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Safe handling of renewable fuels and fuel mixtures



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Abstract

The overall objective of this research project was to create new data on the safety-technical characteristics of renewable fuels, low-rank coals and mixtures of these. Knowledge of basic handling and safety-technical characteristics is of crucial significance for the design of handling equipment and safety systems, and for the assessment of explosion and fire hazards.

Ten biomass and lignite fuel samples and two fuel mixtures were included in the work programme. Extensive physical and chemical characterisation was performed for all samples. The reactivity of the combustible dusts was characterised by thermal analysis (DTA-TGA). Based on the thermal runaway temperatures of the dusts the samples are listed by order of reactivity.

Self-ignition properties of the fuels were studied at normal and elevated pressure (1 - 25 bar). The results of the self-ignition tests for the fuel samples are mainly in line with the reactivity tests carried out on DTA-TGA. The elevated pressure had a significant effect on the self-ignition temperature of all fuel samples. Partial inerting increased the self-ignition temperature, but to a rather low degree.

Dust explosion tests were performed in 1 m³ vessels and 20 litre spheres. Dust explosion parameters were determined both in normal conditions and at elevated temperature and pressure. A linear correlation was found between the maximum explosion pressure P_{max} and the initial pressure. The values measured for the rate of pressure rise, the K_{St} -value, were considerably more scattered. Most differences can probably be associated with diverse turbulence conditions inside the explosion vessel. In most cases, the lower oxygen concentration LOC slightly increased with increasing initial pressure. LOC decreases with increasing initial temperature, around 1 - 3 vol% per 100 °C temperature rise.

Suppression systems are frequently used in industry as a measure of explosion protection. The suppression tests were conducted in a heatable the 1 m³ vessel. Increasing the temperature makes the suppression of the dust explosion more demanding. Use of partly inert atmosphere led to an increased efficiency of the explosion suppression system.

Executive summary

Partners

VTT Energy was the co-ordinator of the project programme, and the partners were as follows:

Partner 1: VTT Energy (VTT), Finland

Partner 2: Laboratorio Oficial J. M. Madariaga (LOM), Spain

Partner 3: Institut National de l'Environnement Industriel et des Risques (INERIS), France

Partner 4: TNO Prins Maurits Laboratory (TNO), The Netherlands

Partner 5: DMT Gesellschaft für Forschung und Prüfung mbH, Institute of Fire and Explosion Protection (DMT), Germany

Objectives

The overall objective of this research project was to create new data on safety-technical characteristics of renewable fuels, low-rank coals and mixtures of these. The safety aspects of fuel storage, handling and feeding are an important issue in the development of new energy production technologies like pressurised IGCC and PFBC power production systems. Knowledge of basic handling and safety-technical characteristics of fuels and fuel mixtures is of crucial significance to the design of handling and feeding equipment, and safety systems, and to the assessment of explosion and fire hazards.

The technical objectives of the study are:

- to characterise selected fuels and fuel mixtures by physical and chemical analyses with regard to their safety-technical characters
- to study the spontaneous ignition behaviour of these fuels and fuel mixtures and to assess the necessary preventive actions in various ambient conditions
- to determine the explosion characteristics of the most critical dusts and dust mixtures in both atmospheric conditions and at elevated pressure and temperature, being essential for the design of protective devices
- to experimentally verify explosion-prevention concepts in both ambient and elevated conditions (inertisation, suppression).

Physical and chemical characterisation

Ten fuel samples and two fuel mixtures were included in the work programme. The selected samples were divided into four main groups:

Wood fuels and wood wastes: Wood dust, bark, forest residue and Spanish pine.

Agricultural residues and energy crops: Barley straw, rapeseed straw, Miscanthus and Sorghum.

Low-rank coals: German lignite and Spanish lignite.

Fuel mixtures: Mixture of wood dust and German lignite; mixture of barley straw and Spanish lignite

All fuel batches, ranging from 50 to 1800 kg/sample, were dried and milled to a particle size of < 0.2 mm to obtain reasonably homogenous samples for testing. Chemical characterisation included proximate and ultimate analyses. Extensive physical characterisation was performed by all partners, including particle size distribution, density, thermal and electrical conductivity and ignition properties.

Reactivity of fuel samples

The reactivity of the combustible dusts was characterised by thermal analysis employing a Differential Thermal Analysis (DTA) and a Thermo-Gravimetric Analysis (TGA). The objective of this task was to rank the fuel samples according to their tendency to self-ignition. The tests at ambient pressure were carried out by INERIS and analyses at elevated pressure by VTT.

Based on the thermal runaway temperatures of the dusts the samples are listed by order of reactivity. Classification of the dust samples indicates that the low-grade coals are clearly more reactive than the biomass samples. The mixtures also show a higher reactivity than the pure biomass fuels. Increasing the pressure leads to a higher oxygen partial pressure, increasing the aggressiveness of the oxidation. This results in a higher reactivity of the sample at elevated pressures.

A statistical treatment of the results was used to create a predictive criterion based on simple tests and analyses, aimed at the prediction of the theoretical reactivity of a determined fuel as a function of its chemical composition. The statistical treatment of the developed data base resulted in four significant multi-

ple regressions with a confidence level above 95%. To have a more comprehensive assessment and a more reliable result, a “standard” definition for the reactivity would be needed and testing of considerably more fuel samples.

Self-ignition tests

Low-temperature reactions of organic substances with atmospheric oxygen may lead to self-heating. It is well-known that the self-ignition temperature of a dust deposit is a function of the size of the deposit; i.e., the self-ignition temperature falls when the size of the deposit (or storage volume) increases.

Self-ignition properties of the fuels were studied at normal and elevated pressure (1 - 25 bar) by VTT and INERIS. The following conclusions were drawn from the study:

- The results of the self-ignition tests for the fuel samples are mainly in line with the reactivity tests carried out on DTA-TGA. The lignites are the most reactive fuels both at ambient and elevated pressure. The pure wood fuels, wood and Spanish pine, were least reactive with regard to spontaneous ignition. The wood wastes, bark and forest residue are more reactive than the agricultural straw residues. The different straws showed very similar self-ignition temperatures.
- The mixtures of lignites and biomasses were in reactivity comparable to the most reactive pure lignites.
- The elevated pressure had a significant effect on the self-ignition temperature of all fuel samples: the self-ignition temperature of the fuel fell when the pressure was elevated.
- Partial inerting, i.e., decreasing the oxygen concentration of the ambient air atmosphere, increased the self-ignition temperature, but to a rather low degree. To establish significantly safer conditions in fuel storage, an inert atmosphere with an oxygen content well below 7% is obviously required.

Explosion test methodology

The explosion tests are usually performed in ambient conditions in a 1 m³ pressure vessel according to ISO 6184/1 standard procedure, or in a smaller 20 litre

laboratory sphere. At high initial pressure and for “difficult” dust it is necessary to define and develop new explosion test procedures. This task included the following development work:

- development of a dispersion nozzle that makes it possible to disperse a large amount of difficult flowing dusts
- definition of the injection procedure at elevated pressure.

The tests were carried out simultaneously in the TNO 20 litre sphere and in the LOM 1 m³ vessel. A multifactorial experimental design was used to optimise the experimental work. The conclusions were:

- TNO was forced to disperse the dust when it was already on the bottom of the sphere. A nozzle type was designed which directed the air flow during the injection over the bottom of the sphere.
- LOM used the conventional method of introducing the dust from an external dust container. A rebound nozzle was chosen for dispersion of the dust into the explosion vessel.

Dust explosion tests

The knowledge of the explosions parameters at high initial pressure and temperature is fundamental for the design of safety measures at IGCC and PFBC power plants. The objective of this task was to experimentally determine the explosion indices of the selected fuels and fuel mixtures. Dust explosion tests were performed by four of the partners: TNO (20 litre sphere), LOM (20 litre sphere and 1 m³ vessel), DMT (1 m³ vessel) and INERIS (1 m³ vessel). The tests were performed both in normal conditions and at elevated temperature and pressure. Dust explosion parameters, maximum explosion pressure P_{\max} , rate of pressure rise described by the K_{St} -value, and lower oxygen concentration LOC were determined.

A linear correlation was found between the maximum explosion pressure and the initial pressure. The maximum explosion pressure is directly proportional to the initial pressure. The results also show a fairly good conformity considering the different tests methods. Further, the explosion pressures of all fuels fall in the same order of magnitude.

An approximately linear relation also exists for the rate of pressure rise, the K_{St} -value. There is, however, a large discrepancy in the slope of the lines for the same fuel samples. Most differences in the measured K_{St} -values can probably be associated with diverse turbulence conditions inside the explosion vessel. Various turbulence levels may be ascribed to different equipment and methods, and generally to the inhomogeneous nature of the fuel samples. The influence of turbulence on the K_{St} -value and more generally the violence of the explosion phenomenon should be further investigated, especially with regard to standardisation work.

In most cases, the lower oxygen concentration LOC slightly increased with increasing initial pressure. The tendency is clear for wood but for the lignites and the mixture the effect of initial pressure is more irregular. LOC decreases with increasing initial temperature. For all powders tested this decrease was more or less the same, around 1 - 3 vol% per 100 °C temperature rise.

Suppression of dust explosions

Suppression systems are frequently used in industry as a measure of explosion protection. After static or dynamic pressure sensors detect an explosion, the control unit activates the outlet valves of one or more containers filled with suppressant agent. The suppressant agent is injected into the vessel and the explosion is stopped at an early stage. The reduced explosion overpressure P_{red} is far below the maximum explosion overpressure.

The suppression tests were conducted by DMT in a heatable the 1 m³ vessel. The explosion suppression system comprised a 12.3 litre HRD container filled with monoammonium phosphate (4 kg and 8 kg) and a static pressure sensor. After detecting the explosion at the activation overpressure P_A , the suppressant was blown into the 1 m³ vessel through two nozzles with a diameter of 1 inch (25.4 mm). The reduced explosion overpressure P_{red} was determined in respect of the activation pressure P_A at 4 bar initial pressure and temperatures of 20 °C and 150 °C. Wood and German lignite were used as dust samples. Increasing the temperature makes the suppression of the dust explosion more demanding (low activation pressure). Use of partly inert atmosphere, e.g. by reducing the oxygen concentration from 21% to 17%, led to an increased efficiency of the explosion suppression system.

Preface

This report presents the results of the project “Safe handling of renewable fuels and fuel mixtures” (JOR3-CT95-0032) in part financed by the European Commission within the framework of the Joule 3 Programme. The contributors to the project have been:

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List of symbols

A	surface of the electrodes in the test cell
b	half edge length
C _p	specific heat
D ₅₀	mean particle size described by the sieve mesh dividing the sample into two equal parts
(dP/dt) _{max} or MRPR	maximum rate of pressure rise
E _a	activation energy
I	current through the sample
K _{St}	value constant characterising explosibility
l	distance between the two electrodes
LOC	lower oxygen concentration
M and N	constants depending on the physico-chemical properties of dust (heat of reaction, apparent activation energy, density, heat capacity, thermal conductivity, oxygen concentration, frequency factor)
MEC	minimum explosive concentration
MIE	minimum ignition energy
MIT _c	minimum ignition temperature for a cloud
MIT _l	minimum ignition temperature for a layer
n	reaction order.
N	E/R where E is apparent activation energy and R is gas constant
P _{O₂}	partial pressure of oxygen in the test condition
P ₀	partial pressure of oxygen in ambient air
P _i	initial pressure
P _{inj}	injection pressure
P _{red}	reduced (suppressed) explosion pressure
p _s	pressure load corresponding to the minimum yield strength of the vessel
P _{max} or MEP	maximum explosion pressure
R	resistance of the powder
r	characteristic dimension of the deposit in meters (half-edge length of cube, radius of sphere or long cylinder)
r ₂	maximum oxidation rate, i.e., 0.017%/s
r _m	rate at the temperature in the middle of ΔT (T _{r2} - T ₀)

t	time necessary for the temperature at the centre to reach $T = [T_a - 0.86 (T_a - T_0)]$
T_a	stabilised oven temperature
T_c	self-ignition temperature in K (or critical temperature)
T_m	temperature in the middle of ΔT ($T_{r_2} - T_0$)
T_0	initial sample temperature
T_{r_2}	temperature at which r_2 was achieved
V	potential applied
Z	frequency factor

Greek symbols

α	Thermal diffusivity
λ	thermal conductivity
ρ	bulk density
ρ_E	electrical resistivity
δ_c	form factor for the storage volume (2.6 for cube, 2 for infinite cylinder, 0.88 for slab)
$\Delta\Theta$	self-ignition (control) temperature corresponding to INERIS definition

List of acronyms

DMT	Gesellschaft für Forschung und Prüfung mbH, Institute of Fire and Explosion Protection, Germany
LOM	Laboratorio Oficial J. M. Madariaga, Spain
TNO	Prins Maurits Laboratory, The Netherlands
INERIS	Institut National de l'Environnement Industriel et des Risques, France
VTT	Technical Research Centre of Finland, VTT Energy, Finland

1. Introduction

The use of renewable fuels, agricultural residues and low-grade fuel mixtures for producing electricity and/or heat is being promoted within the European Union for environmental and agricultural-political reasons. New atmospheric biomass combustion, gasification and pyrolysis technologies are being developed for small and medium-scale power production. New large-scale energy production technologies like pressurised IGCC and PFBC power production systems are also being demonstrated. The use of renewable fuels and fuel mixtures sets new requirements on the pretreatment and feed of fuels into the gasification or combustion process. These fuels require extensive safety measures in handling due to their inhomogeneous character, dustiness, and flammability.

Dust explosion hazards in atmospheric pressure bins and handling equipment are usually provided for by arranging the discharge of explosion pressure through explosion discs or relief vents and by using explosion suppression systems. The design phase of these safety measures requires input data on safety-technical properties of new fuels and fuel mixtures created in experimental activities.

Total inertisation has been applied in pressurised fuel feeding and handling, which, in the demonstration and industrial size class, results in extremely high operating costs. By using partial inertisation with, for example, flue gases a sufficient level of operational safety may be achieved at reasonable costs. Therefore, the knowledge of the level of the limiting oxygen concentration required for preventing self-ignition and dust explosions at both atmospheric and elevated pressure is of significant importance.

Numerous bulk fuels (coal, biomass, wastes...) continuously produce heat due to oxidation processes. Low-temperature reactions with atmospheric oxygen lead to self-heating. With a sufficient material volume and a low heat conductivity, heat build-up and spontaneous ignition may occur. Spontaneous heating and ignition phenomena are especially hazardous in storage bins and process equipment, as smouldering material deposits are potential ignition sources of more extensive fire and dust explosions.

Existing information on the assessment of risk and prescription of safety measures is largely concerned with spontaneous ignition risks and dust explosions

initiated at atmospheric pressure and temperature. There is, however, hardly any information available on ignition and explosion properties of renewable fuels and biomass-coal mixtures. As the large-scale power industry is changing over to processes operating at high pressures and temperatures, the knowledge of safety-technical basic characteristics of fuels under these elevated conditions is of essential significance when designing handling and feeding equipment, planning safety systems and instructions and evaluating fire and explosion hazards [1].

2. Objectives

The overall objectives of this research project were to create new data on the safety-technical characteristics of renewable fuels, low-rank coals and mixtures of these. The technical objectives of the study were:

- to characterise the selected fuels and fuel mixtures (biomass, wood wastes, low rank coals) by physical and chemical analyses with regard to their safety-technical characters;
- to study the spontaneous ignition behaviour of these fuels and fuel mixtures and to assess the necessary preventive actions in various ambient conditions;
- to determine the explosion characteristics of the most critical dusts and dust mixtures in both atmospheric conditions and at elevated pressure and temperature, being essential for the design of protective devices;
- to experimentally verify explosion-prevention concepts under both ambient and elevated conditions (inertisation, suppression);
- to create a criterion based on simple analyses to assess the reactivity of the fuels with regard to their safety-technical properties.

3. Project organisation

VTT Energy was co-ordinator of the project programme and the partners are listed below. The abbreviations used to identify the partners are put in brackets.

Partner 1: VTT Energy (VTT), Finland

Partner 2: Laboratorio Oficial J. M. Madariaga (LOM), Spain

Partner 3: Institut National de l'Environnement Industriel et des Risques (INERIS), France

Partner 4: TNO Prins Maurits Laboratory (TNO), the Netherlands

Partner 5: DMT Gesellschaft für Forschung und Prüfung mbH, Institute of Fire and Explosion Protection (DMT), Germany

4. Work programme

The work programme was divided into five tasks and several subtasks. The scientific and technical description of the project mainly follows this division.

