ	120	122	1 mH	NO

Т	able	LVI:	MIE	- 20%	hard	coal
	abic	L V I.		- 20 /0	naru	coai



Figure 61: MIE - 20% hard coal

# 6.1.4 Anthracite

## 6.1.4.1 Investigation of 40% anthracite and 60% sewage sludge ashes

The first mixture for the MIKE 3-apparatus was 40% anthracite and 60% sewage sludge ash. The concentration ranged from 300 mg to 1500 mg. This mixture did not ignite for ten times. Because it is not possible for the MIKE 3-apparatus to provide ignition energies above 1000 mJ the testing result is, that such mixtures have a MIE above 1000 mJ. The course of the explosion is shown in **Figure 62** and the results can be seen in **Table LVII**.

Concentration [mg]	Ignition energy	Ignition delay time set [ms]	Effective ignition delay time [ms]	Induction	Ignition
300	1000 mJ	120	122	1 mH	NO
600	1000 mJ	120	122	1 mH	NO
900	1000 mJ	120	122	1 mH	NO
1200	1000 mJ	120	122	1 mH	NO
1500	1000 mJ	120	122	1 mH	NO

Table LVII: MIE - 40% anthracite



Figure 62: MIE - 40% anthracite

#### 6.1.4.2 Investigation of 25% anthracite and 75% sewage sludge ashes

Because it is known that anthracite dust does not react, the next trial was made with a much lower anthracite content of 25% anthracite. If this mixture did not react, tests between 25% and 40% anthracite would not be necessary. The concentration for 25% anthracite and 75% sewage ash varied from 300 mg to 1500 mg. This mixture did not ignite for ten times, so the testing result is, that such mixtures have a MIE above 1000 mJ. The course of the explosion can be seen in **Figure 63** and the results can be taken from **Table LVIII**.

Concentration [mg]	Ignition energy	Ignition delay time set [ms]	Effective ignition delay time [ms]	Induction	Ignition
300	1000 mJ	120	122	1 mH	NO
600	1000 mJ	120	122	1 mH	NO
900	1000 mJ	120	122	1 mH	NO
1200	1000 mJ	120	122	1 mH	NO
1500	1000 mJ	120	123	1 mH	NO

Table LVIII: MIE - 25% anthr	acite
------------------------------	-------



Figure 63: MIE - 25% anthracite

# 6.1.5 Graphite

## 6.1.5.1 Investigation of 40% graphite and 60% sewage sludge ashes

The mixture of 40% graphite dust and 60% sewage ash was the first investigated dust. The concentration changed from 300 mg to 1500 mg and the test was made with an ignition delay time of 120 ms and ignition energy of 1000mJ. Forty percent of graphite dust with 60% sewage sludge ash did not ignite for ten times. So the testing result is that mixtures of 40% graphite and 60% sewage sludge ash have a MIE above 1000 mJ. The course of the testing is shown in **Figure 64** and the results can be seen in **Table LIX**.

Concentration [mg]	Ignition energy	Ignition delay time	Effective ignition	Induction	Ignition
1 01	0,	set	delay time		
		[ms]	[ms]		
300	1000 mJ	120	120	1 mH	NO
600	1000 mJ	120	120	1 mH	NO
900	1000 mJ	120	120	1 mH	NO
1200	1000 mJ	120	120	1 mH	NO
1500	1000 mJ	120	120	1 mH	NO

Table	1 IX·	MIF	- 40%	araphite
Iable			- 40 /0	graphic





## 6.1.5.2 Investigation of 25% graphite and 75% sewage sludge ashes

The last trial was made with a concentration of 20% graphite dust and 80% sewage ash. The dust concentration varied was from 300 mg to 1500 mg. Even with an ignition energy of 1000 mJ and an ignition delay time of 120 ms, 20% graphite dust with 80% sewage sludge ash did not ignite for ten times. So these mixtures have a MIE above 1000 mJ. The results of the tests can be seen in **Table LX** and the course is shown in **Figure 65**. Because this mixture did not react, tests between the content of 40% to 25% graphite were not necessary.

Concentration [mg]	Ignition energy	Ignition delay time set [ms]	Effective ignition delay time [ms]	Induction	Ignition
300	1000 mJ	120	120	1 mH	NO
600	1000 mJ	120	120	1 mH	NO
900	1000 mJ	120	120	1 mH	NO
1200	1000 mJ	120	120	1 mH	NO
1500	1000 mJ	120	120	1 mH	NO

Table LX: MIE - 25% graphit
-----------------------------



Figure 65: MIE - 25% graphite

# 6.2 Maximum explosion overpressure p<sub>MAX</sub>, maximum rate of pressure rise (dp/dt)<sub>MAX</sub> and K<sub>ST</sub>-value

Although MIKE 3 investigations showed that the MIE is above 1000 mJ, explosion scenarios cannot be eliminated. Therefore investigations about the possibility of the dust mixtures building explosive atmospheres had to be conducted in the 20-I-apparatus.

# 6.2.1 Brown Coal

## 6.2.1.1 Investigation of 40% brown coal and 60% sewage sludge ashes

The upper limit for the percentage of coal in the mixture for the RecoPhos reactor is 40%, so the first investigations in the 20-I-apperatus were made with a dust mixture of 40% brown coal and 60% sewage ash. The concentration ranged from 250 g/m<sup>3</sup> to 2750 g/m<sup>3</sup>. The maximum was between 750 g/m<sup>3</sup> and 1250 g/m<sup>3</sup>. Based on these results the maximum explosion overpressure  $p_{MAX}$  is 5.5 bar and the maximum rate of pressure rise (dp/dt)<sub>MAX</sub> is 164 bar/s. The calculated K<sub>ST</sub>-value is 44 m\*bar/s, that corresponds to the dust explosion class 1. The course of the explosion is shown in **Figure 66** and the results can be seen in **Table LXI**.

#### Table LXI: Values 40% brown coal

Series	Concentration [g/m³]	Maximum explosion overpressure р <sub>мах</sub> [bar]	Maximum rate of pressure rise (dp/dt) <sub>MAX</sub> [bar/s]	Ignition delay time [ms]	Chemical igniter energy
1	250	0	0	60	10kJ
1	500	0.4	3	60	10kJ
1	750	5.2	92	60	10kJ
1	1000	5.5	159	60	10kJ
1	1250	5	119	60	10kJ
1	1500	4.7	126	60	10kJ
1	1750	5	132	60	10kJ

Series	Concentration [g/m³]	Maximum explosion overpressure p <sub>MAX</sub> [bar]	Maximum rate of pressure rise (dp/dt) <sub>MAX</sub> [bar/s]	Ignition delay time [ms]	Chemical igniter energy
1	2000	4.5	133	60	10kJ
1	2250	4.3	133	60	10kJ
1	2500	4.5	136	60	10kJ
1	2750	4.4	149	60	10kJ
2	500	2.8	13	60	10kJ
2	750	5.4	133	60	10kJ
2	1000	5.4	159	60	10kJ
2	1250	5.2	169	60	10kJ
2	1500	5.1	154	60	10kJ
2	1750	5.3	157	60	10kJ



Figure 66: Explosion overpressure and rate of pressure rise - 40% brown coal

#### 6.2.1.2 Investigation of 35% brown coal and 65% sewage sludge ashes

The second trial was made with 35% brown coal dust and 65% sewage ash, in a concentration range of 250 g/m<sup>3</sup> to 2000 g/m<sup>3</sup>. The maximum was between 1000 g/m<sup>3</sup> and 1250 g/m<sup>3</sup>. To ensure the accuracy of the results, two series of testings were executed. Based on these results the maximum explosion overpressure  $p_{MAX}$  was 5.5 bar and the maximum rate of pressure rise (dp/dt)<sub>MAX</sub> was 148 bar/s. So the calculated K<sub>ST</sub>–value was 40 bar\*m/s. This means that a mixture of 35% brown coal dust and 65% sewage ash belongs to the dust explosion class 1. The results can be seen in **Table LXII** and the course of the explosion is shown in **Figure 67**.