- TASK A: Characterisation of fuels and fuel mixtures
 - Sample selection
 - Parameter selection
 - Physical & chemical characterisation
 - Elaboration of predictive criterion
- TASK B: Explosion test methodology
 - Definition of test conditions
- TASK C: Self-ignition tests
 - Tests at ambient pressure
 - Tests at elevated pressure
 - Inertisation tests
 - Time factor evaluation
- TASK D: Dust explosion tests
 - Tests at normal conditions
 - Tests at high pressure
 - High temperature and pressure tests
- TASK E: Explosion protection
 - Inerting tests
 - Suppression tests
 - Combined protection systems

5. Characterisation of fuels and fuel mixtures

With regard to safe fuel handling, it is of great importance to assess the combustibility of fuels in terms of their reactivity (reaction with oxygen in air to produce an exothermic process), flammability and explosibility. Physical and chemical properties were determined using standard procedures.

5.1 Sample selection and identification

Ten fuel samples and two fuel mixtures were included in the work programme. The selected samples were divided into four main groups:

Wood fuels and wood wastes:

1. Wood dust, representing a clean wood fuel. 90% birch and 10% spruce.
2. Bark, left over from debarking of pinewood and used extensively by the paper and pulp industry as fuel.
3. Forest residue, wood chips prepared from small wood, branches and tops containing green parts, bark and some stemwood.
4. Spanish pine, a Spanish wood fuel used, e.g., by the company Union Fenosa in preparing wood-based oil with the Waterloo Flash Pyrolysis Process.

Agricultural residues and energy crops:

5. Barley straw, representing a large agricultural energy resource. Produced and collected in Luvia, Spain.
6. Rapeseed straw, with combustion properties differing from those of normal straw.
7. Miscanthus, produced and collected from a 4-year old culture at Ter Avil, the Netherlands.
8. Sorghum, produced and collected in France.

Low rank coals:

9. German lignite, typical Rhenish brown coal.
10. Spanish lignite, poor coal obtained from open pit mines in Meirama, in the North-West of Spain.

Fuel mixtures:

11. Mixture of wood dust and German lignite
12. Mixture of barley straw and Spanish lignite

Three fuels, one from each group, were chosen as secondary samples. These were wood dust, barley straw and German black lignite. The most extensive test programme was performed for these fuels, including, e.g., high-pressure explosion tests, inerting tests and suppression tests in 1 m³ explosion test facilities, requiring a very large amount of dust to be prepared (600 to 1 800 kg per sample). For the mixtures a ratio of about 25 kg biomass/75 kg coal was used to give a volume ratio of about 1/1.

To prepare suitable dusts for the explosion tests it was decided to mill and sieve the fuels to a particle size of 50% below 200 µm and dry them to a moisture content of less than 10%. The sample preparation and delivery proved to be a time and effort consuming task, which, however, had to be performed with a great accuracy to create a basis for reliable testing and repeatable results.

According to the ISO 6184/1 [2] standard the particle size of the dust used in the explosion test should be below 68 µm to obtain the maximum explosion effects. To produce dust of such a fine grade by milling and sieving the biomass samples was found to be technically a too complicated and time-consuming task. Therefore, it was decided to use the criteria given above for the particle size distribution. On the basis of previous experience it was anticipated that a dust of this particle size could be readily dispersed into the test vessel. The coarser particle size was also considered to be representative for dust generated in practical fuel-handling processes.

5.2 Physical characterisation

5.2.1 Particle size distribution

The particle size and moisture content were the two primary properties requiring a lot of pretreatment of the samples to fit into the desired limits mentioned above. The particle size distribution was determined by normal sieving procedure by VTT, LOM, TNO and DMT, by INERIS using Malvern 2600 C laser system following the French standard NF X 11-666, and by VTT using a dry Laser beam based method. The method of mechanical sieving was evaluated by sieving a number of fuels for 10, 15, 18 and 21 minutes. The results suggest a minimum sieving time of 15 minutes.

The particle size distributions of all fuels determined by VTT are shown in Figure 1. The sieving time was 15 minutes. The mean particle size D_{50} , described by the mesh of the sieve dividing the fuel sample in two equal parts. Comparison between the results of the different laboratories, Table 1, shows in general an acceptable

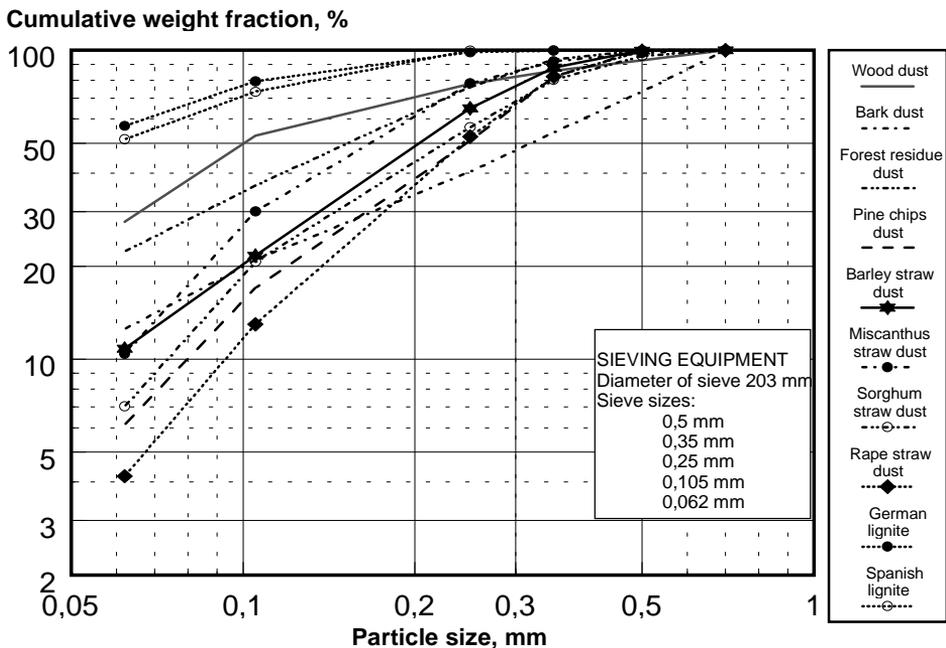


Figure 1. Particle size distribution of all fuel samples (VTT).

Table 1. Comparison of mean particle size D_{50} obtained by different laboratories.

DUST SAMPLE	VTT sieve, 15 min D_{50}/mm	VTT Laser beam D_{50}/mm	LOM sieve, 10 min D_{50}/mm	INERIS Malvern D_{50}/mm	DTM sieve D_{50}/mm	TNO sieve D_{50}/mm
Wood	0.10	0.08	0.24	0.095	0.073	0.311
Bark	0.32	0.08	0.17	0.057		0.321
Forest residue	0.15	0.23	0.21	0.102		0.189
Spanish pine	0.24	0.32	0.25	0.178		0.247
Barley straw	0.20	0.12	0.25	0.253	0.175	0.48
Mischanthus	0.17	0.13	0.21	0.143		0.179
Sorghum	0.23	0.12	0.25	0.178		0.223
Rapeseed straw	0.24	0.22	0.35	0.318		0.251
German lignite	0.06	0.03	0.07	0.058	0.039	0.113
Spanish lignite	0.05	0.05	0.06	0.040	0.050	0.066
Mixture 1			0.10			
Mixture 2			0.10			

deviation in mechanical sieving, considering the inhomogeneous nature of biomasses. The Malvern and Laser beam method, however, produced smaller D_{50} values for the fibrous biomass fuel samples. It should be noted that the form of the particles can influence the results obtained by the laser technique, especially if they are not spherical.

5.2.2 Density of dust samples

The density of the fuel samples was determined using three different approaches. The apparent density (or bulk density) corresponds to the overall volume occupied by a given mass of powder, including the pore and interstice volumes. The apparent density was determined to give a rough idea of the values encountered. A one-litre cubic volume was filled up with a measured amount of powder sample without tamping.

The “compacted” density of the fuel samples was measured with a so-called FEM method (Federation Europeenne de la Manutention), a method generally used by machine manufacturers for dimensioning equipment for bulk materials. The compacted bulk density correlates well with the real volume weight of the fuel in bins and intermediate stores.

The true density of a solid material is based on the volume occupied by its mass excluding the volume of all pores. The true density of the samples was determined with an air pycnometer (LOM) and a helium pycnometer (INERIS), in which the volume of gas present in the test chamber is compressed by a piston with a known displacement volume. The "free" volume is calculated using the pressure difference and the known volume of displacement. The results of LOM ranged 1 340 - 2 070 kg/m³, while those determined by INERIS were more consistent, between 1 340 - 1 430 kg/m³.

The bulk density of wood dust, barley straw and rapeseed straw dusts ranged 120 - 190 kg/m³. The other wood fuels were heavier, as were also the straws of Miscanthus and Sorghum, which have a more woody-like stem than the normal cereals. The range of bulk density of these fuels was 214 - 350 kg/m³. The bulk density of the low-grade coals and the mixtures was significantly higher, 320 - 677 kg/m³. The determined bulk densities are shown in Figure 2.

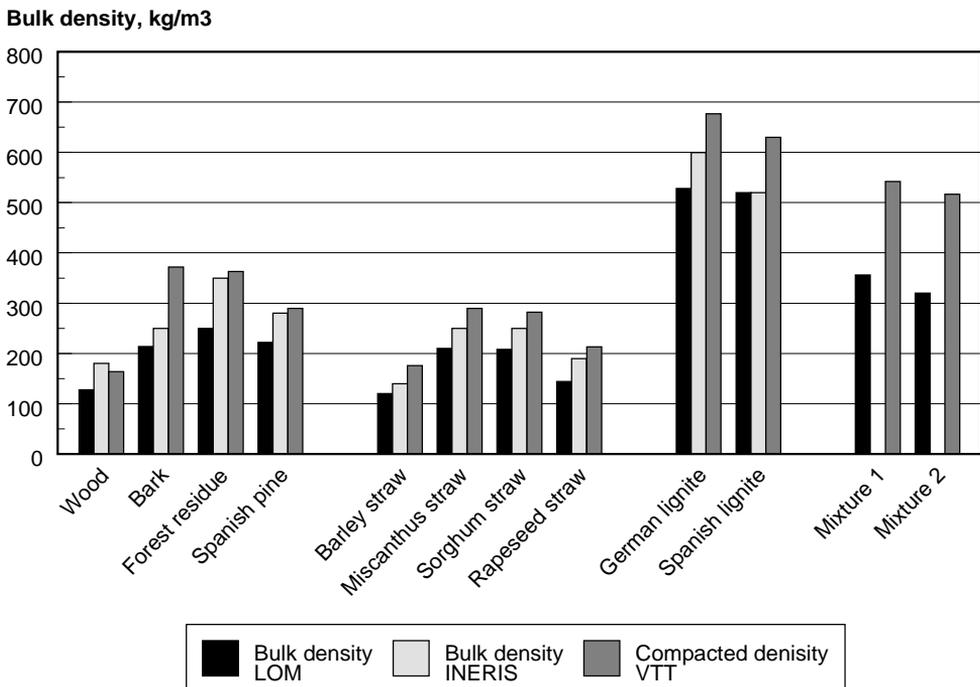


Figure 2. Bulk density of fuel samples.

5.2.3 Particle form

A scanning electron microscope was used to take pictures of the various samples. The photographs were taken with a magnification of 71.5x (Appendix 1, Figures 1 - 10). It can be seen that the woody samples are very fibrous with clusters of very small particles. The agricultural dusts are somewhat fibrous whereas the lignites are more granular.

5.2.4 Thermal conductivity

The thermal conduction coefficient of the dust deposits was estimated by IN-ERIS using the following procedure: A one-litre cubic volume was filled with the dust sample without tamping, tapping, or using any other procedure that could increase the density, and placed in an isothermal oven where the temperature was stabilised and lower than the self-ignition temperature for that volume. A thermocouple placed in the centre of the volume was used to record the temperature as a function of time [3].

The thermal conductivity was estimated following the equation given below (as a result of nondimensional Fourier number):

$$\alpha t / b^2 = 0.1 \quad (1)$$

where α is thermal diffusivity

b half edge length

T_a stabilised oven temperature

T_o initial sample temperature

t time necessary for the temperature at the centre to reach $T = (T_a - 0.86(T_a - T_o))$.

By taking the values of the bulk density ρ , the values of α , and an approximation of the values for the specific heats of the dusts C_p , the values of thermal conductivity λ for the ten samples can be estimated:

$$\lambda = \alpha \cdot \rho \cdot C_p \quad (2)$$

where λ is thermal conductivity

ρ bulk density

C_p specific heat.

The thermal conductivity of two samples (wood dust and German lignite) was measured by VTT using the standard method ISO 8301(1991): "Determination of steady-state thermal transmittance and related properties - Heat flow meter apparatus". Compacted fuel samples (FEM density) were used in these measurements. All results are presented in Table 2. The difference between the calculated and measured values for wood dust may depend on the different bulk density values used in the basic measurements.

Table 2. Thermal conductivity of the dust samples.

Dust sample	Ta °C	To °C	T °C	Time hr	α m ² /s	ρ kg/m ³	Cp J/kgK	INERIS λ W/m*K	VTT λ W/m*K
Wood	170	22	43	0.27	2.6·10 ⁻⁷	180	1 675	0.078	0.0518
Bark	140	26	43	0.35	2.0·10 ⁻⁷	250	1 675	0.083	
Forest residue	145	26	43	0.46	1.5·10 ⁻⁷	350	1 675	0.089	
Spanish pine	165	21	42	0.29	2.4·10 ⁻⁷	260	1 675	0.105	
Barley straw	165	19	40	0.22	3.2·10 ⁻⁷	140	1 256	0.056	
Miscanthus	160	20	40	0.24	2.9·10 ⁻⁷	250	1 256	0.091	
Sorghum	160	21	41	0.245	2.8·10 ⁻⁷	250	1 256	0.089	
Rapeseed straw	165	22	43	0.31	2.3·10 ⁻⁷	190	1 256	0.054	
German lignite	105	20	32	0.66	1.1·10 ⁻⁷	600	1 256	0.080	0.0863
Spanish lignite	110	18	31	0.74	0.9·10 ⁻⁷	520	1 256	0.061	

5.2.5 Electrical conductivity

The electrical conductivity was determined by TNO according to British Standard 5958 Part 1. A Perspex resistivity cell is filled with the sample. The test cell is shown in Figure 3. Inside the cell two round plates with a diameter of 50 mm act as electrodes. The electrodes are 5 mm apart. The measured resistance depends on the compaction of the sample. To create as constant a compaction as possible the sample is compacted by tapping the test cell gently on the table. A potential of 500 V is applied to one of the electrodes and the resulting current through the sample is measured. Then the potential is raised to 1000 V and the measurement is repeated. This test is performed in triplicate. The volume resistivity of the sample can be calculated with the following equation:

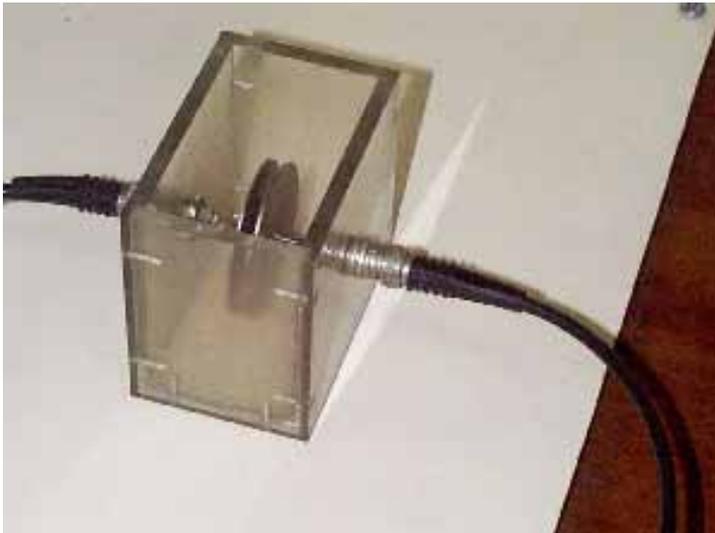


Figure 3. The powder resistivity cell, TNO.

$$\rho_E = R \cdot A/l = V/I \cdot A/l \quad (3)$$

where ρ_E is electrical resistivity
A surface of the electrodes in the test cell
l distance between the two electrodes
R the resistance of the powder
V the potential applied
I the current through the sample.

For the applied test cell the equation will be:

$$\rho_E = 0,393 \cdot V/I \quad (4)$$

With respect to the results it can be said that samples with a resistivity till $10^4 \Omega \cdot m$ are known as conductive, samples with a resistivity higher than $10^6 \Omega \cdot m$ are known as resistive. Table 3 shows the results of the physical characterisation.

Table 3. Physical characteristics and electrical resistivity, TNO.

Dust sample	Sample particle size (D_{50}) μm	Relative humidity %	Electrical resistivity $\Omega \cdot m$
Wood	311	4	$1.5 \cdot 10^{13}$
Bark	321	3	$4.4 \cdot 10^{12}$
Forest residue	189	5	$2.8 \cdot 10^{12}$
Spanish Pine	247	7	$1.9 \cdot 10^{11}$
Barley Straw	480	6	$6.5 \cdot 10^9$
Miscanthus	179	5	$1.1 \cdot 10^{11}$
Sorghum	223	6	$9.4 \cdot 10^{10}$
Rapeseed straw	251	10	$6.2 \cdot 10^8$
German lignite	66	7	$5.8 \cdot 10^{11}$
Spanish lignite	113	3	$2.2 \cdot 10^{13}$

5.3 Chemical characterisation

The chemical analyses of the normal fuel properties of the fuel samples were carried out and the proximate and ultimate analyses are presented in Tables 4 and 5. The ash content of the low-grade brown lignite from Spain is considerably higher than that of the other fuels. The ash content of the agro-fuels is also in general higher than that of the woody fuels.

Table 4. Proximate analyses (LOM).

Dust sample	Humidity %	Ash %	Volatile matter %	Lower heating value (LHV) MJ/kg
Wood	1.65	0.39	81.87	18.16
Bark	5.75	1.70	68.55	19.83
Forest residue	5.30	2.55	73.25	19.73
Spanish pine	7.40	0.25	78.15	18.52
Barley straw	15.35	4.20	63.30	17.18
Miscanthus	7.00	1.00	73.20	18.32
Sorghum	6.55	5.25	70.40	17.59
Rapeseed straw	11.50	5.40	50.95	17.06
German lignite	9.85	3.85	46.10	23.52
Spanish lignite	16.65	17.10	36.60	17.23
Mixture 1	9.50	3.10	53.00	22.34
Mixture 2	14.65	14.10	47.50	17.87

Table 5. Ultimate analyses (LOM).

Dust sample	S %	C %	H %	N %
Wood	0.01	47.31	6.25	0.18
Bark	0.03	50.87	5.96	0.26
Forest residue	0.05	49.00	6.24	0.81
Spanish pine	0.01	47.35	6.43	0.10
Barley straw	0.08	43.65	6.11	1.21
Miscanthus	0.00	46.01	6.21	0.43
Sorghum	0.06	49.39	6.05	1.30
Rapeseed straw	0.38	40.01	6.27	0.70
German lignite	0.30	58.50	5.30	0.83
Spanish lignite	2.30	43.71	5.16	0.70
Mixture 1	0.19	55.48	5.64	0.62
Mixture 2	1.65	43.58	5.41	0.74

5.4 Ignition properties of dust samples

Several safety-technical properties of the selected fuels with respect to their behaviour on hot surfaces and as dust clouds were determined. The ignition sensibility of a substance can be evaluated through the determination of the following parameters:

- Minimum ignition temperature: this is the lowest temperature at which the ignition of a sample occurs. The test may be conducted using a sample in the form of a cloud (MIT_c) or a layer (MIT_l).
- Minimum ignition energy (MIE): lowest energy stored in a capacitor which upon discharge is sufficient to produce ignition of the most easily ignitable dust mixture with air under specified tests conditions.

Most of the ignition properties of the samples were determined by LOM. To obtain a comparison of two different laboratories it was decided that TNO should determine the minimum ignition energy as well. The minimum ignition energy was determined with MIKE 3.2, a commercially available Hartmann-type apparatus.

The Hartmann-type apparatus consist of a glass tube with a volume of 1.2 litre is used [4]. The tube is closed at the top with a metal lid. Three electrodes are placed at 0,1 m from the bottom. The dust sample is placed on the bottom of the tube and is dispersed with air from a pressure vessel. The pressure vessel of 60 millilitres is filled with air to 7 bar. Some (delay)time after suspending the dust a capacitor is discharged at the electrodes, resulting in a spark and possibly an ignition. By moving one of the electrodes towards the other electrode at high speed, the moment of spark discharge can be determined. Spark delay times vary from 60 to 180 ms. In the spark circuit an inductance is inserted to increase the igniting power of the spark. The ignition energy recorded is the gross energy stored in the capacitor. The dust concentration is varied until the minimum ignition energy is found. At the highest energy level where no ignition occurs, at least 10 tests are performed. The tests are performed at room temperature.

In the determination of the minimum ignition energy the actual explosion effect is not studied. The gentle way of dust dispersion used in the Hartmann apparatus favours ignition (excessive turbulence counteracts the ignition process). There-

fore the Hartmann apparatus has proved to be suitable for minimum ignition energy measurements.

The minimum ignition energy depends strongly on the initial temperature: increasing the temperature causes the minimum ignition energy to decrease. Dust properties (particle size, moisture content, etc.) can also influence the minimum ignition energy markedly. The presence of flammable vapours in a dust-air mixture reduces in most cases the minimum ignition energy of the resulting "hybrid" mixture, especially when the pure dust-air mixture is difficult to ignite. In this test the minimum ignition energy is determined under atmospheric test conditions at room temperature.

The minimum ignition energy is used to assess the risk of ignition by various types of electrostatic discharges. For the assessment of mechanical sparks the minimum ignition temperature of the dust cloud must be known as well.

Since the exact energy that is just able to ignite a certain mixture is very difficult to find and would require a lot of effort, the minimum ignition energy is usually reported as a value in between two values. The low energy value is not able to ignite the dust/air mixture and the high energy value is able to ignite the dust/air mixture.

The minimum ignition temperature of a dust layer (MIT_l) is determined on a heated plate. A 5 mm thick dust layer is placed on the plate and the minimum temperature is measured at which a smouldering combustion of the sample is initiated within two hours [5].

The minimum ignition temperature in a dust cloud (MIT_c) is measure by dispersing a dust cloud in a heatable oven [5].

Table 6 shows the ignition properties of the dust samples determined by LOM and TNO. Considering the technical difficulties of determining an exact value of the MIE, the results obtained by LOM and TNO were reasonably well in line.

Table 6. Ignition properties of the dust samples.

Dust sample	MIT ₁ °C	MIT _c °C	MIE (LOM) mJ	MIE (TNO) mJ
Wood	340	420	60	30 < MIE < 100
Bark	310	460	50	30 < MIE < 100
Forest residue	300	440	50	30 < MIE < 100
Spanish pine	350	460	450	MIE > 1000
Barley straw	310	440	620	MIE > 1000
Miscanthus	290	460	260	MIE > 1000
Sorghum	300	440	1400	MIE > 1000
Rapeseed straw	330	420	>1000	MIE > 1000
German lignite	230	420	30	30 < MIE < 100
Spanish lignite	240	400	450	300 < MIE < 1000
Mixture 1	260	440	70	30 < MIE < 100
Mixture 2	260	400	700	300 < MIE < 1000

5.5 Reactivity and self-ignition

The reactivity of the combustible dusts was characterised by thermal analysis using Differential Thermal Analysis (DTA) and a Thermo-Gravimetric Analysis (TGA) [6, 7]. The objective of this task was to rank the fuel samples according to their tendency to self-ignition. The tests at ambient pressure were completed by INERIS and analyses at elevated pressure by VTT. The methods and, in particular, the sample size used in the thermobalance measurements differed substantially, which should be considered in the direct comparison and interpretation of the results.

Whether self-heating will occur or not in a given dust under a specific set of conditions depends on several parameters:

- physical (particle size and form, specific surface area, pore shape and volume, true and apparent density, heat capacity, phase transitions, etc.)
- chemical (chemical and molecular composition, moisture content, surface structure, heat of combustion, etc.)

- reactional (reactivity with oxygen or water, decomposition, thermal stability, etc.)
- environmental (storage or deposit shape and volume, temperature, pressure, oxygen and vapour concentrations in the atmosphere, residence time, related heat and mass transfer restrictions).

One approach to the problem would be to characterise the influence of each parameter separately. INERIS has developed a methodology to assess the self-ignition tendency of a substances conducting the minimum number of tests to get meaningful information that can directly be used by design and safety engineers [7]:

1. Thermal stability tests in air (DTA-TGA) to rank the dusts in terms of reactivity to oxygen. Facilitates the first estimation of self-heating risks with respect to other similar and known dusts found in the same conditions.
2. Isothermal oven to determine the relationship between the self-ignition temperature and storage or deposit size.
3. Application of the thermal explosion theory of Frank-Kamenetskii makes it possible to extrapolate the results of the isothermal tests so that we can estimate the self-ignition temperature for any volume of dust [8, 9]. This is a lumped parameter model used on a semi-empirical level, integrating the physical, chemical, and reactional parameters cited above.
4. An adiabatic calorimeter can be used to establish the thermal kinetics of the system to estimate the time required to go from the initial state to thermal explosion (ignition) under adiabatic conditions.

5.5.1 Test procedure (INERIS)

About 4 g of the powder to be tested is placed into one of the sample holders made of a fine metal screen and the same volume of powdered alumina put into another identical sample holder [7]. The sample is then placed in a vertical tubular oven and its weight is recorded as a function of time as the oven is heated up from room temperature to 600 °C (or higher) at a rate of 0,08 K/s. The temperatures of the sample and of inert alumina (control substance) are recorded at the same time by placing a thermocouple at the centre of each substance. During the experiment, air at a flow rate of 0,12 dm³/s, preheated to the temperature of

the oven, is led through the oven to provide the oxygen necessary for oxidation. By comparing the difference in temperature between the sample and inert substance as a function of the oven temperature, as they are heated up in the oven, exothermic or endothermic phenomena on a semi-quantitative basis can be identified.

5.5.2 Results of DTA-TGA tests

From the DTA-TGA curves (temperature difference versus control temperature) the following temperatures are identified (Figure 4):

- The control temperature at which the rate of weight loss becomes significant, representing devolatilisation by evaporation, pyrolysis, or combustion.
- The control temperature $\Delta\Theta$ at which the temperature difference between the sample and the inert material is $\Delta T= 50$ K, which corresponds to the INERIS definition of thermal runaway (self-ignition temperature) in this experiment.

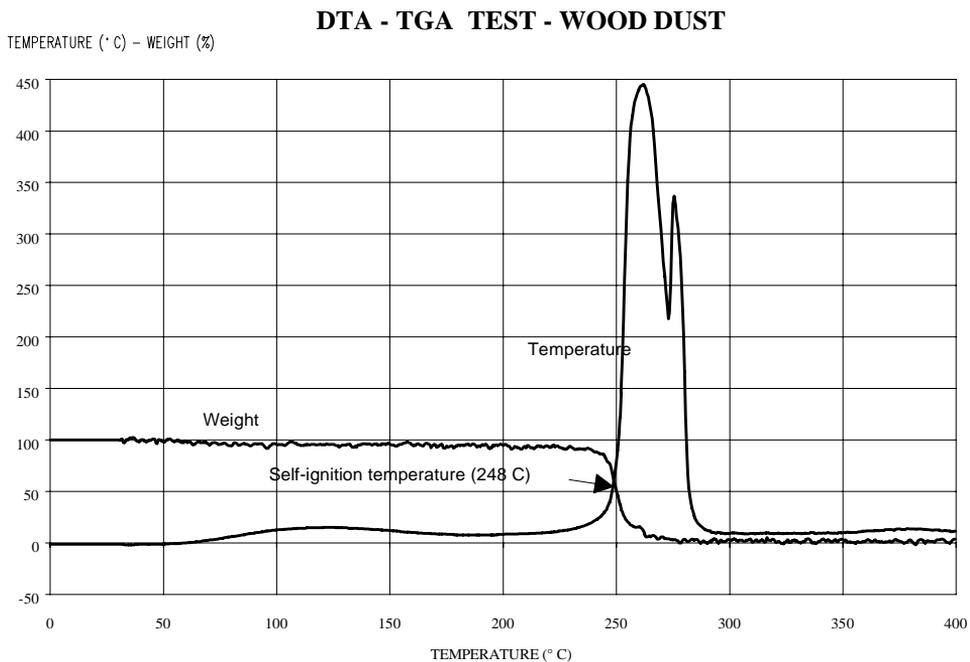


Figure 4. DTA-TGA measurement of wood dust (INERIS).

INERIS rank dusts into three categories with respect to reactivity:

- Dusts that are relatively inreactive (thermal runaway temperature greater than 400 °C)
- Dusts that are moderately reactive (thermal runaway temperature between 250 and 400 °C)
- Dusts that are the most reactive (thermal runaway temperature less than 250 °C).

Based on the thermal runaway temperatures of the dusts and the above classification scheme, the samples are listed by order of reactivity (by oxidation) in Figure 5. It is of interest that the reactivity of mixtures 1 and 2 is very close to that of the most reactive component, which represents 75% by weight of the mixture.

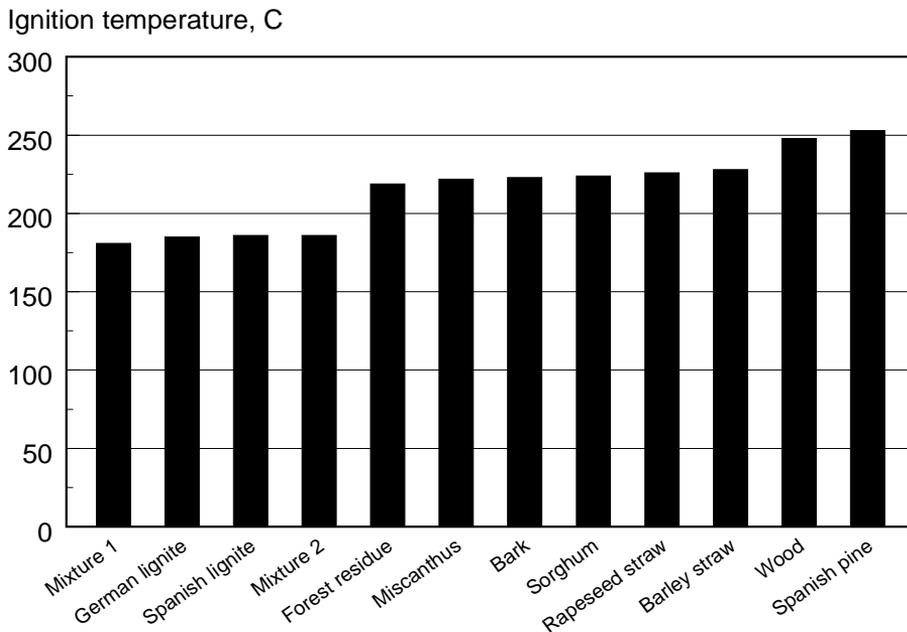


Figure 5. Reactivity classification DTA-TGA (INERIS).

5.5.3 Thermobalance measurements at elevated pressure (VTT)

Thermobalance measurements were carried out at VTT at 25 bar pressure for all 10 dust samples and at atmospheric pressure for three samples (wood, barley straw and German lignite). The objective was to measure and compare the reactivity of the samples at normal and elevated pressure. Additionally, some more fundamental characterisation of the oxidation behaviour of the dust samples concerned was discussed. As previously mentioned, the procedure used and described below differed from that used at INERIS in respect of sample size and the measured parameter (weight loss).