Series	Concentration [g/m³]	Maximum explosion overpressure p <sub>MAX</sub> [bar]	Maximum rate of pressure rise (dp/dt) <sub>MAX</sub>	Ignition delay time [ms]	Chemical igniter energy
4	050		[bar/s]		401-1
1	250	0	0	60	TUKJ
1	500	3.3	26	60	10kJ
1	750	5.2	93	60	10kJ
1	1000	5.6	100	60	10kJ
1	1250	5.4	164	60	10kJ
1	1500	5.2	138	60	10kJ
1	1750	5.3	159	60	10kJ
1	2000	5.2	125	60	10kJ
2	500	2.1	6	60	10kJ
2	750	4.3	42	60	10kJ
2	1000	5.5	113	60	10kJ
2	1250	5.4	132	60	10kJ
2	1500	5.2	103	60	10kJ

Table LXII: Values 35% brown coal



Figure 67: Explosion overpressure and rate of pressure rise - 35% brown coal

#### 6.2.1.3 Investigation of 30% brown coal and 70% sewage sludge ashes

The next trial was 30% brown coal dust and 70% sewage ash. Based on the results of this series, the maximum explosion overpressure  $p_{MAX}$  was 5.2 bar, the maximum rate of pressure rise (dp/dt)<sub>MAX</sub> was 102 bar/s and the K<sub>ST</sub>-value was 28 bar\*m/s. The categorisation with these values means a dust explosion class of 1. The results of the tests can be seen in **Table LXIII** and the course of the explosion is shown in **Figure 68**.

Series	Concentration [g/m³]	Maximum explosion overpressure p <sub>MAX</sub> [bar]	Maximum rate of pressure rise (dp/dt) <sub>MAX</sub> [bar/s]	Ignition delay time [ms]	Chemical igniter energy
1	500	0	0	60	10kJ
1	750	4.5	42	60	10kJ
1	1000	4.4	31	60	10kJ
1	1250	4.9	38	60	10kJ
1	1500	4.8	97	60	10kJ
1	1750	5.2	113	60	10kJ
1	2000	4.3	90	60	10kJ
1	2250	4.5	70	60	10kJ

Table LXIII:	Values 30%	brown	coal
--------------	------------	-------	------

Series	Concentration [g/m³]	Maximum explosion overpressure p <sub>MAX</sub> [bar]	Maximum rate of pressure rise (dp/dt) <sub>MAX</sub> [bar/s]	Ignition delay time [ms]	Chemical igniter energy
2	1000	4.4	36	60	10kJ
2	1250	5.1	91	60	10kJ
2	1500	5	89	60	10kJ
2	1750	4.9	82	60	10kJ



Figure 68: Explosion overpressure and rate of pressure rise - 30% brown coal

#### 6.2.1.4 Investigation of 25% brown coal and 75% sewage sludge ashes

The fourth trial was made with a concentration of 25% brown coal dust and 75% sewage ash. The dust concentration ranged from 750 g/m<sup>3</sup> to 2500 g/m<sup>3</sup>. After the two series, the results were  $p_{MAX} = 4$  bar,  $(dp/dt)_{MAX} = 42$  bar/s and the K<sub>ST</sub>-value was calculated to 11 m\*bar/s, so this dust mixture belongs to the dust explosion class 1. The results are shown in **Table LXIV** and the course of the explosion can be seen in **Figure 69**.

Series	Concentration [g/m³]	Maximum explosion overpressure p <sub>MAX</sub> [bar]	Maximum rate of pressure rise (dp/dt) <sub>MAX</sub>	Ignition delay time [ms]	Chemical igniter energy
1	750	0	[bar/s]	60	1041
1	750	0	0	60	TUKJ
1	1000	0	0	60	10kJ
1	1250	3.2	16	60	10kJ
1	1500	3.7	26	60	10kJ
1	1750	3.2	11	60	10kJ
1	2000	4	24	60	10kJ
1	2250	3.7	40	60	10kJ
1	2500	3.5	39	60	10kJ
2	750	0	0	60	10kJ
2	1000	4.1	44	60	10kJ
2	1250	3.7	22	60	10kJ
2	1500	3.9	22	60	10kJ
2	1750	3.7	19	60	10kJ

Table LXIV: Values 25% brown coal



Figure 69: Explosion overpressure and rate of pressure rise – 25% brown coal

#### 6.2.1.5 Investigation of 20% brown coal and 80% sewage sludge ashes

With a concentration of 20% brown coal dust and 80% sewage ash the last trial was made. The dust concentration ranged from 750 g/m<sup>3</sup> to 2000 g/m<sup>3</sup>. With this large range of concentration and the strong igniter of 10 kJ, no explosion occurred. Therefore a mixture of 20% brown coal dust and 80% sewage sludge ash is not explosive. So the maximum explosion overpressure  $p_{MAX}$ , the maximum rate of pressure rise (dp/dt)<sub>MAX</sub> and the K<sub>ST</sub>-value cannot be found. The results of the tests can be seen in **Table LXV**.

Series	Concentration [g/m <sup>3</sup> ]	Maximum explosion overpressure	Maximum rate of pressure	Ignition delay time [ms]	Chemical igniter energy
		Duay	rise	[]	onorgy
		[bar]	(dp/dt)		
		[bai]	[bar/s]		
1	750	0	0	60	10kJ
1	1000	0	0	60	10kJ
1	1250	0	0	60	10kJ
1	1500	0	0	60	10kJ
1	1750	0	0	60	10kJ
1	2000	0	0	60	10kJ

Table LXV: Values 20% brown coal

# 6.2.2 Charcoal

## 6.2.2.1 Investigation of 40% charcoal and 60% sewage sludge ashes

Because the upper limit of coal dust in the RecoPhos reactor is 40%, the first investigations in the 20-I-apperatus were made with a dust mixture of 40% charcoal and 60% sewage ash. The concentration of the dust for the two series varied from 500 g/m<sup>3</sup> to 2500 g/m<sup>3</sup>. The results of the tests can be taken from **Table LXVI**. These results are the base for calculating the maximum explosion pressure and the maximum rate of pressure rise. The values for the dust 40% charcoal and 60% sewage ash are: maximum explosion overpressure  $p_{MAX}$  was 5.5 bar, the maximum rate of pressure rise (dp/dt)<sub>MAX</sub> was 102 bar/s and the calculated K<sub>ST</sub>-value was 28 bar\*m/s. The categorisation with these values results in a dust explosion class of 1. The course of the explosion tests are shown in **Figure 70**.

Series	Concentration	Maximum	Maximum	Ignition	Chemical
	[g/m <sup>3</sup> ]	explosion	rate of	delay time	igniter
	191	overpressure	pressure	[ms]	energy
		ρ	rise	[]	0
		[bar]	(dp/dt) <sub>MAX</sub>		
		[50.]	[bar/s]		
1	500	0	0	60	10k.l
·	000	Ũ	Ū	00	rono
1	750	4.6	34	60	10kJ
1	1000	5.6	103	60	10kJ
	(0.50				
1	1250	4.8	47	60	10kJ
1	1500	1 E	57	60	1061
I	1500	4.5	57	00	TUKJ
1	1750	4 1	29	60	10k.J
·			_0		i onto
1	2000	3.9	83	60	10kJ
1	2250	3.2	26	60	10kJ
	0500				4.01.1
1	2500	3.5	41	60	10kJ
2	500	0	0	60	10kJ
		-	-		
2	750	0	0	60	10kJ
2	1000	5.4	100	60	10kJ

Table LXVI: Values 40% charcoal

Series	Concentration [g/m³]	Maximum explosion overpressure PMAX [bar]	Maximum rate of pressure rise (dp/dt) <sub>MAX</sub> [bar/s]	lgnition delay time [ms]	Chemical igniter energy
2	1250	4.9	51	60	10kJ
2	1500	4.7	64	60	10kJ
2	1750	4.5	52	60	10kJ



Figure 70: Explosion overpressure and rate of pressure rise – 40% charcoal

#### 6.2.2.2 Investigation of 35% charcoal and 65% sewage sludge ashes

The second trial was made with a lower percentage of charcoal in the mixture coal and sewage ash. The concentration was 35% charcoal and 65% sewage sludge ash. In a concentration range of 500 g/m<sup>3</sup> to 2250 g/m<sup>3</sup>. Investigations showed that a maximum of the explosibility is in the range of between 1500 g/m<sup>3</sup> and 2000 g/m<sup>3</sup>. The results of the trial can be seen in **Table LXVII**. Out of these results the maximum explosion overpressure  $p_{MAX}$  was 4.8 bar and the maximum rate of pressure rise (dp/dt)<sub>MAX</sub> was 34 bar/s. With the help of the rate of pressure rise the K<sub>ST</sub>-value was calculated. The K<sub>ST</sub>-value of 35% charcoal and 65% sewage sludge ash was 9 bar\*m/s. This value signifies that the dust belongs to the dust explosion class 1. The course of the trials is shown in **Figure 71**.

Series	Concentration [g/m³]	Maximum explosion overpressure p <sub>MAX</sub> [bar]	Maximum rate of pressure rise (dp/dt) <sub>MAX</sub>	Ignition delay time [ms]	Chemical igniter energy
1	500	0	0	60	10kJ
1	750	0	0	60	10kJ
1	1000	0	0	60	10kJ
1	1250	1.2	2	60	10kJ
1	1500	5.1	35	60	10kJ
1	1750	4.8	28	60	10kJ
1	2000	0	0	60	10kJ
2	1000	0	0	60	10kJ
2	1250	0	0	60	10kJ
2	1500	4.5	24	60	10kJ
2	1750	4.1	22	60	10kJ
2	2000	4.2	32	60	10kJ
2	2250	4.1	15	60	10kJ

Table LXVII: Values 35% charcoal



Figure 71: Explosion overpressure and rate of pressure rise – 35% charcoal

## 6.2.2.3 Investigation of 30% charcoal and 70% sewage sludge ashes

This experiment was performed with a concentration of 30% charcoal dust and 70% sewage ash. The dust concentration range was from 500 g/m<sup>3</sup> to 2000 g/m<sup>3</sup>. Despite performing the tests with such a large range of concentration and the strong igniter of 10 kJ, no explosion occurred. As a result it can be said that a mixture of 30% charcoal dust and 70% sewage sludge ash is not explosive. The results of the trial can be taken from **Table LXVIII**.

Series	Concentration [g/m³]	Maximum explosion overpressure p <sub>MAX</sub> [bar]	Maximum rate of pressure rise (dp/dt) <sub>MAX</sub> [bar/s]	Ignition delay time [ms]	Chemical igniter energy
1	500	0	0	60	10kJ
1	750	0	0	60	10kJ
1	1000	0	0	60	10kJ
1	1250	0	0	60	10kJ
1	1500	0	0	60	10kJ
1	1750	0	0	60	10kJ
1	2000	0	0	60	10kJ

#### Table LXVIII: Values 30% charcoal

# 6.2.2.4 Investigation of 25% charcoal and 75% sewage sludge ashes

Although the previous trial had not produced an explosion, more tests with less charcoal were made to ensure the accuracy of the results. A mixture of 25% charcoal and 75% sewage ash did not ignite, even with a strong igniter of 10 kJ. The concentration range was wide and went from 500 g/m<sup>3</sup> to 2000 g/m<sup>3</sup>. Therefore a mixture of 25% charcoal and 75% sewage sludge ash is not explosive.

## 6.2.2.5 Investigation of 20% charcoal and 80% sewage sludge ashes

The last trial was made with a mixture of 20% charcoal and 80% sewage ash. The concentration range was very wide, from 500 g/m<sup>3</sup> to 2000 g/m<sup>3</sup>. Despite performing the tests with such a large range of concentration and the strong igniter of 10 kJ, no explosion occurred, so this mixture can be taken as non-explosive.

# 6.2.3 Hard coal

## 6.2.3.1 Investigation of 40% hard coal and 60% sewage sludge ashes

The mixture of 40% hard coal dust and 60% sewage ash was the first to investigate. The concentration varied from 60 g/m<sup>3</sup> to 1750 g/m<sup>3</sup>, with explosions at the concentration level of 750 g/m<sup>3</sup> to 1750 g/m<sup>3</sup>. The maximum was between 1000 g/m<sup>3</sup> and 1500 g/m<sup>3</sup>. To check the results, two series of testings were executed. Based on these results the maximum explosion overpressure  $p_{MAX}$  was 5.8 bar and the maximum rate of pressure rise (dp/dt)<sub>MAX</sub> was 144 bar/s. So the calculated  $K_{ST}$ -value was 39 m\*bar/s, that accords to the dust explosion class 1. The course of the explosion is shown in **Figure 72** and the results can be seen in **Table LXIX**.

Series	Concentration [g/m³]	Maximum explosion overpressure р <sub>мах</sub> [bar]	Maximum rate of pressure rise (dp/dt) <sub>MAX</sub> [bar/s]	Ignition delay time [ms]	Chemical igniter energy
1	60	0	0	60	10kJ
1	125	0	0	60	10kJ
1	500	0	0	60	10kJ
1	750	0	0	60	10kJ
1	1000	5.7	96	60	10kJ
1	1250	5.3	67	60	10kJ
1	1500	5.5	104	60	10kJ
1	1750	0	0	60	10kJ
2	750	5.1	57	60	10kJ
2	1250	5.7	133	60	10kJ
2	1500	5.8	180	60	10kJ
2	1750	5.6	184	60	10kJ

Table LXIX: Values 40% hard coal



Figure 72: Explosion overpressure and rate of pressure rise - 40% hard coal

## 6.2.3.2 Investigation of 35% hard coal and 65% sewage sludge ashes

The second trial was made with 35% hard coal dust and 65% sewage ash, in a concentration range of 500 g/m<sup>3</sup> to 1500 g/m<sup>3</sup>. For this mixture the maximum explosion overpressure  $p_{MAX}$  was 5.4 bar, the maximum rate of pressure rise (dp/dt)<sub>MAX</sub> was 122 bar/s and the K<sub>ST</sub>-value was 33 bar\*m/s, so the dust explosion class is 1. The results can be seen in **Table LXX** and the course of the explosion is shown in **Figure 73**.

Series	Concentration [g/m³]	Maximum explosion overpressure p <sub>MAX</sub> [bar]	Maximum rate of pressure rise (dp/dt) <sub>MAX</sub> [bar/s]	Ignition delay time [ms]	Chemical igniter energy
1	500	0	0	60	10kJ
1	750	4.5	34	60	10kJ
1	1000	5.2	100	60	10kJ
1	1250	4.6	80	60	10kJ
1	1500	4.0	94	60	10kJ
1	1750	4.0	89	60	10kJ

Table LX	X: Value	es 35% h	ard coal
----------	----------	----------	----------

Series	Concentration [g/m³]	Maximum explosion overpressure p <sub>MAX</sub> [bar]	Maximum rate of pressure rise (dp/dt) <sub>MAX</sub> [bar/s]	Ignition delay time [ms]	Chemical igniter energy
2	250	0	0	60	10kJ
2	500	4.5	58	60	10kJ
2	750	5.6	123	60	10kJ
2	1000	5.5	145	60	10kJ
2	1250	4.7	82	60	10kJ
2	1500	4.4	120	60	10kJ
2	1750	3.8	75	60	10kJ



Figure 73: Explosion overpressure and rate of pressure rise – 35% hard coal

## 6.2.3.3 Investigation of 30% hard coal and 70% sewage sludge ashes

With a concentration of 30% hard coal dust and 70% sewage ash the third trial was made. The dust concentration range was from 500 g/m<sup>3</sup> to 2000 g/m<sup>3</sup>. It must be taken into account that there was a large fluctuation range between the two series of one concentration. To ensure the results one more series was done. After the third series the fluctuation was smaller, so the results were  $p_{MAX} = 5.1$  bar,  $(dp/dt)_{MAX} = 81$  bar/s and the K<sub>ST</sub>-value is 22 m\*bar/s, so this dust mixture belongs to the dust explosion class 1. The results can be seen in **Table LXXI** and the course of the explosion is shown in **Figure 74**.