The tests were carried out using the pressurised thermobalance set-up of VTT shown in Figure 6, which can be used in atmospheric and pressurised measuring conditions. The experimental setup is described in [6]. The experiments were performed under non-isothermal conditions. The sample (100 mg, particle size <0.2 mm) was put into the cylindrical sample holder as a layer around the center bar and the wire mesh wall. The diameter of the sample holder was 8 mm and

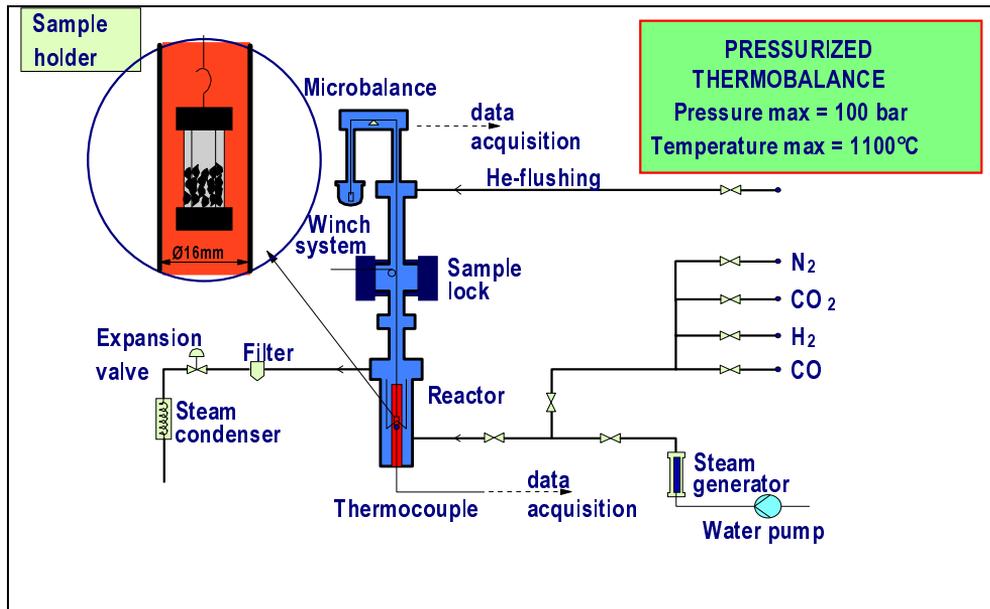


Figure 6. The pressurised thermobalance set-up.

the thickness of the sample layer about 1 mm. After the adjustments in the reactor were completed (i.e. pressure and gas flow), the sample was heated at a heating rate of 0,03 K/s starting from the room temperature.

In the measurements, the weight change as a function of temperature rise was measured both in nitrogen (pyrolysis) and in air. These measurements were carried out at 1 bar and 25 bar pressures. From the thermobalance output, the weight-change rates (%/s, dry sample) were derived and plotted in the same graph as shown in Figure 7. The temperature interval selected for the study was 150 °C and 250 °C, and the maximum rate was selected as 0,02 %/s. On this basis, the following parameters were taken as indicators for describing the proneness of self-ignition:

- The temperature (symbol T_0) at which the weight change rate in air deviated from that measured in nitrogen.
- The form factor describing the slope of the oxidation rate employing the following method (high number indicates high acceleration):

$$form\ factor = \frac{\frac{r_2 - r_m}{T_{r2} - T_m}}{\frac{r_m - r_o}{T_m - T_o}} \quad (5)$$

where r_o is the rate at T_o

r_2 the maximum oxidation rate, i.e., 0.02 %/s

T_{r2} the temperature at which r_2 was achieved

r_m the rate at the temperature in the middle of ΔT ($T_{r2} - T_o$)

T_m the temperature respectively.

The kinetic parameters (apparent activation energy E_a and frequency factor Z) for oxidation (i.e. the temperature interval $T_o - T_{r2}$) were determined using conventional linear regression ($\ln r$ vs. $1/T$) according to Arrhenius [10].

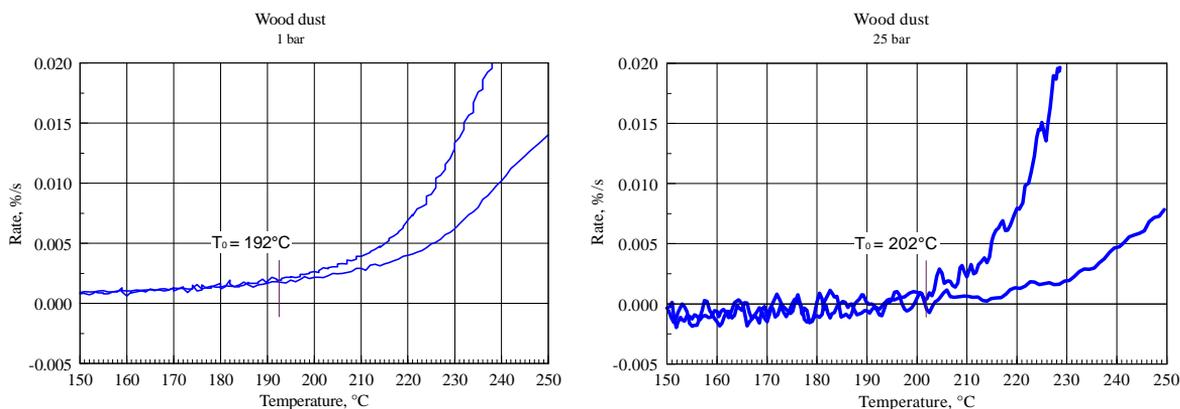


Figure 7. The weight change rates in air and nitrogen as a function of temperature (T_0 indicated); wood dust; pressure 1 bar and 25 bar.

5.5.4 Results and comparison of reactivity measurements

Results of the thermobalance measurements at atmospheric and elevated pressures at VTT are presented in Table 7. Activation Energy E_a and frequency factor Z (as logarithm) are also given. Two linear zones were observed in the results of the measurements carried out at 1 bar and therefore two kinetic parameter values are given in this case. This was not observed at 25 bar.

The onset temperature (T_0) indicates when the oxidation starts, and the form factor how fast the rate increases. Accordingly, the most reactive fuel would be the one having a low T_0 and a high form factor. For example, these terms seem to be fulfilled for black lignite under pressurised conditions, $T_0 = 170$ °C, form factor = 37 (the highest among the measured ones). Increasing the pressure leads to a higher oxygen partial pressure, increasing the aggressiveness of the oxidation. This should result in a higher reactivity of the sample at elevated pressures. Of the three samples tested at both 1 and 25 bar pressure, this was true for German lignite and especially for barley straw.

Table 7. Temperatures T_0 , form factors, activation energies E_a and frequency factors Z (as logarithm) measured both in atmospheric and in pressurised conditions.

Dust sample	P bar	T_0 °C	Form factor	E_a kJ/mol	ln Z, (%/s)
Wood	1	192	5	69.8 131.3	11.81 27.05
Barley	1	220	17	149.7 353.4	29.22 77.62
German lignite	1	180	15	64.7 119.5	10.51 24.54
Wood dust	25	202	3	225.1	50.09
Bark	25	193	2	122.9	25.69
Forest residue	25	185	4	132.4	28.13
Spanish pine	25	196	4	175.7	41.82
Barley straw	25	176	5	161.5	35.88
Miscanthus [*]	25	191	3	182.0	41.39
Sorghum	25	192	5	279.7	65.65
Rape seed straw	25	200	2	236.3	53.73
German lignite	25	170	37	171.9	39.59
Spanish lignite	25	186	11	209.3	47.00

* The duplication of this test resulted in very high rate after ignition.

Organising the dust samples according to reactivity and with the above described criteria for reactivity determinations by INERIS and VTT (Table 8) indicates that the low-grade coals are clearly more reactive than the biomass samples. The mixtures also show higher reactivity than the pure biomass fuels.

According to the tests by INERIS, the pure wood samples (wood, Spanish pine) would be less reactive than the other biomass samples. This was not, however, so evident in the pressurised measurements by VTT. From practical experience it is well-known that the lignites are very susceptible to spontaneous heating, especially newly excavated or thermally dried coals. It is also generally known about thermoanalysis that the heating rate affects the reaction temperature (the ignition temperature). INERIS and VTT used different heating rates and sample sizes in these measurements. This has to be observed when comparing the re-

sults. In addition, thermoanalytical data provides information for modelling self-heating, which could be a topic of a continuation work.

Table 8. Dust samples in ascending order according to their reactivity.

INERIS, 1 bar (ignition temp.)	VTT, 25 bar (ignition temp.)	VTT, 25 bar (form factor)
Mixture 1	German lignite	German lignite
German lignite	Barley straw	Spanish lignite
Spanish lignite	Forest residue	Sorghum
Mixture 2	Spanish lignite	Barley straw
Forest residue	Miscanthus	Wood
Miscanthus	Sorghum	Forest residue
Bark	Bark	Spanish pine
Sorghum	Spanish pine	Miscanthus
Rapeseed straw	Rapeseed straw	Rapeseed straw
Barley straw	Wood	Bark
Wood		
Spanish pine		

6. Self-ignition tests

Low-temperature reactions of organic substances with atmospheric oxygen lead to self-heating. With a sufficient material volume and low heat conductivity, heat build-up and spontaneous ignition may occur. When storing and handling biomass and other fuels, the tendency to spontaneous ignition and the temperature required for ignition should be known. Primarily four factors contribute to spontaneous ignition: oxidation tendency, ambient temperature, amount and characteristics of the material and shape of the material storage vessel.

The ambient temperature and the amount and form of the stored material are of significance, as heat generation typically occurs in proportion to volume and heat losses occur through the surface. As the volume increases according to the third power and the surface area according to the second one, there is a critical amount of material in which the generated heat is able to escape through the surface relatively quickly to prevent the temperature within the material from reaching the ignition point. The prevailing pressure also affects the tendency to spontaneous ignition via oxidation and heat transfer.

6.1 Test procedure

It is well-known that the self-ignition temperature of a dust deposit is a function of the size of the deposit; that is, the self-ignition temperature decreases when the size of the deposit (or storage volume) increases. The aim of the experiments in isothermal ovens is thus to determine the self-ignition temperature (also called the critical temperature) of the pulverised sample in volumes of varying sizes. Spontaneous ignition is usually studied for at least three samples of different volumes to be able to extrapolate the results for larger amounts. The samples are usually small, or else the time required by the tests would be too long. Then, using a model for thermal explosions, the self-ignition temperature can be extrapolated to larger volumes all the way up to the industrial scale.

6.1.1 Tests by INERIS

As described in [7], the dust is placed in cubical vessels of 8 to 1 000 cm³, the sides of which are made of fine screen material to allow air to pass but which are

able to contain the sample. The volume is then put into a ventilated oven pre-heated to the test temperature. A thermocouple is placed in the centre of the volume and another in the oven next to the sample and the temperatures are recorded as a function of time to see if there is ignition or not. The experiment is repeated for the same volume but with a fresh sample at a higher or lower temperature, depending on the result of the previous experiment until the critical temperature has been determined to within ± 5 K. The self-ignition temperature is defined as the ambient temperature at which ignition inside the sample can be detected.

The influence of pressure and oxygen concentration on the relationship between the self-ignition temperature and the storage size has been studied in a high-pressure reactor on one dust from each of the three categories of the potential fuels. The basic principle of the experiment is the same as for the tests run at atmospheric pressure.

The autoclave is 10 litre in volume and can operate at pressures up to 30 bar at 300 °C. The pressure is monitored by a pressure transducer. There are four thermocouples in the reactor: one to record the sample temperature and three to record the temperatures at various places in the reactor; one is placed just next to the sample. To ensure that oxygen in the reactor is not depleted to an extent that it impedes the self-heating process, a flow rate of 0.05 dm³/s of pre-heated air coming from a gas cylinder is maintained. For experiments in oxygen-poor atmospheres, the required gas composition is premixed in gas cylinders and used directly. The gas exiting the autoclave is monitored by an oxygen analyser.

The temperatures, pressure, and oxygen concentration at the reactor exit are recorded by a digital data-logger and transferred to a PC.

The experimental procedure was as follows:

- The dust is placed in the wire mesh baskets (8 or 120 cm³). The filling is accomplished by slightly tapping the cages so that the dust settles into all of the corners; the excess dust is levelled off.
- The cage is placed in the reactor at ambient temperature, a thermocouple is placed in the centre of the sample, and then the reactor is closed air tight.
- The autoclave is then purged with nitrogen.

- When the oxygen has been eliminated for the autoclave, the heating elements are switched on and the reactor is heated to the pre-determined test temperature while maintaining the flow of nitrogen.
- After the temperatures in the autoclave and in the sample are stabilised, the nitrogen flow is replaced by air for three minutes.
- Then, the high pressure valve is closed and the pressure increases up to the test pressure, either 10 or 25 bar gage.
- The test is stopped when ignition occurs or after two to four hours, depending on the sample.

If ignition occurs, the experiment is repeated for a fresh sample under the same conditions except that the test temperature is lowered. This procedure is repeated until the self-ignition temperature is determined.

6.1.2 Tests by VTT

The following method is used at the laboratory of VTT Energy [1] for determining spontaneous ignition at atmospheric and elevated pressure. The principle of the equipment is shown in Figure 8.

A cylindrical fuel sample is placed in a metal mesh vessel and then into an autoclave. The determination is carried out by raising the pressure in the autoclave to the desired level (1 and 25 bar) by introducing simultaneously the desired gas atmosphere in the autoclave. The gas is preheated prior to entering the autoclave. The ambient temperature is increased stepwise at 10 K steps (max 220 °C) with a heat coil located on the inner walls of the autoclave. Prior to feed, the temperature of the gas is raised to that of the autoclave to secure a steady temperature around the sample. The gaseous atmosphere is dynamic, as the gas flows slowly through the autoclave and the composition of the gaseous atmosphere is nearly constant. The sample is kept at each temperature stage a sufficiently long time (usually from one to several hours) to monitor whether the ambient temperature is sufficiently high for spontaneous ignition. The temperatures at the midpoint, and at one end of the sample as well as the temperature of the gas, are recorded. The temperature of the gas volume near the sample at the moment of spontaneous ignition is given as the result of the determination.

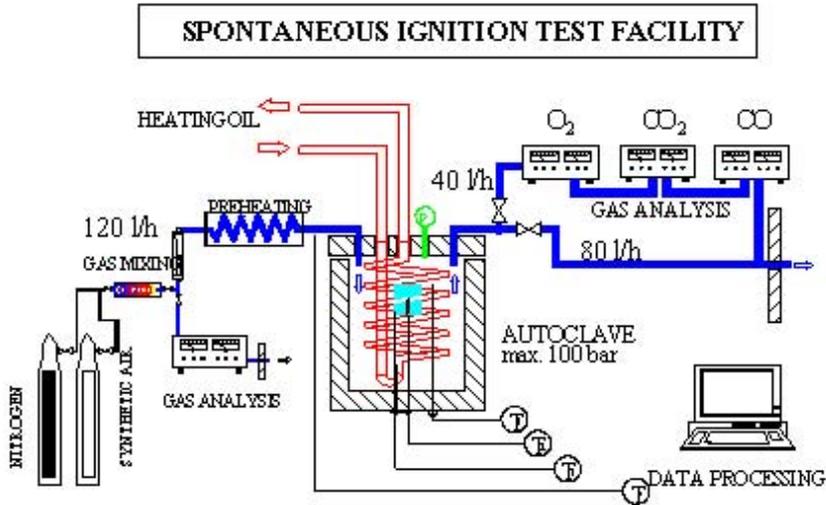


Figure 8. Schematic of dynamic test equipment for spontaneous ignition (VTT).

The composition of the inlet and outlet gas is measured with continuously operating gas analysers to control the stability of the gaseous atmosphere and also to record the moment of spontaneous ignition on the basis of change in the composition of the outlet gas. Nitrogen is typically used as inert gas to dilute the oxygen concentration below ignition limits except for the use of special gases, such as flue gas for inerting. The temperatures and gas analyses are recorded automatically by a data logger. The sample is changed rather often, depending on the temperature level, irrespective of whether it is ignited or not. The ignition characteristics can change, if the same sample is kept at the elevated temperature too long.

6.2 Frank-Kamenetskii theory

In order to extrapolate the ignition temperatures to volumes larger than those studied experimentally, the Frank-Kamenetskii's thermal explosion model can be used [8, 9]. Briefly this theory is based on an energy balance, whereby the heat generated in the dust deposit by an exothermic reaction of order 0 and following the Arrhenius equation, is completely dissipated to the surroundings in order to obtain a stable steady state. Heat transfer is controlled by conduction from the centre of the deposit to the surface. If all heat is not dissipated, the temperature

in the dust rises until a thermal steady state is reached or until there is ignition. This theory shows that for a given volume of a given dust, there exists a "critical" temperature beyond which a steady state is not possible and ignition occurs in all cases. The lumped parameter equation that represents this state is given below:

$$\ln(\delta_c T_c^2 / r^2) = M - N/T_c \quad (6)$$

where δ_c is form factor for the storage volume (2.6 for a cube, 2 for an infinite cylinder, 0.88 for a slab)

T_c is self-ignition temperature in K (or critical temperature)

r is characteristic dimension of the deposit in meters (the half-edge length of a cube, the radius of a sphere or long cylinder)

M and N are constants that depend on the physico-chemical properties of the dust (heat of reaction, apparent activation energy, density, heat capacity, thermal conductivity, oxygen concentration, frequency factor).

N is E/R where E is apparent activation energy and R is gas constant.