Series	Concentration [g/m³]	Maximum explosion overpressure p <sub>MAX</sub> [bar]	Maximum rate of pressure rise (dp/dt) <sub>MAX</sub> [bar/s]	Ignition delay time [ms]	Chemical igniter energy
1	500	0.0	0	60	10kJ
1	750	3.9	20	60	10kJ
1	1000	5.1	87	60	10kJ
1	1250	4.1	50	60	10kJ
1	1500	5.0	59	60	10kJ
1	1750	3.0	49	60	10kJ
1	2000	3.0	65	60	10kJ
2	500	0.0	0	60	10kJ
2	750	3.4	16	60	10kJ
2	1000	3.3	14	60	10kJ
2	1250	5.1	77	60	10kJ
2	1500	3.9	37	60	10kJ
3	500	0.2	4	60	10kJ
3	750	3.2	14	60	10kJ
3	1000	5.0	78	60	10kJ
3	1250	4.6	47	60	10kJ
3	1500	4.2	32	60	10kJ

Table LXXI: Values 30% hard coal



Figure 74: Explosion overpressure and rate of pressure rise - 30% hard coal

#### 6.2.3.4 Investigation of 25% hard coal and 75% sewage sludge ashes

The next trial was 25% hard coal dust and 75% sewage ash. Based on the results of this series, the maximum explosion overpressure  $p_{MAX}$  was 3.8 bar, the maximum rate of pressure rise (dp/dt)<sub>MAX</sub> was 36 bar/s and the K<sub>ST</sub>-value was 10 bar\*m/s. The categorisation with these values resulted in a dust explosion class of 1. The results of the tests can be seen in **Table LXXII** and the course of the explosion is shown in **Figure 75**.

Series	Concentration [g/m³]	Maximum explosion overpressure p <sub>MAX</sub> [bar]	Maximum rate of pressure rise (dp/dt) <sub>MAX</sub> [bar/s]	Ignition delay time [ms]	Chemical igniter energy
1	500	0	0	60	10kJ
1	750	0	0	60	10kJ
1	1000	0	0	60	10kJ
1	1250	3.7	17	60	10kJ
1	1500	3.8	36	60	10kJ
1	1750	3.6	34	60	10kJ
1	2000	2.9	4	60	10kJ

#### Table LXXII: Values 25% hard coal
Series	Concentration [g/m³]	Maximum explosion overpressure p <sub>MAX</sub> [bar]	Maximum rate of pressure rise (dp/dt) <sub>MAX</sub> [bar/s]	Ignition delay time [ms]	Chemical igniter energy
2	750	0	0	60	10kJ
2	1000	2.9	6	60	10kJ
2	1250	0	0	60	10kJ
2	1500	0.1	7	60	10kJ
2	1750	3.3	13	60	10kJ
2	2000	3.2	19	60	10kJ
2	2250	3.2	22	60	10kJ



Figure 75: Explosion overpressure and rate of pressure rise – 25% hard coal

#### 6.2.3.5 Investigation of 20% hard coal and 80% sewage sludge ashes

With a concentration of 20% hard coal dust and 80% sewage ash the last trial was made. The dust concentration range was from 750 g/m<sup>3</sup> to 1750 g/m<sup>3</sup>. With this large range of concentration and the strong igniter of 10 kJ, no explosion occurred. Therefor a mixture of 20% hard coal dust and 80% sewage sludge ash is not explosive. So the maximum explosion overpressure  $p_{MAX}$ , the maximum rate of pressure rise (dp/dt)<sub>MAX</sub> and the K<sub>ST</sub>-value cannot be found. The results of the tests can be seen in **Table LXXIII**.

Series	Concentration [g/m³]	Maximum explosion overpressure p <sub>MAX</sub> [bar]	Maximum rate of pressure rise (dp/dt) <sub>MAX</sub> [bar/s]	Ignition delay time [ms]	Chemical igniter energy
1	750	0	0	60	10kJ
1	1000	0	0	60	10kJ
1	1250	0	0	60	10kJ
1	1500	0	0	60	10kJ
1	1750	0	0	60	10kJ

#### Table LXXIII: Values 20% hard coal

### 6.2.4 Anthracite

### 6.2.4.1 Investigation of 40% anthracite and 60% sewage sludge ashes

The concentration of the mixture 40% anthracite and 60% sewage sludge ash for the two trials varied from 500 g/m<sup>3</sup> to 2250 g/m<sup>3</sup>. The results of the tests can be taken from **Table LXXIV**. Despite performing the testing with such a large range of concentration and the strong igniter of 10 kJ, no explosion occurred. Therefore this mixture can be taken as non-explosive. The course of the explosion tests are shown in **Figure 76**. Because this mixture was not explosive, mixtures with less than 40% anthracite dust can be supposed to be safe. For the RecoPhos reactor this investigation is sufficient, because the upper limit of the carbon source is 40%.

Series	Concentration [g/m³]	Maximum explosion overpressure p <sub>MAX</sub> [bar]	Maximum rate of pressure rise (dp/dt) <sub>MAX</sub> [bar/s]	Ignition delay time [ms]	Chemical igniter energy
1	500	0	0	60	10kJ
1	750	0	0	60	10kJ
1	1000	0	0	60	10kJ
1	1250	0	0	60	10kJ
1	1500	0	0	60	10kJ
1	1750	0	0	60	10kJ
1	2000	0	0	60	10kJ
1	2250	0	0	60	10kJ

#### Table LXXIV: Values 40% anthracite



Figure 76: Explosion overpressure and rate of pressure rise – 40% anthracite

### 6.2.5 Graphite

#### 6.2.5.1 Investigation of 40% graphite and 60% sewage sludge ashes

40% graphite dust and 60% sewage ash was the first mixture investigated. The concentration went from 750 g/m<sup>3</sup> to 1500 g/m<sup>3</sup>, but there was no explosion. So there is no maximum explosion overpressure  $p_{MAX}$  and also no maximum rate of pressure rise  $(dp/dt)_{MAX}$ . Thus it is not possible to calculate a K<sub>ST</sub>-value. This result means that a dust of 40% graphite and 60% sewage ash is not explosive, despite the large range of the tested concentration and the strong igniter of 10 kJ. The results – that there was no explosion - can be taken from **Table LXXV**. For the RecoPhos reactor mixtures of 40% or less graphite was used. Because 40% graphite and 60% sewage sludge ash was not explosive, mixtures with 40% graphite dust or less can be supposed to be safe.

Table LXXV: Values 40%	6 graphite

Series	Concentration [g/m³]	Maximum explosion overpressure р <sub>мах</sub> [bar]	Maximum rate of pressure rise (dp/dt) <sub>MAX</sub> [bar/s]	Ignition delay time [ms]	Chemical igniter energy
1	750	0	0	60	10 kJ
1	1000	0	0	60	10 kJ
1	1250	0	0	60	10 kJ
1	1500	0	0	60	10 kJ
1	1750	0	0	60	10 kJ



Figure 77: Explosion overpressure and rate of pressure rise – 40% graphite

# 7 Results

## 7.1 Carbon Sources

The results of the investigations on the carbon carriers with the help of the MIKE 3-apparatus and the 20-I-apparatus are summed up in **Table LXXVI**. For the minimum ignition energy the MIKE 3-apparatus was used, which can only produce sparks with an energy of 1000 mJ. Just one coal – brown coal - ignited. This means that the other substances have minimum ignition energies above 1000 mJ. In the 20-I-apparatus the ignition energy was higher, so all substances reacted.

#### Table LXXVI: Results carbon carriers

Carbon carrier	Explosion overpressure p <sub>max</sub> [bar]	Rate of pressure rise (dp/dt) <sub>max</sub> [bar/s]	K <sub>s⊺</sub> - value [bar*m/s]	Dust explosion class	Minimum ignition energy [mJ]
Brown coal	7.6	475	129	St 1	59,69
Charcoal	7.4	481	131	St 1	MIE above 1000 mJ
Hard coal	7.6	510	139	St 1	MIE above 1000 mJ
Anthracite	4.5	29	8	St 1	MIE above 1000 mJ
Graphite	1.4	3	1	St 1	MIE above 1000 mJ

There is an impact on the explosibility of dust caused by the volatility. According to Eckhoff, pulverized coal and coal dust do not have explosion properties unless there is a content of seven to eight percent of volatiles [25]. This dependency can also be seen in **Figure 78**, which shows the five investigated carbon sources. Anthracite has a volatile matter of 8% and it can be seen that this dust reacts in the 20-I-chamber. The reaction is not as intense as from hard coal, charcoal or brown coal, but it can be hazardous. On the contrary, graphite has a low reaction of just 1.4 bar with a strong igniter of 10 kJ. If a decision about safety requirements was just made using the explosion overpressure, safety equipment would have to be installed for all tested coals. But all the dusts are within dust explosion class 1. For the tests with the sewage sludge ash, it has to be determine if the strong reaction of hard coal, charcoal and brown coal can lead to a minimization or if the inert part of sewage ash is too low to influence the explosion properties.



Figure 78: Explosion pressure in relation to volatile components

The second investigated parameter is the maximum rate of pressure rise. This gives an idea of how fast the explosion occurred. **Figure 79** shows the maximum rate of pressure rise of the five carbon carriers. Graphite and anthracite dust have a low rate of pressure rise, so the reaction is slow and more like a combustion than an explosion. The hazard for using these two carbon carriers in a process is confined to their burning and therefore just fire prevention is needed.



Figure 79: Rate of pressure rise in relation to volatile components

Hard coal has the highest rate of pressure rise with 510 bar/s. If the ignition is strong enough to ignite hard coal dust, the dust reacts very fast and also very strongly, as seen by the high explosion overpressure of 7.6 bar. In contrast brown coal reacts more slowly, but it has a minimum ignition energy of about 60 mJ, so a lower energy is necessary to produce an explosion.

Another point to explain the different values for the explosion pressure and the rate of pressure rise is the caloric value. As seen in **Figure 80** the caloric value of brown coal is lower than the value of hard coal. So if an ignition source is strong enough to ignite hard coal, this coal has a higher caloric value and so the reaction is much stronger. For the ignition of brown coal the ignition source needs less energy, but also the reaction is slower. Anthracite and graphite have also a very high caloric value, but the ignition energy has to be very high too. The higher the volatile matter, the lower the caloric value, because of the high energy needed to eliminate the volatile components.



Figure 80: Calorific value in relation to the volatile components

A comparison among the five  $K_{ST}$ - values is also possible. **Figure 81** shows the  $K_{ST}$ - value as a function of the volatile components of the carbon carrier. All five carbon sources belong to the dust explosion class 1. This class has  $K_{ST}$ - values from 1 to 200 m\*bar/s. Hard coal, charcoal and brown coal have  $K_{ST}$ - values around 130 to 140, so they are in the upper range of class 1. By contrast anthracite and graphite have very low values and so they will be really safe to use. It has to be taken into account that these two substances are more expensive than, for example, charcoal. So the costs for the material and the plant should be calculated before the decision is made as to which carbon carrier will be taken.



Figure 81: K<sub>ST</sub>- value in relation to volatile components

# 7.2 Brown Coal and sewage sludge ash

### 7.2.1 Minimum ignition energy (MIE)

Results of the tested minimum ignition energy in the MIKE 3-apparatus can be seen in **Table LXXVII**. Because it is not possible for the MIKE 3-apparatus to provide ignition energies above 1000 mJ the testing result is that mixtures of 20% to 40% brown coal and the rest sewage sludge ash have a MIE above 1000 mJ. Brown coal dust on its own has a minimum ignition energy of about 60 mJ. This means that the inert part – the sewage sludge ash – helps to minimize the hazard of an explosion by increasing the ignition energy.

Brown coal	Sewage	Ignition	No ignition
dust	sludge ash		at 1000 mJ
[%]	[%]		ignition
			energy
40	60		Х
35	65		Х
30	70		Х
25	75		Х
20	80		Х

 Table LXXVII: Results MIE brown coal – sewage sludge ash

### 7.2.2 Maximum explosion overpressure and maximum rate of pressure rise

To get an overview of all the tested dust mixtures, all important values can be seen in **Table LXXVIII**. It can be seen that a reduction of the coal dust in the mixture sewage sludge ash (inert part) and coal dust results in a minimization of the explosibility.

Brown coal dust	Sewage sludge ash	Maximum explosion	Maximum rate of	K <sub>s⊤</sub> - Value [bar*m/s]	Dust explosion
[%]	[%]	overpressure	pressure		class
		[bar]	lse [bar/s]		
100	0	7.6	475	129	St 1
40	60	5.5	164	44	St 1
35	65	5.5	148	40	St 1
30	70	5.2	102	28	St 1
25	75	4	42	11	St 1
20	80	0	0	0	St 0

Table LXXVIII: Results brown coal - sewage ash

Between 100% brown coal dust and 40% brown coal dust, the maximum explosion overpressure falls from 7.6 bar to 5.5 bar. Also the maximum rate of pressure rise shows a great reduction from 475 bar/s to 164 bar/s, so the inert part has an impact on the explosibility. Another large reduction occurs between 25% and 20% brown coal dust. At the percentage of 20% brown coal dust and 80% sewage sludge ash, the mixture is not explosive. The reduction of the maximum overpressure in relation to the brown coal dust can be seen in **Figure 82**. For a safe process a mixture of just 20% brown coal or less and the rest of sewage sludge ash has to be used.





In **Figure 83** the maximum pressure rise as a function of the brown coal can be seen. The rate of pressure rise is nearly linear with the ratio of brown coal dust between 20% and 40% brown coal in the dust mixture.



Figure 83: Pressure rise as a function of brown coal

**Figure 84** shows the rising  $K_{ST}$ -value in dependence on the mixture of brown coal dust and sewage sludge ash.  $K_{ST}$ -values from 1 to 200 belongs to the dust explosion class 1, so the mixtures are in the lower limit of the class 1. This figure also shows that the  $K_{ST}$ -value is also rising linearly.





# 7.3 Charcoal and sewage sludge ash

## 7.3.1 Minimum ignition energy (MIE)

The results from tests made with the MIKE 3-apparatus of the minimum ignition energy can be seen in **Table LXXIX**. Because it is not possible for the MIKE 3-apparatus to provide ignition energies above 1000 mJ the testing result is that mixtures of 20% to 40% charcoal and the rest sewage sludge ash have a MIE above 1000 mJ.

Charcoal dust [%]	Sewage sludge ash [%]	Ignition	No ignition at 1000 mJ ignition energy
40	60		Х
35	65		Х
30	70		Х
25	75		Х
20	80		Х

Table LXXIX: Results MIE charcoal - sewage sludge ash

## 7.3.2 Maximum explosion overpressure and maximum rate of pressure rise

To get an overview of all the tested dust mixtures in the 20-I-apparatus, all important values can be taken from **Table LXXX**. It can be seen that a mixture with 30% charcoal or less is not explosive. So the limit of the content of charcoal is 30%. If the process needs more than 30% charcoal, safety equipment has to be installed. For the RecoPhos project this means that the reactor should be operated with less than 30% charcoal. **Figure 85** shows the maximum explosion pressure as a function of the charcoal content.

Charcoal	Sewage	Maximum	Maximum	K <sub>st</sub> - Value	Dust
dust	sludge ash	explosion	rate of	[bar*m/s]	explosion
[%]	[%]	overpressure	pressure		class
[···]	[]	[bar]	rise		
		[]	[bar/s]		
100	0	7 4	481	313	St 1
100	Ū	7.4	401	010	011
40	60	55	102	28	St 1
10		0.0		20	011
35	65	4.8	34	9	St 1
30	70	-	-	-	St 0
25	75	-	-	-	St 0
20	80	-	-	-	St 0

Table LXXX: Results charcoal - sewage ash



Figure 85: Explosion pressure as a function of charcoal content

The maximum rate of pressure rise in relation to the charcoal in the mixture is shown in **Figure 86**. It can be seen that the explosion pressure with a coal content of more than 30% the hazardousness is rising. The K<sub>ST</sub>-value dependence on the carbon content is shown in **Figure 87**. Even with a content of 40% charcoal this value is very low, therefore a mixture of 60% sewage sludge ash and 40% charcoal can be said to be safe.