The results of the experiments in the isothermal ovens can be used to determine these two semi-empirical constants. Once the two constants have been determined, one can extrapolate the self-ignition temperature (in K) as a function of deposit size (r in meters) up to larger dimensions using equation (1) expressed in a more convenient form:

$$r = \delta_c^{1/2} T_c \exp(-M/2 + N/2T_c) \quad (7)$$

6.3 Results of the self-ignition tests

6.3.1 Tests at ambient pressure

INERIS determined the critical temperature for four volumes: 8, 120, 340, and 1 000 cm³. The critical temperatures as a function of volume are given for ten dust samples in Table 9. The critical self-ignition temperature clearly decreases as a function of volume.

Table 9. Self-ignition temperature of four sample volumes (INERIS).

Dust sample	Critical temperature, °C,			
	8 cm ³	120 cm ³	340 cm ³	1 000 cm ³
Wood	232	197	182	172
Bark	207	167	152	142
Forest residue	202	167	157	147
Spanish pine	237	197	182	167
Barley straw	222	187	177	167
Miscanthus	212	182	172	162
Sorghum	212	182	172	162
Rapeseed straw	223	193	178	168
German lignite	152	127	112	107
Spanish lignite	162	132	122	112

The extrapolation curves for the ten samples are grouped by dust type in the figures of Appendix B. The self-ignition curves for the four wood fuels and wood wastes are shown in Figure 9. The results of the oven tests are consistent with the DTA-TGA tests: the lignites are the most reactive whereas the wood sample is the least reactive. The other samples are in between.

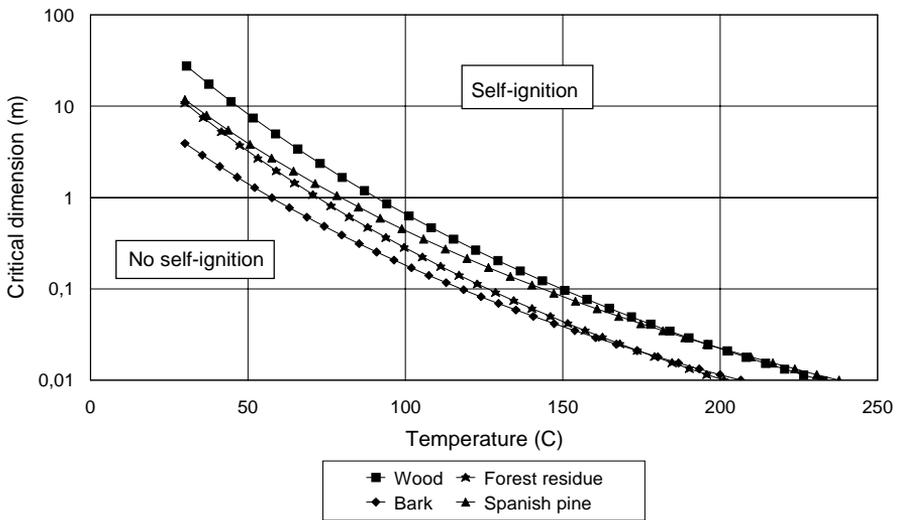


Figure 9. Extrapolation of critical dimension as a function of temperature (INERIS).

VTT carried out self-ignition tests at ambient pressure on three dust samples: wood, barley straw and German lignite. The results of the tests in the dynamic test facility with three different size of cylindrical samples are presented in Table 10. A comparison of the INERIS and VTT test results is presented in Chapter 6.3.4.

The German lignite is clearly more reactive than the biomass samples. The same relation between self-ignition temperature and volume as in the tests by INERIS can also be seen in VTT's tests.

Table 10. Self-ignition temperature of three sample volumes (VTT).

Dust sample	Critical temperature, °C		
	50 cm ³	100 cm ³	400 cm ³
Wood	218	211	195
Barley straw	199	190	175
German lignite	144	133	112

6.3.2 Tests at elevated pressure

Tests results by INERIS at elevated pressures of 10 and 25 bar are summarised in Table 11 as a function of the parameters volume (8 and 120 cm³) and pressure (1, 10 and 25 bar). The three samples examined were wood dust, barley straw, and German lignite.

Table 11. Self-ignition tests at elevated pressures (INERIS).

Dust sample	Pressure	8 cm ³	120 cm ³
Wood	1	232	197
	10	206	175
	25	195	168
Barley straw	1	222	187
	10	200	168
	25	187	143
German lignite	1	152	127
	10	127	99
	25	115	91

A theoretical treatment of the self-ignition problem employing the Frank-Kamenetskii model and integrating the effect of pressure shows that only the parameter M in equation (6) depends on the partial pressure of oxygen in the following way [8]:

$$M = M_0 + n \ln (P_{O_2}/P_0) \quad (8)$$

where P_{O_2} is partial pressure of oxygen at the test condition
 P_0 partial pressure of oxygen in ambient air
 n reaction order.

The constant M_0 then depends on all of the parameters cited previously except the oxygen concentration.

Thus

$$\ln (\delta_c T_c^2 / r^2) = M_0 + n \ln (P_{O_2}/P_0) - N/T_c \quad (9)$$

M_0 and N have been determined previously in isothermal oven tests at atmospheric pressure and the self-ignition temperatures at 10 and 25 bar for two volumes have been used to determine n for each of the three samples tested at elevated pressure :

Wood	$n = 0.54$
Barley straw	$n = 0.57$
German lignite	$n = 0.75$

Thus, equation (9) can now be used to estimate the self-ignition temperature as a function of storage size (characteristic dimension) at any partial pressure of oxygen for the three samples tested. Figures in Appendix B show the extrapolation curves based on this equation for three experimental pressures along with the experimental points. The self-ignition temperature of German lignite at different pressures is shown in Figure 10.

It is of interest that the reaction order for the two biomass samples is very similar and is close to 1/2 whereas the reaction order for the German lignite is significantly higher, 3/4. This means that the German lignite is more sensitive to the partial pressure of oxygen than the other samples.

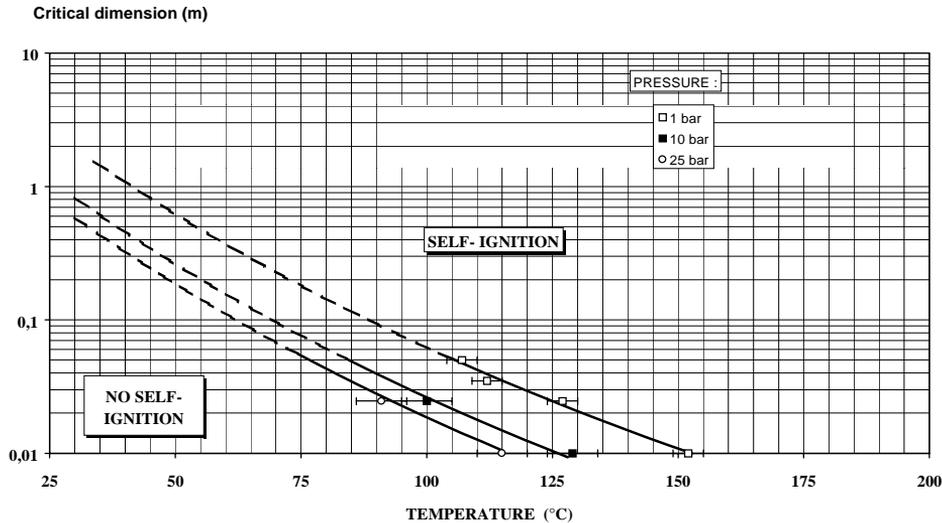


Figure 10. Extrapolation of the critical dimension of German lignite as a function of temperature and pressure (INERIS).

VTT carried out self-ignition tests with all dust samples, including the two mixtures at an elevated pressure of 25 bar. The extrapolated curves of the self-ignition temperature as a function of cylinder diameter (diameter = two times the critical radius of the cylinder) for the woody fuel samples are shown in Figure 11. The order of reactivity (increasing from wood to Spanish pine) is the same as in the ambient pressure tests by INERIS (Figure 9).

The effect of pressure on self-ignition tendency is presented for three dust samples: wood, barley straw and German lignite in Figure 12. The increasing pressure clearly enhances the self-ignition tendency of the fuels, which also was noticed in Figure 10.

6.3.3 Self-ignition in oxygen-depleted atmosphere

A sufficiently inert atmosphere is required to avoid or to suppress self-ignition of fuels during handling and storage. This level is considerably lower than the lower oxygen concentration required to inhibit a dust explosion. Especially for pressurised feeding systems the knowledge of the critical inerting level is of

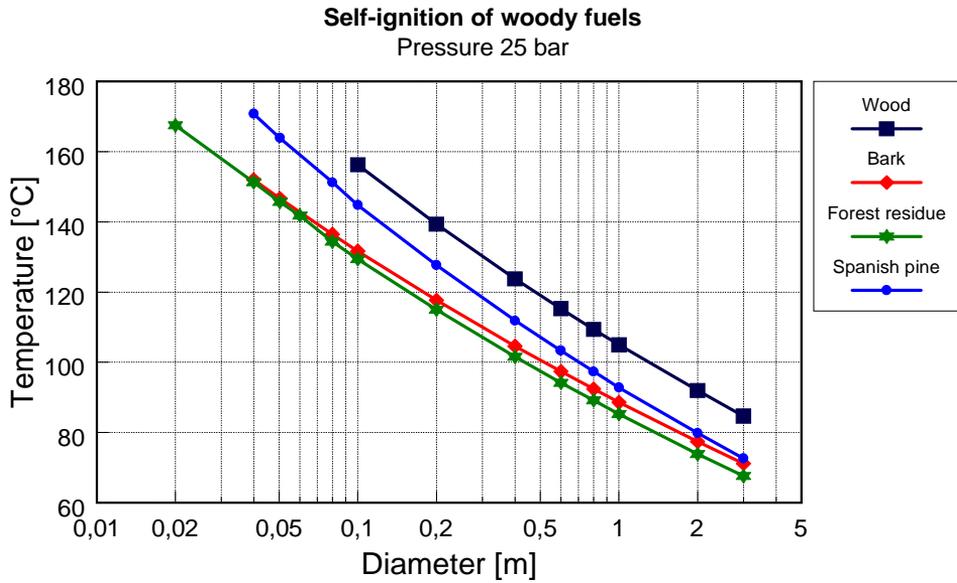


Figure 11. Extrapolation of self-ignition temperature as a function of the diameter at 25 bar pressure (VTT).

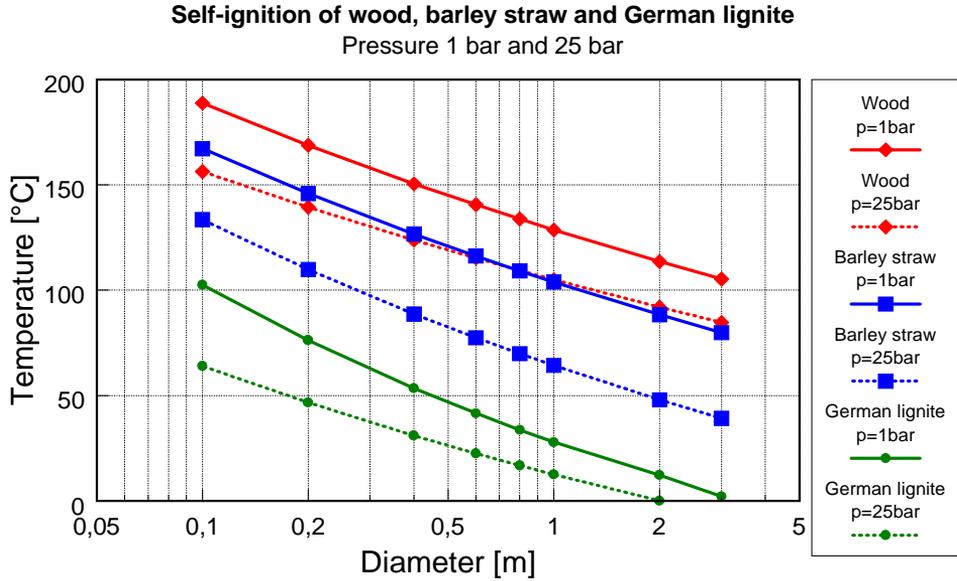


Figure 12. Self-ignition of three fuel samples in ambient and elevated pressure (VTT).

particular importance, as the feeding bins have to be pressurised with an inert media to assure safe conditions. Two options are in practice available to create the partly inert atmosphere: substituting air with nitrogen or dry flue gases.

VTT performed self-ignition tests in a partly inert atmosphere with three dust samples: wood, barley straw and German lignite. Tests were carried out both at 1 bar and 25 bar pressure. The inert gas was a simulated dry flue gas, with approximately the same composition as a flue gas from a gas turbine:

Oxygen $O_2 = 14\%$
 Carbon dioxide $CO_2 = 7\%$
 Nitrogen $N_2 = 79\%$

Extrapolated self-ignition temperatures for German lignite as a function of the diameter are shown in Figure 13. The results indicate that the self-ignition temperature is somewhat higher in partly inert atmosphere, but the difference, and hence the increased safety due to inerting in this case, is rather small.

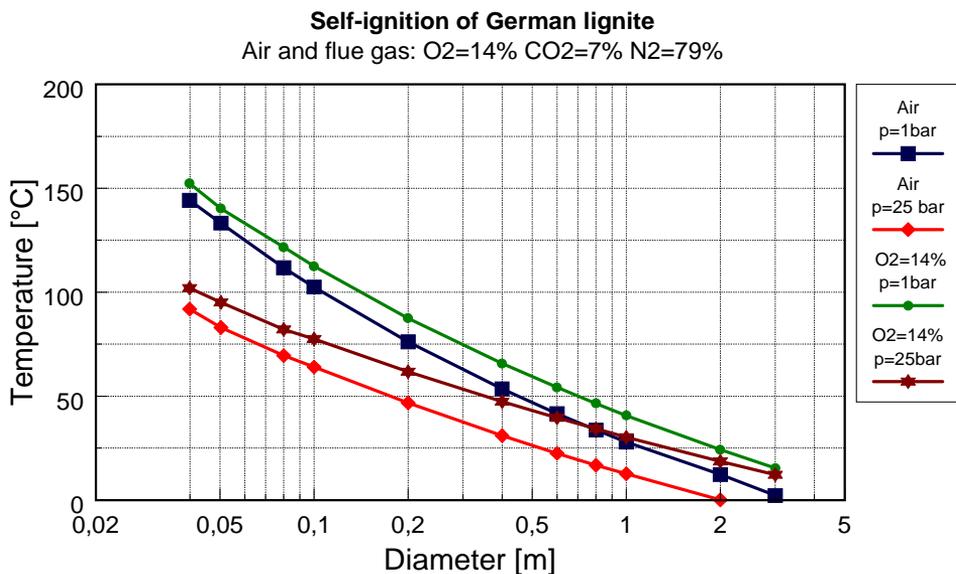


Figure 13. Self-ignition of German lignite in partly inert atmosphere at pressures 1 bar and 25 bar (VTT).

INERIS determined experimentally self-ignition temperatures in oxygen-depleted atmospheres for the wood and German lignite samples. The self-ignition temperature and the maximum temperature attained after ignition for the German lignite in the 8 cm³ volume at 25 bar for the three concentrations tested (7, 14, and 21%) are plotted in Figure 14. It can be seen that the self-ignition temperature is relatively insensitive to the oxygen partial pressure whereas the maximum combustion temperature is strongly dependent of this pressure.

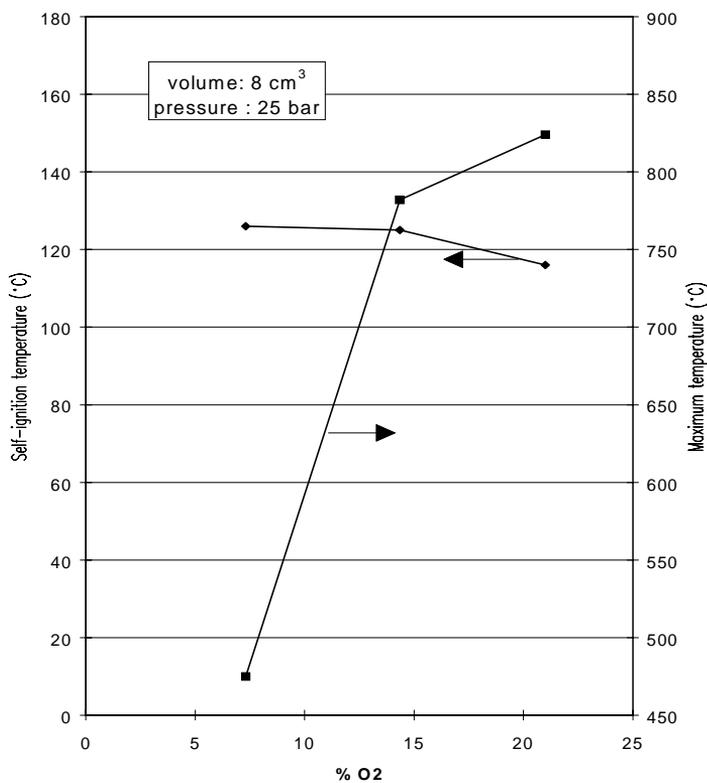


Figure 14. Self-ignition and maximum combustion temperature of German lignite as a function of oxygen concentration (INERIS).