Figure 86: Rate of pressure rise in relation to charcoal content





# 7.4 Hard coal and sewage sludge ash

## 7.4.1 Minimum ignition energy (MIE)

All results of the tested minimum ignition energy of hard coal can be seen in **Table LXXXI**. Because it is not possible for the MIKE 3-apparatus to provide ignition energies above 1000 mJ the testing result shows that mixtures of 20 to 40% hard coal and the rest sewage sludge ash have a MIE above 1000 mJ.

Hard coal dust	Sewage sludge ash	Ignition	No ignition at 1000 mJ ignition
			energy
40	60		Х
35	65		Х
30	70		Х
25	75		Х
20	80		Х

Table LXXXI: Results MIE hard coal - sewage sludge ash

### 7.4.2 Maximum explosion overpressure and maximum rate of pressure rise

An overview of all the tested hard coal – sewage sludge ash mixtures in the 20-I-apparatus can be seen in **Table LXXXII**.

Hard coal dust [%]	Sewage sludge ash [%]	Maximum explosion overpressure [bar]	Maximum rate of pressure rise [bar/s]	K <sub>s⊤</sub> - Value [bar*m/s]	Dust explosion class
100	0	7.6	510	139	St 1
40	60	5.8	144	39	St 1
35	65	5.4	122	33	St 1

Table LXXXII: Results hard coal - sewage ash

Hard coal dust [%]	Sewage sludge ash [%]	Maximum explosion overpressure [bar]	Maximum rate of pressure rise [bar/s]	K <sub>sT</sub> - Value [bar*m/s]	Dust explosion class
30	70	5.1	81	22	St 1
25	75	3.8	36	10	St 1
20	80	-	-	-	-

Apparent from the results from the first tests, a reduction of the coal dust results in a minimization of the explosibility. As shown in **Figure 88**, the maximum explosion overpressure greatly reduced nearly 1 bar between 30% and 25% hard coal dust and between 20% and 25% there is a minimization of the maximum explosion overpressure to 0 bar. The minimum required ratio of ashes to achieve this effect is 80% sewage sludge ash. **Figure 89** shows the maximum rate of pressure rise (dp/dt)<sub>MAX</sub> in relation to the ratio of hard coal dust. It can be seen that the maximum rate of pressure rise is increasing nearly linearly between 20% and 40% hard coal in the mixture. Thus a falling inert part of the mixture impacts the velocity of an explosion.



Figure 88: Explosion pressure in relation to the ratio of hard coal



Figure 89: Rate of pressure rise in relation to hard coal

The  $K_{ST}$ -value as a function of the hard coal dust is shown in **Figure 90**. Values from 1 to 200 bar\*m/s are counted for the dust explosion class 1, so it can be seen that all mixtures have a very low value in the first class.



Figure 90: KST-value as a function of hard coal

# 7.5 Anthracite and sewage sludge ash

## 7.5.1 Minimum ignition energy (MIE)

All results of the tested minimum ignition energy of anthracite and sewage sludge ash can be seen in **Table LXXXIII**. Because it is not possible for the MIKE 3-apparatus to provide ignition energies above 1000 mJ the testing result is that mixtures of 25 to 40% anthracite coal and the rest sewage sludge ash have a MIE above 1000 mJ.

Anthracite dust	Sewage sludge ash	Ignition	No ignition at 1000 mJ ignition
			energy
40	60		Х
25	75		Х

Table LXXXIII: Results MIE anthracite - sewage sludge ash

### 7.5.2 Maximum explosion overpressure and maximum rate of pressure rise

The first tested mixture of anthracite and sewage sludge ash was 40% anthracite dust and the rest was sewage ash. Despite performing the testings with such a large range of concentrations and the strong igniter of 10 kJ, no explosion occurred, so this mixture can be taken as non-explosive. Because this mixture was not explosive, mixtures with 40% or less anthracite dust can be assessed to be safe. For the RecoPhos process the upper limit of the carbon source is 40%, so no more investigations were needed. **Table LXXXIV** shows the results from the test.

Table LXXXIV: Results anthracite - sewage ash

Anthracite dust [%]	Sewage sludge ash [%]	Maximum explosion overpressure [bar]	Maximum rate of pressure rise [bar/s]	K <sub>sT</sub> - Value [bar*m/s]	Dust explosion class
100	0	4.5	29	8	St 1
40	60	-	-	-	St 0

## 7.6 Graphite and sewage sludge ash

### 7.6.1 Minimum ignition energy (MIE)

All results of the tested minimum ignition energy can be seen in **Table LXXXV**. Because it is not possible for the MIKE 3-apparatus to provide ignition energies above 1000 mJ the testing result is that mixtures of 25 to 40% graphite and the rest sewage sludge ash have a MIE above 1000 mJ.

Graphite dust	Sewage sludge ash	Ignition	No ignition at 1000 mJ ignition
			energy
40	60		Х
25	75		Х

Table LXXXV: Results MIE graphite - sewage sludge ash

### 7.6.2 Maximum explosion overpressure and maximum rate of pressure rise

40% graphite dust and 60% sewage ash was the first investigated mixture in the 20-lapparatus. No explosion occurred. Therefore a dust of 40% graphite and 60% sewage ash is not explosive, even for the large range of the tested concentrations and the strong igniter of 10 kJ. For the RecoPhos process mixtures of 40% or less graphite are used. For this reason no more tests were made, because mixtures with 40% graphite dust or less and the rest sewage sludge ash can be assessed to be safe. **Table LXXXVI** shows the results from the tests in the 20-l-apparatus.

Table LXXXVI: Results graphite - sewage ash

Graphite dust [%]	Sewage sludge ash [%]	Maximum explosion overpressure [bar]	Maximum rate of pressure rise [bar/s]	K <sub>s⊤</sub> - Value [bar*m/s]	Dust explosion class
100	0	1.4	3	1	St 1
40	60	-	-	-	St 0

# 8 Conclusions

Historically many explosions in the process industry have their origins in dust explosions. For a dust mixture of sewage sludge ash and a carbon carrier this hazard is present. The aim of this thesis was to find out the safest dust mixture of sewage sludge ash and a carbon source. Brown coal, charcoal, hard coal, anthracite and graphite were used to perform the experiments for evaluating the possibility of the occurrence of dust explosions.

The critical parameters for such tests are the explosion indices maximum pressure rise, maximum explosion pressure and the minimum ignition energy. The tests were made with two apparatuses from Kühner AG (MIKE 3 and 20-I-apparatus) with different ratios of carbon carriers. During the tests the impact of the composition of the different coals became apparent. It can be said that volatility plays a role in the explosibility. The more volatile the carbon carriers, the higher the explosiveness. Brown coal has the highest percentage of volatile material, so it is possible to ignite brown coal dust with an ignition energy lower than 1000 mJ.

Furthermore various mixtures with different ratios of the carbon carrier in the mixture were tested to find the concentration where no explosion occurs. Sewage sludge ash is the inert part of the mixture, therefore the amount of inert material necessary to supress an explosion, has to be found. **Figure 91** shows the maximum explosion overpressure in dependent on the ratio of the carbon carrier in the dust mixture and in **Figure 92** the maximum rate of pressure rise as a function of the ratio of the carbon carrier can be seen.



Figure 91: Maximum explosion pressure in relation to the ratio of carbon carrier



Figure 92: Maximum rate of pressure rise in relation to ratio of carbon carrier

It can be seen that graphite and anthracite (in the figure as red and purple points) are nonreactive and even with a low part of the inert material (60%) no explosion occurs. In contrast brown coal and hard coal need more than 75% of the inert material to prevent an explosion. With charcoal an explosion of the mixture sewage sludge ash and charcoal dust can occur with a content of more than 20% charcoal dust. All results for evaluating the possibility of the occurrence of dust explosions can be taken from **Table LXXXVII**. The content of the carbon carrier in the mixture sewage sludge ash and carbon source can be seen in column one. The red line shows the boundary between explosive and non-explosive.

Brown coal [%]	Maximum explosion pressure Pmax [bar]	Maximum rate of pressure rise (dp/dt)max	K <sub>st</sub> value [bar*m/s]	Minimum ignition energy MIE [mJ]	Dust explosion class
		[bar/s]			
100	7.6	475	129	59,69	St 1
40	5.5	164	44	above 1000 mJ	St 1
35	5.5	148	40	above 1000 mJ	St 1
30	5.2	102	28	above 1000 mJ	St 1
25	4	42	11	above 1000 mJ	St 1
20	0	0	-	above 1000 mJ	St 0
Charcoal					
[%]					
100	7.4	481	313	above 1000 mJ	St 1
40	5.5	102	28	above 1000 mJ	St 1
35	4.8	34	9	above 1000 mJ	St 1
30	0	0	-	above 1000 mJ	St 0
25	0	0	-	above 1000 mJ	St 0
20	0	0	-	above 1000 mJ	St 0
Hard coal					
100	7.6	510	139	above 1000 mJ	St 1
40	5.8	144	39	above 1000 mJ	St 1
35	5.4	122	33	above 1000 m.J	St 1
30	5.1	81	22	above 1000 m.J	St 1
25	3.8	36	10	above 1000 mJ	St 1
20	0	0	-	above 1000 mJ	St 0

Table LXXXVII: All results

Anthracite [%]	Maximum explosion pressure Pmax [bar]	Maximum rate of pressure rise (dp/dt)max [bar/s]	K <sub>s⊤</sub> value [bar*m/s]	Minimum ignition energy MIE [mJ]	Dust explosion class
100	4.5	29	8	above 1000 mJ	St 1
40	0	0	-	above 1000 mJ	St 0
35	0	0	-	above 1000 mJ	St 0
30	0	0	-	above 1000 mJ	St 0
25	0	0	-	above 1000 mJ	St 0
20	0	0	-	above 1000 mJ	St 0
Graphite [%]					
100	1.4	3	1	above 1000 mJ	St 1
40	0	0	-	above 1000 mJ	St 0
35	0	0	-	above 1000 mJ	St 0
30	0	0	-	above 1000 mJ	St 0
25	0	0	-	above 1000 mJ	St 0
20	0	0	-	above 1000 mJ	St 0

If the RecoPhos process needs a mixture of 40% carbon carrier only graphite and anthracite can be used to produce an explosion-safe mixture of dust. The utilization of charcoal assumes a percentage of 30% or lower for the carbon dust. If the process just needs carbon carriers of 20% or less, hard coal and brown coal can be used for a safe process. For all other mixtures of sewage sludge ash and carbon source, safety equipment has to be installed.

# References

- Crowl, D. A., Understanding explosions, Center for Chemical Process Safety of the American Institute of Chemical Engineers, New York, 2003.
- [2] Abbasi, T. and S. A. Abbasi, Dust explosions-cases, causes, consequences, and control, J.
   Hazard. Mater. 140 (2007), 1-2, 7–44.
- [3] Cashdollar, K. L., Overview of dust explosibility characteristics, Journal of Loss Prevention in the Process Industries 13 (2000), 3-5, 183–199.
- [4] Bartknecht, W. and Zwahlen, G., Dust-explosions, Springer-Verlag, Berlin, New York, 1989.
- [5] European Parliament and the Council (16.12.1999), Directive 1999/92/EC of the European Parliament and of the Council of 16 December 1999 on minimum requirements for improving the safety and health protection of workers potentially at risk from explosive atmospheres Directive 1999/92/EC. Source: Official Journal L 023, 28/01/2000, P. 0057 - 0064. Available online at http://eur-lex.europa.eu/, checked on 11.05.2014.
- [6] Seton, Warnzeichen. Available online at www.seton.at, checked on 23.04.2014.
- [7] Cesana, C. and Siwek, R., Handbuch, Birsfelden, 2011.
- [8] EN 14034-1, 2011, Determination of explosion characteristics of dust clouds Part 1: Determination of the maximum explosion pressure pmax of dust clouds.
- [9] EN 14034-2, 2006, Determination of explosion characteristics of dust clouds Part 2:
   Determination of the maximum rate of explosion pressure rise (dp/dt)max of dust clouds.
- [10] Eckhoff, R. K., Explosion hazards in the process industries, Gulf Pub., Houston, TX, 2005.
- [11] EN 13821, 2002, Potential explosive atmospheres Explosion prevention and protection -Determination of minimum ignition energy of dust/air mixtures.

- [12] Cesana, C. and Siwek, R., MIKE 3, Birsfelden, 2001.
- [13] Wagner, K. W. and Käfer, R., PQM Prozessorientiertes Qualitätsmanagement, Hanser, München, 2010.
- [14] Kural, O. C., Coal, Istanbul Technical University, Istanbul, Türkiye, 1994.
- [15] Anton Lissner, Charakteristische Eigenschaften der Braunkohlen, Akademie-Verlag GmbH, Berlin, 1960.
- [16] Kaltschmitt, M., Energie aus Biomasse, Springer, Dordrecht, Heidelberg, London, New York, NY, 2009.
- [17] EN 1860-2, 2005, Appliances, solid fuels and firelighters for barbecueing Part 2: Barbecue charcoal and barbecue charcoal briquettes Requirements and test methods.
- [18] Wirtschaftsvereinigung Bergbau (Ed.), Das Bergbauhandbuch, Glückauf, Essen, 1994.
- [19] Gmelin (Ed.), Gmelins Handbuch der anorganischen Chemie Kohlenstoff Teil B Lieferung 2, Chemie GmbH, Weinheim, Bergstraße, 1968.
- [20] mindat.org, Graphite: Graphite mineral information and data. Available online at http://www.mindat.org/min-1740.html, checked on 25.04.2014.
- [21] Glor, M., Ignition hazard due to static electricity in particulate processes, Powder Technology 135-136 (2003), 223–233.
- [22] Glor, M., Overview of the occurrence and incendivity of cone discharges with case studies from industrial practice, Journal of Loss Prevention in the Process Industries 14 (2001), 2, 123–128.
- [23] Glor, M., Electrostatic ignition hazards in the process industry, Journal of Electrostatics 63 (2005), 6-10, 447–453.
- [24] Glor, M., Ignition source static electricity: Incident investigation, Journal of Electrostatics 67 (2009), 2-3, 242–246.
- [25] Eckhoff, R. K., Prevention and mitigation of dust explosions in the process industries: A survey of recent research and development, Journal of Loss Prevention in the Process Industries 9 (1996), 1, 3–20.