6.3.4 Comparison of results

One of the objectives of the self-ignition research was to compare the methods applied by INERIS and VTT. The procedures and test equipment used for high-pressure tests were essentially identical. For the ambient pressure measurements INERIS used the conventional oven test procedure. The procedure to heat the samples to the ignition point and also to some extent the way of identifying the ignition point differed between the laboratories. INERIS and VTT used different sample configurations, INERIS cubic and VTT cylinder shaped samples. The Frank-Kamenetskii theory takes this into account in the form factor δ_c in equation (6). Further, VTT used a compacted sample simulating the actual bulk density of material in the storage bin, while INERIS used the normal loose bulk density. This may affect the heat transfer through the sample.

Figure 15 presents a comparison between the actual self-ignition measurements of INERIS and VTT at ambient pressure. The ignition temperatures for the separate sample volumes are essentially in line, which suggests that the procedures are comparable within a fairly narrow error margin. The extrapolated values for wood at 1 bar and 25 bar are shown in Figure 16, and also show an acceptable consistence (critical dimension = diameter).

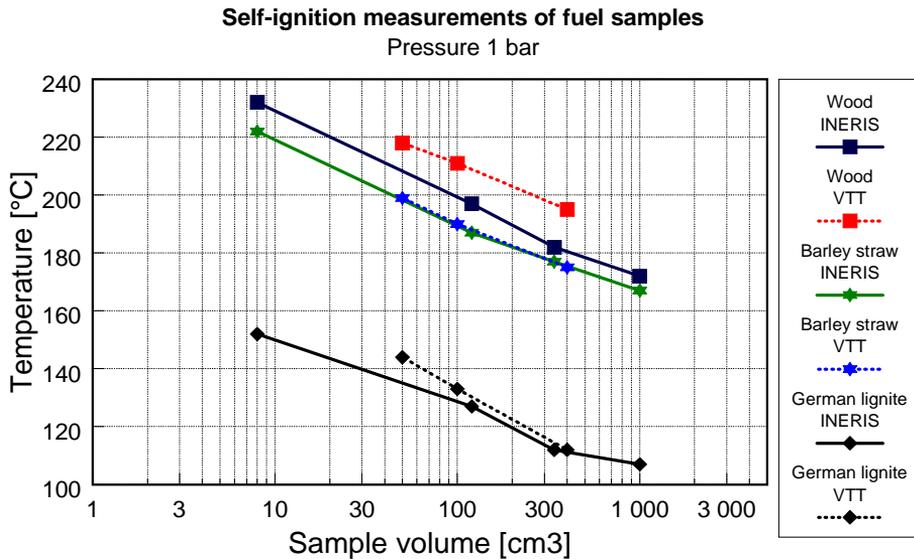


Figure 15. Comparison of self-ignition measurements by INERIS and VTT.

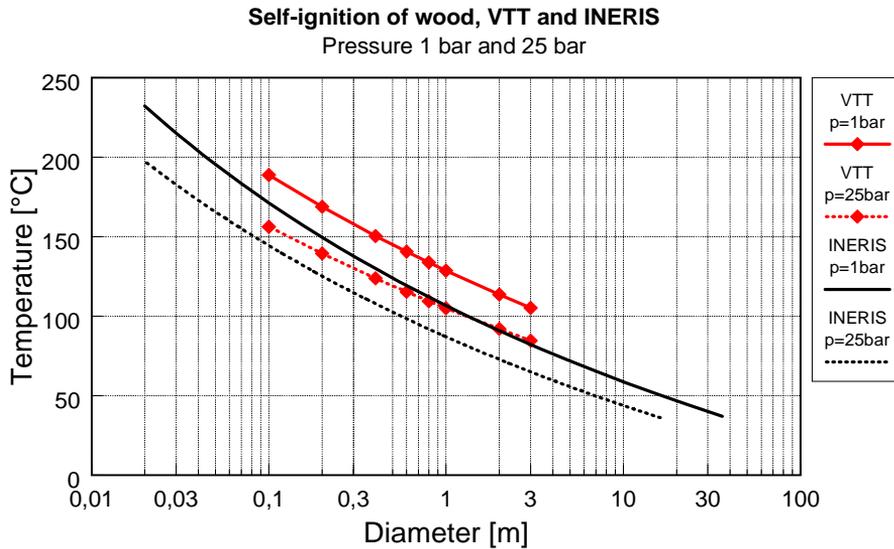


Figure 16. Comparison of extrapolated self-ignition temperatures by INERIS and VTT.

Comparison of self-ignition risks in storage bins with a critical dimension of 1 m (cubic bin of 8 m³ or cylindrical bin of 6.3 m³ with equal diameter and height), Table 12, shows that the VTT's procedure gives slightly more conservative self-ignition values. The influence of pressure and the reactivity of the lignite are, however, quite evident. When evaluating the results, the fairly rough extrapolation from very small samples to large storage volumes has to be considered.

Table 12. Self-ignition temperatures of storage bins with a critical dimension of 1 m.

Dust sample	INERIS, 1 bar °C	INERIS, 25 bar °C	VTT, 1 bar °C	VTT, 25 bar °C
Wood	91	73	114	92
Barley straw	92	73	88	58
German lignite	42	<25	12	≈ 0

6.3.5 Self-ignition of fuel mixtures

The fuel mixtures wood/German lignite (Mixture 1) and barley straw/Spanish lignite (Mixture 2) exhibit in the thermogravimetric tests a reactivity comparable to that of very reactive pure lignites. The self-ignition tests carried out by VTT at 25 bar pressure confirmed the reactivity of the mixtures. The self-ignition temperatures measured with three different sample sizes in the tests showed a marked decrease in ignition temperature for the larger. This resulted in the extrapolation of self-ignition temperatures even lower than those calculated for lignites (Figure 17). The reason may be the high reactivity of lignite in the mixtures opposed by the low thermal conductivity of biomass.

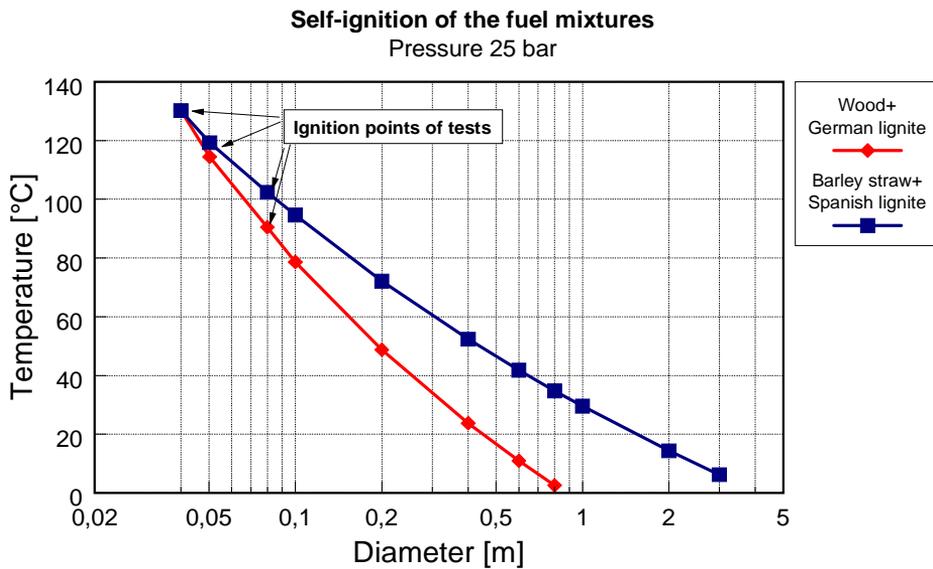


Figure 17. Self-ignition behaviour of the fuel mixtures at 25 bar pressure (VTT)

6.3.6 Time factor evaluation

The time needed for self-ignition, the so-called induction time, was estimated for two fuel samples, wood and German lignite, at 25 bar pressure according to [8]. The ignition time was calculated based on the fact that the heat transferring from the surroundings to the middle of the fuel bin requires a certain time. Heat conductivity values presented in Table 2 were used. Heat transfer between the sur-

rounding atmosphere and the wall of the storage bin was neglected. The calculations give only a very rough estimate of the induction time. The dependence of the critical bin diameter (and corresponding self-ignition temperature) on the induction time is seen in Figure 18.

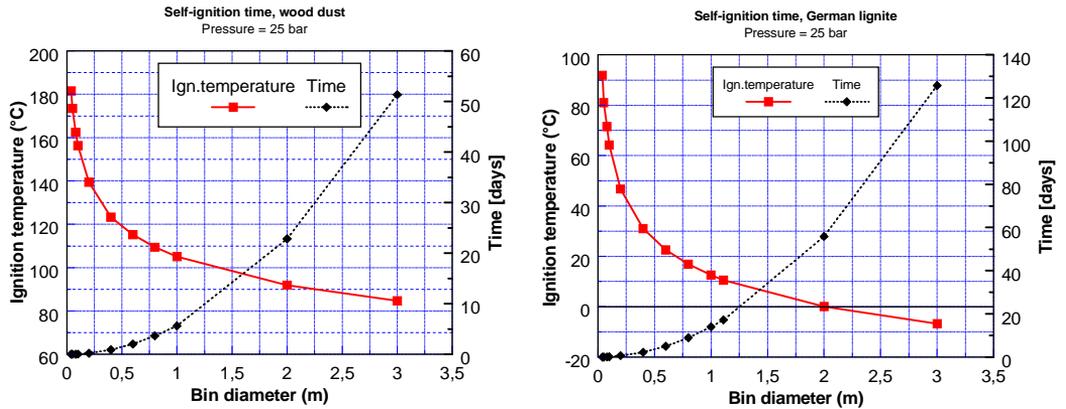


Figure 18. Self-ignition time as a function of critical bin diameter (VTT).

6.4 Discussion

The following conclusions can be made regarding the self-ignition tendency:

- The results of the self-ignition tests for the fuel samples are mainly in line with the reactivity tests carried out on DTA-TGA. The lignites are the most reactive fuels both at ambient and elevated pressure. They also exhibit the lowest self-ignition temperatures. The pure wood fuels, wood and Spanish pine, were least reactive with regard to spontaneous ignition. The wood wastes, bark and forest residue, which also contain green parts and bark, are, however, more reactive than the agricultural straw residues. The different straws indicated very similar self-ignition temperatures.
- The mixtures of lignites and biomasses were in reactivity comparable with the most reactive pure lignites. The self-ignition tests gave significantly low ignition temperatures, even lower than for the pure lignites. This may be due

to the combination of the high reactivity of the low-grade coal and the low thermal conductivity of the biomass.

- The elevated pressure had a significant effect of the self-ignition temperature of all fuel samples. Increasing the pressure decreases the self-ignition temperature of the fuel. This may be ascribed to the higher partial pressure of oxygen in the surrounding atmosphere. The magnitude of the pressure effect was approximately the same for all fuel samples.
- Partial inerting, that is decreasing the oxygen concentration of the ambient air atmosphere, increased the self-ignition temperature, but to a rather low degree. To establish significantly safer conditions in fuel storage, obviously an inert atmosphere with an oxygen content well below 7% is required. No essential difference between the inerting effect of pure nitrogen and dry flue gases with the corresponding oxygen concentration could be noted.
- The self-ignition test equipment and procedures practised by INERIS and VTT produced comparable results. The small variations were mainly assigned to the different sample configurations and the different compactness (bulk density) of the samples used in the tests by these two laboratories.

7. Explosion test methodology

7.1 Explosion properties

The severity of dust explosion can be determined by measuring the following parameters [1, 11]:

- Maximum explosion pressure (P_{\max} or MEP): is the maximum value of pressure measured during an explosion process, determined by tests over a wide range of fuels concentrations.
- Maximum rate of pressure rise ($((dP/dt)_{\max}$ or MRPR): maximum slope of a tangent through the point of inflexion in the rising portion of the pressure vs. time curve, determined by tests over a wide range of fuels concentrations.
- K_{St} -value: is a constant that characterises the explosibility of the material, and is obtained by mathematical transformation from the $(dP/dt)_{\max}$ value, according to the cubic law:

$$K_{St} = \left(\frac{dP}{dt} \right)_{\max} V^{1/3} \quad (10)$$

- Minimum explosive concentration (MEC): is the lowest concentration of dust mixture with air capable to undergo an ignition process.
- Lower Oxygen Concentration (LOC): the oxygen concentration of partly inert atmosphere in which an explosion is not possible.

The explosion indices are explained in Figure 19.

7.2 Objectives

Normally the explosion tests are performed in ambient conditions in a 1 m³ pressure vessel according to ISO 6184/1 standard procedure [2], or in a smaller 20-litre laboratory sphere. Previous explosion tests carried out with wood dusts have shown that it is necessary to define and develop test procedures to facilitate

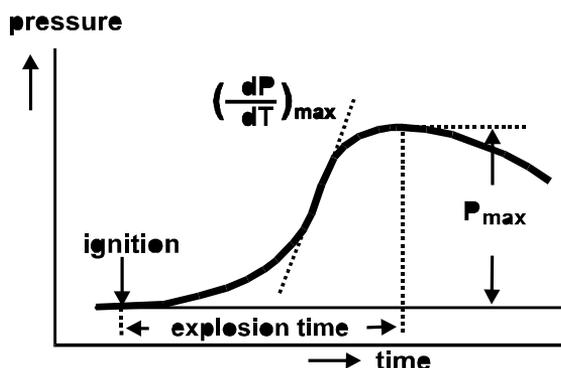


Figure 19. Recording of explosion pressure versus time during a dust explosion test.

obtaining repeatable and comparable test results for these “difficult” dusts [12]. This need is emphasised by performing the tests at elevated pressure and/or high temperatures. The objective of this subtask is to develop injection procedures, dust dispersing methods and ignition procedures for these kinds of dust and tests in elevated conditions.

The explosion characteristics determined in the dust explosion tests are highly dependent on turbulence inside the test vessel immediately before and during the explosion. The turbulence depends strongly on the dust injection process applied. In order to obtain comparable results in different test conditions it is of crucial significance to keep the turbulence conditions constant, also at high initial pressures and elevated initial temperatures. At 10 bar initial pressure roughly ten times more oxygen is present in the vessel and therefore roughly ten times more fuel can be burned. Consequently, this subtask includes the following development work:

- Development of a dispersion nozzle that makes it possible to disperse a large amount of difficult flowing dusts.
- Definition of the injection procedure at elevated pressure.

It is necessary to test several dispersion nozzles for at least two different dusts under various conditions. Two materials were selected for the experiments: wood

dust was chosen due to its fibrous nature and difficulties shown in grinding and sieving; German lignite was selected as a contrast sample, considering its recognised properties, and with the aim to cover all characteristics of materials included in the programme.

The tests were carried out simultaneously in the TNO 20-litre sphere and in the LOM 1 m³-vessel. However, due to the large amounts of dust that was needed inside the sphere at this high initial pressures, TNO was forced to disperse the dust when it was already on the bottom of the sphere. A nozzle type was designed which directed the air flow during the injection over the bottom of the sphere. LOM used the conventional method of introducing the dust from an external dust container. For this reason LOM used injection nozzles of a different construction.

7.3 Experimental multifactorial design

7.3.1 Introduction

Regarding the explosion test methodology an experimental design was carried out in co-operation by LOM and TNO to define a testing programme and the optimum test conditions. To optimise the experimental work it was decided to consider the study of a well known technique in the experimental field: the multifactorial experimental design. It was also decided to establish at least three levels, qualitative or quantitative, for the variables involved in the design.

In a classical unifactorial design only the levels of the factor to study are modified, while the rest of the factors, which are not specific targets of study, remain constant. Subsequently, only the effect of a concrete factor is analysed. In a multifactorial design the levels of all the implied factors are varied simultaneously. Several advantages of multifactorial design over classical unifactorial design can be pointed out:

- In a multiple factors process, the number of experiments to be carried out with a unifactorial design would be considerably higher than that of a multifactorial design.

- Apart from studying the effect of a factor, the multifactorial study also supplies simultaneous information about the effects of the rest of the factors and about interactions among them.
- Only through a simultaneous variation of all the factors information about the persistence of effect of a determined factor can be obtained when the levels of the rest of the factors are modified, increasing this way the significance of the results.

7.3.2 Parameters

The two selected dust samples, wood and German lignite, were tested for the following variables:

- dispersion devices
- injection pressure
- initial pressure.

Dispersion devices

Three different devices were used in both explosion vessels. For the 1 m³ spherical vessel of LOM, the selected dispersion nozzles were the following:

- Open nozzle: A linear tube with a high number of orifices of different sizes in the end. The sum of the area of the holes totals a cross-sectional area of about 1 500 mm². The diameters of the holes from the centre to the exterior are 8 mm, 6 mm, 5.5 mm, 3.5 mm and 3 mm.
- Ring nozzle: A semicircular hollow pipe with sixteen 8 mm diameter holes and 8 holes in each branch. The total cross-sectional area was approximately 800 mm². This ring nozzle was not identical to the one described in the ISO standard 6184/1.
- Rebound nozzle: A typical nozzle with a V-shaped dispersor in the centre. The V dispersor is located upon a 20 mm diameter hole. The dispersor has also three holes of about 2 mm in diameter, located over the 20 mm hole. When the dust comes out, it crashes against the V walls and then is deflected. The cross-section area is around 320 mm².

TNO used three different types of nozzle that all dispersed the dust already placed on the bottom of the 20-litre sphere (Figure 20). Nozzle 1 is constructed as a tube with a round flat hood fixed firm on the tube. The hood has a diameter of 5.5 cm and 12 small holes with a diameter of 2 mm each that disperse the dust that is placed on top of the nozzle. The tube has six holes with a diameter of 9 mm each, evenly divided on the outline of the tube, that is directing the inlet air over the bottom of the sphere. Nozzle 2 is constructed as a tube with a round spherically shaped hood with a diameter of 8 cm fitted 3 mm above the tube. The hood itself is not perforated and the inlet air is directed through the space between the hood and the tube and over the bottom of the sphere. Nozzle 3 is of the same design as nozzle 2 except for the dimensions. The diameter of the hood is reduced to 6 cm and the space between the tube and the hood is reduced to 2 mm.



Figure 20. Dispersion nozzles designed and used by TNO.

Initial pressure

To cover a wide range of experimentation, three initial pressures were tested, undertaking a typical range of operation pressures in industrial processes and pressurised power production: 1 bar, 8 bar and 15 bar.

Injection procedure

A choice had to be made with regard to the injection procedure of the dust at elevated pressures. In the method of constant pressure difference, the pressure difference between the explosion chamber and the dust container is kept con-

stant. ISO 6184/1 (1) recommends a standard injection pressure difference of 20 bar at ambient pressure (1 bar) explosion tests. In the method of constant pressure ratio, the pressure ratio is kept constant. The third option was to use a proportional pressure difference, where the injection pressure followed a linear relation to the initial pressure. The resulting injection pressures of the different methods at elevated initial pressures are shown in Figure 21.

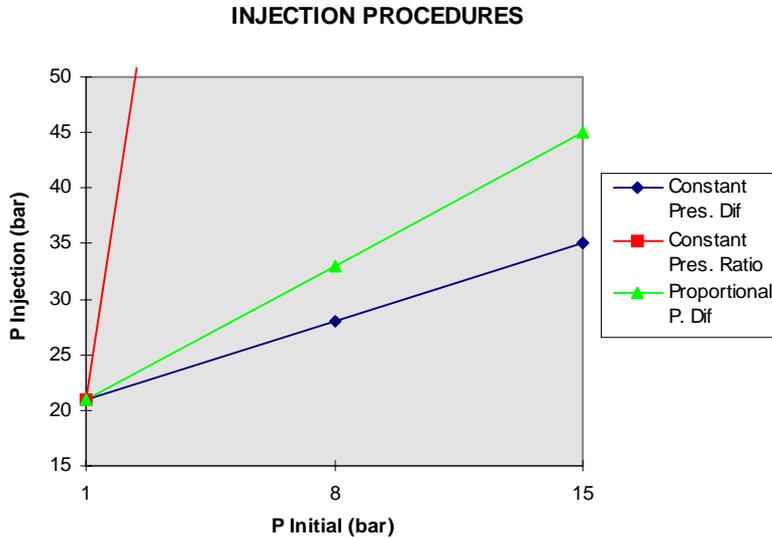


Figure 21. Injection procedures.

The constant pressure ratio method is obviously not applicable in standard vessels for medium to high initial pressures. Although the constant pressure difference is perfectly applicable, but applied in a multifactorial design, it would give no information about the injection pressure effect. The proportional pressure difference criterion seems to be the more adequate, realistic and applicable to the experimental design proposed.

7.3.3 The test programme

Table 13 presents the variable with different levels. The levels are numbered 0, 1 and 2, corresponding to low, medium and high value of the factor, or to a different class of a qualitative factor.

Table 13. Multifactorial test plan for the injection tests.

FACTORS	LEVELS OR KIND OF VARIABLES		
A Initial pressure (P_i)	0 1 bar	1 8 bar	2 15 bar
B Injection pressure (P_{inj})	0 20 bar	1 25 bar	2 30 bar
C Dispersion device	0 nozzle 1	1 nozzle 2	2 nozzle 3

The P_{max} and K_{St} -values were measured as indicators of a good turbulence. The first criterion was that the nozzle gives the same results in ambient conditions as the standard 20-litre sphere. At higher initial pressures, if P_{max} and K_{St} were high, the turbulence must have been high. If the turbulence has been high, good dispersion and injection conditions should have been achieved.

Since there are three variables, each on three levels, a fractional experimental design is proposed based on a 3^3 multifactorial model. A complete 3^3 multifactorial design would have led to 27 tests per sample; this design is excessively expensive and long-term. It was decided to follow a **fractional (incomplete) blocked design**, in which each block is a partial replication, confounding the third order effect ABC. Subsequently, the design is also balanced.

The chosen design is a 3^{3-1} model, composed of 3^1 (3) blocks by replication, and so, with 3^2 (= 9) tests per block. Thus, each block is a 1/3 replication (containing one third of a complete design). This model was proposed by Cox and Cochran in their 3^n series [13]. From now on, the model will be referred as 3^3 , given that each block is simply a fraction of it.

Therefore, the experimental plan consists of two blocks of nine tests each, one for wood dust and the other for German lignite. The total number of 18 tests constituted the two blocks. The experimental plan, according to the order of variables and levels given in Table 13, is as follows:

Block 1 <u>WOOD DUST</u>	Block 2 <u>GERMAN LIGNITE</u>
ABC	ABC
000	000
012	102
101	011
202	022
021	201
110	110
122	212
211	121
220	220

Thus the 122 test means that the initial pressure is 8 bar, the injection pressure is 30 bar ($\Delta P = 30$ bar, and hence the pressure in the injection vessel is $8 + 30 = 38$ bar) and the dispersion device is nozzle 3. Both LOM and TNO performed this test programme.

7.4 Results of the tests by TNO

Two examples of results obtained by TNO are shown below in Figures 22 and 23. The maximum rate of pressure rise (dP/dt) is shown as a function of initial pressure for wood dust and German lignite.

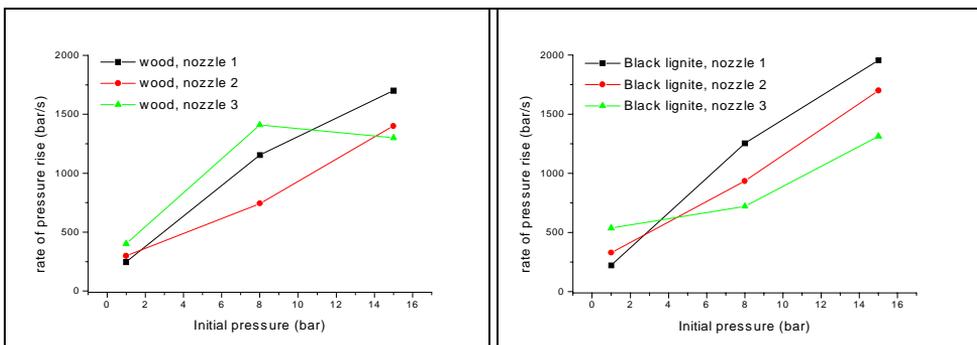


Figure 22. (dP/dt) as a function of initial pressure for wood dust and for three different nozzles.

Figure 23. (dP/dt) as a function of initial pressure for German lignite dust and for three different nozzles.

Although the results for nozzle 1 seem to be in good comparison with the other tests, a visual check of the contents of the sphere after the test showed that only parts of the wood dust was dispersed in a proper way. Lumps of burned material and channels through the unburned material were observed inside the sphere. Possibly, due to restrictive openings inside the nozzle, the turbulence inside the sphere increased to such an extent that the (dP/dt) value rose to a normal value or even higher. However, on the basis of the visual check it was decided to discard nozzle 1.

The results showed that for P_{\max} both nozzle 2 and nozzle 3 resulted in almost the same value. Regarding the rate of pressure rise it seemed that nozzle 2 resulted in somewhat higher values than nozzle 3. However, when the differences in injection pressure were incorporated, the results of wood dust were shifted towards each other, and for German lignite the results of nozzle 2 were definitely higher than those for nozzle 3 at high initial pressures.

At high injection pressures (30 bar) nozzle 3 was damaged because the dispersing hood was blown off the tube. The results discussed here, together with the fact that nozzle 2 had a stronger design, made nozzle 2 the best choice for dispersing dust at the bottom of the sphere at high initial and dispersing pressures. To ensure that the new test procedure gives commensurate results with the standard test procedure with the rebound nozzle, comparativity tests were performed with a bad flowing product. P_{\max} as well as K_{St} -value measured for a wide range of dust concentrations for both procedures were reasonably comparable.

For the dispersion pressure a choice must be roughly made between the constant pressure difference method and the constant pressure ratio. Regarding this methodology Dahoe [14] has clearly shown that, in order to keep the turbulence conditions constant at higher initial pressures, the constant pressure ratio method should be used. Therefore it was decided to use the constant pressure ratio method in the high pressure tests of TNO.

Performing explosion tests at high initial pressures with the constant pressure ratio method introduces two main problems. Since there is more oxygen (in absolute quantities) present in the explosion chamber at higher initial pressures, larger amounts of dust can be combusted. Placement of larger amounts of dusts in the sphere requires an extended discharge time to disperse all the dust. Sec-

only, when using the constant pressure ratio method very high pressures are needed inside the injection vessel. Using the standard atmospheric test as the starting point, a pressure of 315 bar is required inside the dust injection vessel for a test with an initial pressure of 15, if a pressure ratio of 20 is used instead of a 20 bar pressure difference. Not only is this far beyond the capabilities of our test system, problems may also arise with the change in dynamic viscosity of the mixture using these high injection pressures. Concerning this it is the opinion of TNO that, without further research, the injection pressure should be kept below 100 bar.

For the second problem one solution is to use a pressure ratio between the injection vessel and the sphere that is as high as possible, but different from the standard atmospheric test. Using a 2-litre injection vessel instead of a 0.6 litre injection vessel solves the problem of longer discharge times. With the 2-litre injection vessel more air is injected inside the sphere, which results in better dispersion and longer injection times. A different advantage of using the 2-litre injection vessel is that the injection pressure at a certain pressure ratio is less inside the 2-litre injection vessel as it is inside the 0.6 litre injection vessel. This means that a higher pressure ratio can be used when a 2-litre injection vessel is applied. Assuming the use of a 2 litre injection vessel with a maximum pressure of 100 bar, the maximum pressure ratio that can be used is calculated by means of the ideal gas law:

$$P_i \cdot (V_1 + V_2) = P_1 \cdot V_1 + P_2 \cdot V_2 \quad (11)$$

where P_i is absolute initial pressure

P_1 absolute pressure inside the sphere, prior to injection

P_2 absolute pressure inside the injection vessel, prior to injection

V_1 volume of the sphere (= 20 litre)

V_2 volume of the injection vessel (= 2.0 litre).

The maximum pressure ratio that can be applied is calculated to be 10.

In Table 14 the choices for the explosion test set-up for TNO are summarised. It is the opinion of TNO that this test set-up is closest to the ideal test methodology for 15 bar initial pressure we can get. This set-up is used for dust explosion experiments at 10 bar and 15 bar initial pressure.

Table 14. Explosion test methodology for tests at initial pressure of 10 bar and 15 bar.

	Initial pressure = 10 bar	Initial pressure = 15 bar
Nozzle type	nozzle 2	nozzle 2
Dispersion method	dust inside sphere	dust inside sphere
Injection vessel volume	2 litre	2 litre
Pressure ratio	10	10
Injection pressure	55 bar	82.5 bar
Pressure inside sphere	5.5 bar	8.3 bar

7.5 Results of the tests by LOM

Maximum explosion pressure (MEP) and maximum rate of pressure rise (MRPR) were taken as indicators for the severity and turbulence produced in the test in course, and residual mass remaining in the loading vessels was weighed and noted. The amount of residual mass in relation to initial mass was taken as the indicator of good dispersion in the test.

The tests in the 1 m³ hyperbaric vessel of LOM are summarised in Table 15. The criteria to evaluate the performance of the nozzles were as follows:

- C₁ = a failure was considered when the percentage of residual mass found in the charging vessel is higher than 20% in at least in one of the tests.
- C₂ = a failure was considered when the MEP or K_{St}-values are out of the expected range (very low values for a determined pressure) in at least in one of the tests.

Table 15. Summary of dispersion tests.

INITIAL PRESSURE (bar)	OPEN NOZZLE		RING NOZZLE		REBOUND NOZZLE	
	C ₁	C ₂	C ₁	C ₂	C ₁	C ₂
1	OK	OK	FAIL	FAIL	OK	OK
8	FAIL	OK	FAIL	OK	FAIL	OK
15	FAIL	FAIL	FAIL	FAIL	FAIL	OK

It should be borne in mind, that these experiments did not follow any standard method and do not pursue the performance of any standard test with different materials. This research was aimed at studying the behaviour of three variables involved in the dispersion and explosion process for different materials, by means of the development of an statistical random plan design.

General linear models were fitted for the three dependent variables of this experiment, MEP, MRPR and residual mass relating to the four designing factors, two of them categorical: sample identification and dispersion device, and two of them quantitative: initial pressure and injection impulse. Possible interactions between the sample nature and the dispersion device and the injection impulse were introduced in the model.

The mean results of each dispensor, the interaction plots between sample identification and dispersion device for the three dependent variables, and the behaviour observed during the experimental works suggested to discard the second dispensor, the ring nozzle. Table 15 also presented the worst behaviour for the ring nozzle. As can be seen from Figures 24 - 26, this dispensor gives the higher values for residual mass and the lower ones for MEP and MRPR.

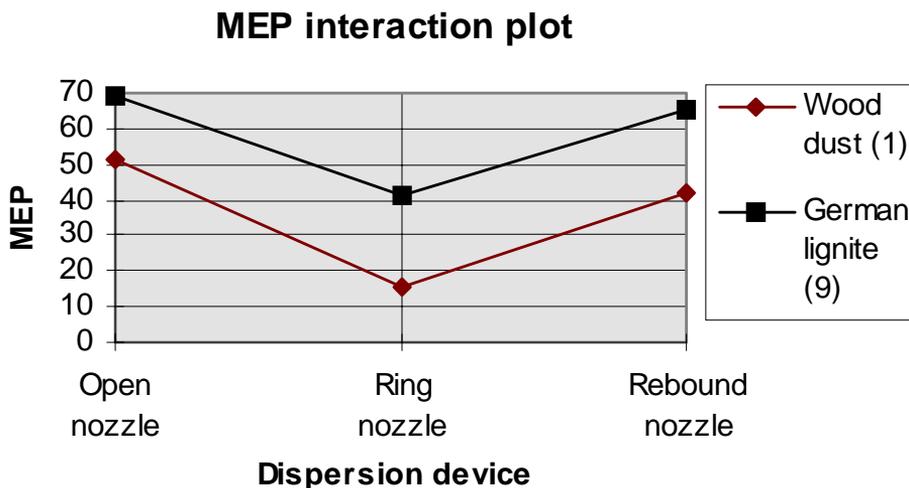


Figure 24. Comparison of MEP mean values (bar).