# Annex I

# Annex II



Sympatec GmbH System-Partikel-Technik

HELOS-Partikelgrößenanalyse WINDOX 4

HELOS (H1473) & RODOS, R5: 0.5/4.5...875µm

#### 30.09.2013, 13:11:44,6090



150,00

180,00

70,19

75,47

37,50

45,00

34,60

38,39

13,93

16,07

9,00

11,00

100,00

615,00

735,00

13,00	17,98	52,50	41,83	215,00	80,46	875,00	100,00		
15,50	20,15	62,50	45,99	255,00	85,12				
Verteilun	gsdichte (log.)			_					
x <sub>m</sub> /μm	q3lg	xm/μm	q3lg	x <sub>π</sub> /μm	q3lg	xm/μm	q3lg		
1,50	0,07	16,93	0,31	68,47	0,59	278,88	0,61		
4,97	0,19	19,94	0,34	82,16	0,62	333,65	0,58		
5,98	0,20	23,18	0,37	97,21	0,64	398,47	0,47		
6,98	0,22	27,39	0,40	114,56	0,66	473,31	0,27		
8,22	0,23	33,54	0,44	136,93	0,67	562,78	0,00		
9,95	0,25	41,08	0,48	164,32	0,67	672,33	0,00		
11,96	0,26	48,61	0,51	196,72	0,65	801,95	0,00		
14,20	0,28	57,28	0,55	234,15	0,63				
Auswertun	g: WINDOX 4.2.1	.0, HRLD		Produkt: LB	31				
Revalidien	mg:			Dichte: 7,80	Dichte: 7,80 g/cm3, Formfaktor: 1,00				
Referenzm	essung: 30.09.1	3:11:28		DispMeth	Disp -Meth : LB2				
Kontamina	tion: 0.00.94			C = 12.06	0.4				
Romannia	. 0,00 /0			Cop(= 12,50	/1				
Meßbeding	ung: md4			Benutzerpar	ameter:				
27 - 1 - A	1000 00			Parameter 1					
Zeitbasis:	1000,00 ms			a bis bissing that do					
Zeitbasis: Start:	1000,00 ms Startknopf			Parameter 2:					
Zeitoasis: Start: Gültigkoit:	Startknopf			Parameter 2:					
Zeitoasis: Start: Gültigkeit:	Startknopf immer			Parameter 2: Parameter 3:					
Zeitoasis: Start: Gültigkeit: Stopp:	Startknopf immer 3,00s Echtzeit			Parameter 2: Parameter 3: Parameter 4:	•				
Zeitoasis: Start: Gültigkeit: Stopp:	Startknopf immer 3,00s Echtzeit Sympatec Gr	nbH		Parameter 2: Parameter 3: Parameter 4:	TI OS-Pa	rtikeloröl	Renanalwe		
Zeitbasis: Start: Gültigkeit: Stopp:	Startknopf immer 3,00s Echtzeit Sympatec Gr System-Parti	nbH kel-Technik		Parameter 2: Parameter 3: Parameter 4: HH	ELOS-Pa	rtikelgröf	Benanalyse		
Zeitoasis: Start: Gültigkeit: Stopp: SYMP	Startknopf immer 3,005 Echtzeit Sympatec Gr System-Partil	nbH kel-Technik		Parameter 2: Parameter 3: Parameter 4: HH	ELOS-Pa	rtikelgröl	Benanalyse WINDOX 4		
Start: Gültigkeit: Stopp:	Startknopf immer 3,00s Echtzeit Sympatec Gr System-Parti	nbH kel-Technik		Parameter 2: Parameter 3: Parameter 4: HH	ELOS-Pa	rtikelgröf	3enanalyse WINDOX 4		
Start: Gültigkeit: Stopp:	Startknopf immer 3,00s Echtzeit Sympatec Gr System-Parti	ubH kel-Technik		Parameter 2: Parameter 3: Parameter 4: HH	ELOS-Pa	rtikelgröl	Benanalyse WINDOX 4		
Start: Gültigkeit: Stopp:	3,005 Echtzeit Sympatec Gr	ubH kel-Technik		Parameter 2: Parameter 3: Parameter 4: HIF	ELOS-Pa	rtikelgröl	Benanalyse WINDOX 4		
Zeitoasis: Start: Gültigkeit: Stopp: SYMP	Startknopf immer 3,005 Echtzeit Sympatec Gr System-Partil	nbH kel-Technik		Parameter 2: Parameter 3: Parameter 4: HIE	ELOS-Pa	rtikelgröf	Benanalyse WINDOX 4		
Zeitoasis: Start: Gültigkeit: Stopp: SYMP TPT HELOS	(H1473) & R	nbH kel-Technik ODOS, R5:	0.5/4.587	Parameter 2: Parameter 3: Parameter 4: HIE	ELOS-Pa	rtikelgröf 3:13:06.3280	Benanalyse WINDOX 4		
Zeitoasis: Start: Gültigkeit: Stopp: SYMP SYMP TPT HELOS	(H1473) & R	nbH kel-Technik ODOS, R5:	0.5/4.587	Parameter 2: Parameter 3: Parameter 4: HIE	ELOS-Pa 0.09.2013, 1	rtikelgröf 3:13:06,3280	Benanalyse WINDOX 4		

x10 = 6,03 μm	x50 = 67,32 μm	x90 = 273,60 µm	SMD	= 16,99 μm	VMD	=105,13 μm
x <sub>16</sub> = 10,77 μm	x <sub>84</sub> = 216,59 µm	x <sub>95</sub> = 412,25 μm	Sv	= 0,35 m²/cm³	Sm	= 452,71 cm²/g



Verteilungs	summe						
x0/µm	Q3/%	x0/µm	Q3/%	x0/µm	Q3/%	x0/μm	Q3/%
4,50	7,60	18,50	23,42	75,00	52,69	305,00	92,84
5,50	9,21	21,50	25,92	90,00	57,49	365,00	96,92
6,50	10,70	25,00	28,66	105,00	61,93	435,00	100,00
7,50	12,08	30,00	32,25	125,00	67,30	515,00	100,00
9,00	13,98	37,50	36,92	150,00	73,17	615,00	100,00
11,00	16,26	45,00	40,89	180,00	78,88	735,00	100,00
13,00	18,34	52,50	44,33	215,00	83,82	875,00	100,00
15,50	20,74	62,50	48,31	255,00	88,32		
Verteilungs	dichte (log.)						
$x_m/\mu m$	q3lg	xm/μm	q <sub>3</sub> lg	x <sub>m</sub> /μm	q₃lg	$x_m/\mu m$	q3lg
1,50	0,07	16,93	0,35	68,47	0,55	278,88	0,58
4,97	0,18	19,94	0,38	82,16	0,61	333,65	0,52
5,98	0,21	23,18	0,42	97,21	0,66	398,47	0,40
6,98	0,22	27,39	0,45	114,56	0,71	473,31	0,00
8,22	0,24	33,54	0,48	136,93	0,74	562,78	0,00
9,95	0,26	41,08	0,50	164,32	0,72	672,33	0,00
11,96	0,29	48,61	0,51	196,72	0,64	801,95	0,00
14,20	0,31	57,28	0,53	234,15	0,61		
Auswertung	: WINDOX 4.2.1.0	, HRLD		Produkt: LB1			



Verteilung	ssumme							
x0/µm	O3/%	x0/µm	O3/%	x0/µm	O3/%	x0/µm	O3/%	
4,50	6,87	18,50	20,32	75,00	48,69	305,00	89,32	
5,50	8,27	21,50	22,49	90,00	53,47	365,00	93,42	
6,50	9,56	25,00	24,91	105,00	57,75	435,00	96,93	
7,50	10,74	30,00	28,18	125,00	62,90	515,00	100,00	
9,00	12,36	37,50	32,63	150,00	68,59	615,00	100,00	
11,00	14,28	45,00	36,56	180,00	74,35	735,00	100,00	
13,00	16,03	52,50	40,05	215,00	79,77	875,00	100,00	
15,50	18,05	62,50	44,16	255,00	84,65			
Verteiluns	sdichte (log.)							
xm/HIII	ale	xm/um	ade	x=/um	ade	xm/mm	ada	
1.50	0.06	16.93	0.30	68.47	0.57	278.88	0.60	
4,97	0.16	19.94	0.33	82.16	0,60	333.65	0.52	
5,98	0,18	23,18	0.37	97.21	0,64	398,47	0,46	
6,98	0,19	27,39	0,41	114,56	0,68	473,31	0,42	
8,22	0,20	33,54	0,46	136,93	0,72	562,78	0,00	
9,95	0,22	41,08	0,50	164,32	0,73	672,33	0,00	
11,96	0,24	48,61	0,52	196,72	0,70	801,95	0,00	
14,20	0,26	57,28	0,54	234,15	0,66			
Answartun	- WINDOX 4 2 1 (	HRID		Produkt: I B	1			
Deschidiant	5. WINDOA 4.2.1.	, 111000		Dishter 7.80	alam Tamafala	term 1.00		
Revalidieru	шg.	12.40		Dicilie. 7,80	g/cm <sup>2</sup> , Formak	101. 1,00		
Referenzme	essung: 30.09-13:	13:49		DispMeth.:	LB2			
Kontaminat	tion: 0,00 %			C <sub>opt</sub> = 16,48	96			
Meßbeding	ung: md4			Benutzerpar	ameter:			
Zeitbasis:	1000,00 ms			Parameter 1:				
Start:	Startknopf		Parameter 2:	Parameter 2:				
Gültizkeit:	immer			Parameter 3:				
Stopp:	3.00s Echtzeit			Parameter 4				