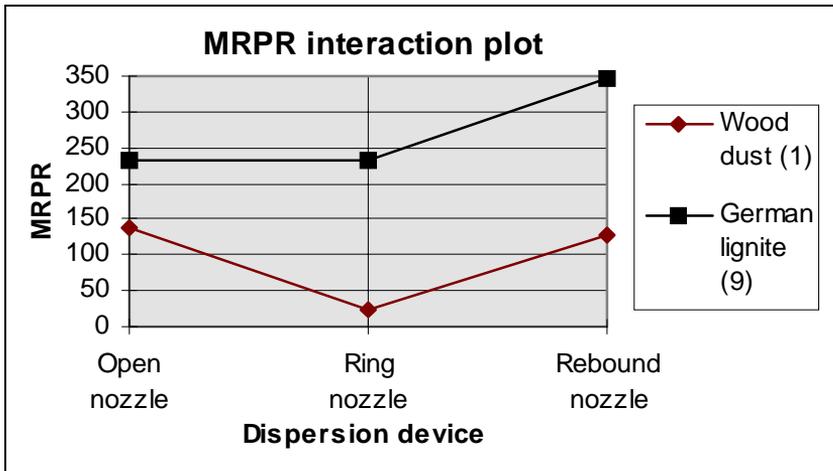


Figure 25. Comparison of MRPR mean values (bar/s).

The different behaviour of biomass compared to that of German lignite can be seen. Thus, the ring nozzle showed the same MRPR mean results for lignite as for open nozzle (Figure 25), and even the lowest residual mass mean (Figure 26). The values for lignite were quite similar and comparable for the three nozzles. This trend was very different for biomass: while open and rebound nozzle values were comparable, ring nozzle values were far from these, and showed the worst dispersion (Figure 26) and explosion results (Figures 24 and 25).

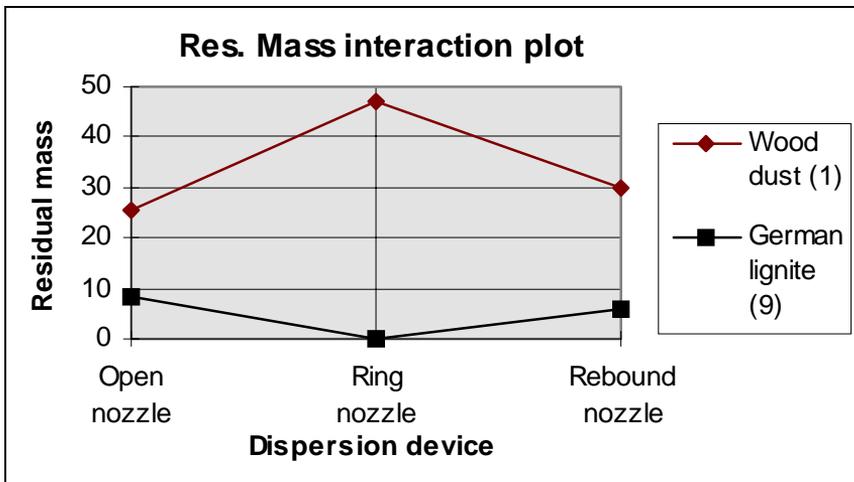


Figure 26. Comparison of residual mass mean values (%).

Once the ring nozzle was rejected, an additional series of tests was carried out. Five tests were added to each block (five for wood dust and five for German lignite), three of them for the open nozzle and two of them for the rebound nozzle (in order to have a comparable number of tests for the two nozzles):

Block 1	Block 2
<u>WOOD DUST</u>	<u>GERMAN LIGNITE</u>
ABC	ABC
100	010
020	200
210	120
002	002
222	222

Due to lack of German lignite, it was impossible to carry out the test 222 of Block 2.

From the analysis of the results the following conclusions can be drawn:

- If the chosen criterion for selecting the best dispersor is maximum severity and minimum residual mass, the following relation was established:

		Rebound nozzle	↔	Open nozzle
MEP	wood	high		low
	German lignite	similar		similar
MRPR	wood	similar		similar
	German lignite	high		low
Residual mass	wood	low		high
	German lignite	similar		similar

- This means that rebound nozzle would be preferred to open nozzle. Table 15 also showed the best behaviour for the rebound nozzle. Furthermore, this nozzle is an enlarged geometrical model of the “standard” nozzle used in the 20 litre apparatus.
- The maximum values of MEP and MRPR were produced for high values of initial pressure and injection impulse. However, the minimum values of residual mass were obtained for low initial pressure and mild injection impulse values.

- Multiple regressions of the dependent variables showed a pronounced effect of injection pressure on the explosion maximum rate of pressure rise MRPR. The initial pressure was found to be a more significant effect on the MEP and residual mass results.
- Apart from the mathematical treatment and results, practical observations gave great experience about the dispersion behaviour of each dispersor and nozzle.

8. Dust explosion tests

8.1 Introduction

Dust explosions are always an actual risk in handling renewable fuels and fuel mixtures. The dusty nature and high reactivity of biofuels, combined with a regular need of a thermal drying stage emphasise the hazards of dust explosions. Experimental data available on explosion characteristics of renewable fuels and fuel mixtures is very limited. This is true particularly considering high-pressure and high-temperature situations. The knowledge of the explosions parameters at high initial pressure and temperatures is fundamental for the design of safety measures at IGCC and PFBC power plants. The objective of this task is to experimentally determine the explosion indices of the selected fuels and fuel mixtures. Because dust explosion tests in these conditions are rather laborious and expensive the programme of this task was limited to a selection of the fuels and fuel mixtures chosen in previous tasks.

Dust explosion tests were performed by four of the partners: TNO (20 litre sphere), LOM (20 litre sphere and 1 m³ vessel), DMT (1 m³ vessel) and INERIS (1 m³ vessel). The equipment and procedures employed by the partners are described and the results summarised in the next chapters.

8.2 Explosion facilities

8.2.1 The 20-litre sphere

The standard 20-litre sphere was used by TNO and LOM in the determination of the explosion characteristics of all fuel samples [15].

The explosion vessel consists of a spherical space of 20 litres, capable of withstanding the maximum static overpressure of 40 bar (Figure 27). The vessel is operated at room temperature. To remove the heat of the explosion a water-jacket is provided.

A dust container (capacity 0,6 litre), in which a known amount of the dust to be tested is pressurised to 20 bar, is connected to the sphere. The dust is dispersed in the vessel (containing air at a sub-atmospheric pressure of 0.4 bar(a)) through a rebound nozzle by opening a valve separating the dust container and the sphere. After dispersion of the dust the pressure in the sphere is atmospheric due to the injection of the compressed air. The dust concentration is defined as the weight of the dust divided by the volume of the vessel.



Figure 27. The standard 20 litre sphere for dust explosion research(TNO).

The dust-air mixture fills the explosion vessel and after a fixed delay time after opening the valve (60 ms) the ignition source in the centre of the explosion vessel is activated. The ignition source is an electric pyrotechnic igniter. The energy released by the ignition source is 10 kJ. The pressure-time history of the explosion is recorded via piezoresistive pressure transducers on a computer.

To determine the maximum explosion overpressure (P_{\max}) and the maximum rate of pressure rise $(dP/dt)_{\max}$, experiments are performed over a wide range of dust concentrations. The maximum values found for the explosion overpressure and rate of pressure rise are reported as the explosion characteristics. The 20-litre sphere and the test procedure have been designed in such a way that the results are commensurate with those from the 1 m³ explosion vessel that is standardised in the ISO standard 6184 Part 1 (1985) and in the VDI guideline VDI 3673 (1979). To this end the measured explosion pressures must be corrected for the relatively larger cooling at the wall. Since the results in the 20-litre sphere show a larger scatter than in the 1 m³ explosion vessel, tests over a wide concentration range have to be performed in triplicate in order to get the same accuracy. The maximum values of the characteristics for each series are then averaged.

The maximum rate of pressure rise depends on the volume of the test vessel, and therefore usually the (volume independent) dust explosion constant K_{St} is quoted. K_{St} is the maximum rate of pressure rise in a volume of 1 m³, calculated with the cubic law.

Using the K_{St} -value, the dusts can be divided into dust explosion classes as follows:

Dust explosion class	K_{St} -value (bar·m/s)
St 1	≤ 200
St 2	201 - 300
St 3	> 300

Both the maximum explosion overpressure and the maximum rate of pressure rise depend on the initial conditions during the explosion. Therefore in practice higher or lower values than the experimentally determined ones may occur. Relevant initial conditions are initial pressure and temperature and level of turbulence. Especially the initial turbulence is not a simple parameter. However, the tests have been designed in such a way that in most practical situations the level of turbulence will not be higher than in the tests.

For the determination of the minimum explosible concentration (MEC) the range of dust concentrations tested in air is extended to lower concentrations. For the highest concentration where no explosion occurs, three tests are performed. For

this determination TNO used a modified ignition source of 2 kJ, while LOM used the normal 10 kJ ignition.

For the determination of the lower oxygen concentration 2 kJ ignitors are also used. By lowering the oxygen concentration step by step the oxygen concentration at which no explosion occurs any more is found. At this oxygen concentration the dust concentration is varied to assure that no dust explosion occurs for any dust concentration.

8.2.2 The strengthened 20-litre sphere

TNO uses a strengthened spherical 20-litre vessel for explosion tests in elevated conditions (Figure 28) [16]. It is constructed of stainless steel, which is capable of withstanding a constant pressure of 150 bar at 250 °C. Since dust explosions rarely generate pressures of more than 10 times the initial pressure, dust explosion testing can be carried out at initial pressures up to 15 bar. The sphere can be pre-evacuated by means of a vacuum pump and has a double wall, allowing circulation of a cooling or heating liquid. In this way the cell can be maintained at temperatures from below zero to 200 °C. The dispersion air is maintained inside the pressure vessel that is separated from the sphere by means of a fast acting valve. At a standard procedure the dust is placed inside this pressure vessel with a volume of 0.6 litre prior to dispersion. Since large amounts of powder are needed at high initial pressures the pressure vessel can be enlarged to 2.0 litre and 6.0 litre. However, previous research has shown that problems arise with the fast acting valve when large amounts of dust must be injected through this valve. Therefore, the procedure developed in this project is used, where the dust is placed on the bottom of the sphere and the dispersion air is directed towards the bottom wall by means of the specially designed nozzle (nozzle 2).

As described previously there are certain advantages of using a 2.0-litre injection vessel. A higher pressure ratio between the injection vessel and the 20-litre sphere can be maintained and the larger pressure vessel results in a longer discharge time. This has the advantage that a larger amount of dust on the bottom of the sphere is also dispersed very well. However, when the same delay time between opening of the fast acting valve and the initiation of the ignition source is used the turbulence at the moment of ignition is much higher when a 2.0-litre



Figure 28. The strengthened 20-litre sphere for dust explosion research (insulation jacket removed)(TNO).

pressure vessel is used. Tests showed that at a standard test procedure it takes about 30 - 40 ms to empty the pressure vessel inside the 20 litre sphere. The standard delay time for the 20 litre sphere is 60 ms. When a 2.0-litre pressure vessel is used, the time to empty the vessel is about 60 - 80 ms, depending on the initial pressure that is needed. For this reason a standard delay time between the opening of the fast acting valve and the firing of the ignitor of 90 ms was chosen for these experiments.

For all test series, tests were performed over a wide concentration range in order to find the maximum values for maximum explosion pressure and maximum rate of pressure rise. The dust was placed on the bottom of the sphere and air from the injection vessel was used to disperse the dust inside the sphere. A pressure ratio of 10 was used between the injection vessel and the sphere. 90 ms after the opening of the fast acting valve the ignitor of 10 kJ was fired and the pressure as

function of time was recorded by means of two pressure sensors placed inside the sphere. From this pressure-time history the maximum explosion pressure and maximum rate of pressure were calculated. For the experiments at elevated initial temperatures insulation was placed around the sphere and the sphere was heated by means of a heating medium (oil) in the double wall of the sphere. The injection vessel does not have a double wall. Therefore a heating jacket was placed around the injection vessel in order to pre-heat the injection air before injection took place.

8.2.3 The 1 m³ vessels

LOM has developed a hyperbarical testing facility for high-pressure dust explosions [17, 18]. The testing station consists of a 1 m³ spherical main vessel connected to a 25-litre cylindrical dust injection container by means of a fast acting detonator valve. After pressuring the main chamber to the desired initial testing pressure, and the dust container to the desired injection pressure, the connecting valve opens to disperse the dust into the main chamber. At a predetermined delay time (600 ms) two 5 000 J chemical ignitors located at the centre of the sphere are activated to initiate the explosion, and then the course of explosion is recorded and processed. Figure 29 illustrates the hyperbaric vessel. This vessel was also used for explosion experiments at ambient pressure.

The method “proportional pressure difference“ described in section 7.2.3 was selected for the injection of the dust. The values of the impulse (ΔP) and final injection pressure ($P_{\text{injection}}$) for increasing initial pressures are shown in Table 16. According to the defined explosion tests methodology, only one loading chamber of the testing station was used during the explosion tests. The selected initial pressures of the explosion tests were 1, 5 and 10 bar, corresponding to normal conditions (atmospheric pressures) and high pressure tests.

DMT determined the explosion characteristics of the different dust samples in a closed, cylindrical 1 m³ vessel built according to VDI-guideline 3673. The length of the 1 m³ vessel was almost equal to its diameter. The dust sample was placed in a 5-litre dust container connected to the 1 m³-vessel, which was then pressurised to 20 bar with compressed air. It was possible to use two dust containers and to double the volume of each to 10 litre. After opening a quick release outlet valve the dust sample was blown into the 1 m³ vessel by expanding

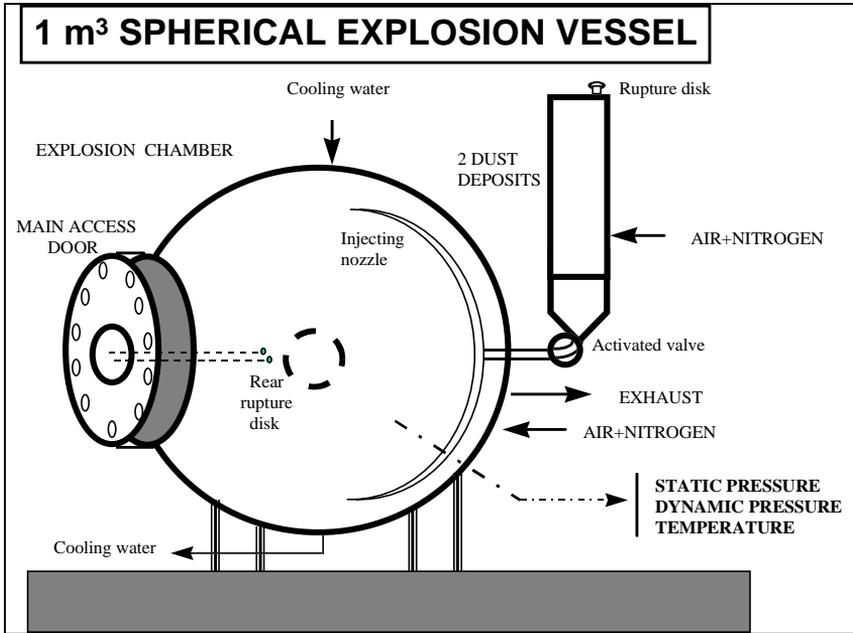


Figure 29. The hyperbaric 1 m³ vessel of LOM.

Table 16. Injection values used during the explosion tests.

P initial (bar)	ΔP (bar)	P injection (bar)
1	20	21
5	23	28
10	26.5	36.5
15	30	45

compressed air. A rebound nozzle was used to disperse the large amounts of dust with low flowability. Generally the dust/air mixtures are ignited by two chemical igniters 600 ms (ignition delay time) after starting the blow-in process of the dust. The igniters are positioned in the middle of the vessel and have a total energy of 10 kJ. Each ignitor has a mass of 1.2 g and consists of zirconium (40% by weight), barium nitrate 30% by weight) and barium peroxide (30% by weight).

For measuring and recording the explosion pressure as a function of time, a quartz pressure transducer combined with an amplifier, control module and PC

data acquisition were used. Throughout the tests the starting dust concentration was varied in steps of appr. 100 g/m^3 until the maximum explosion pressure and maximum rate of pressure rise were obtained.

For tests at elevated temperature DMT used a heatable 1 m^3 vessel of similar configuration (Figure 30) [2]. The strength of the vessels limited the initial pressure to maximum 4 bar and the temperature to $150 \text{ }^\circ\text{C}$.

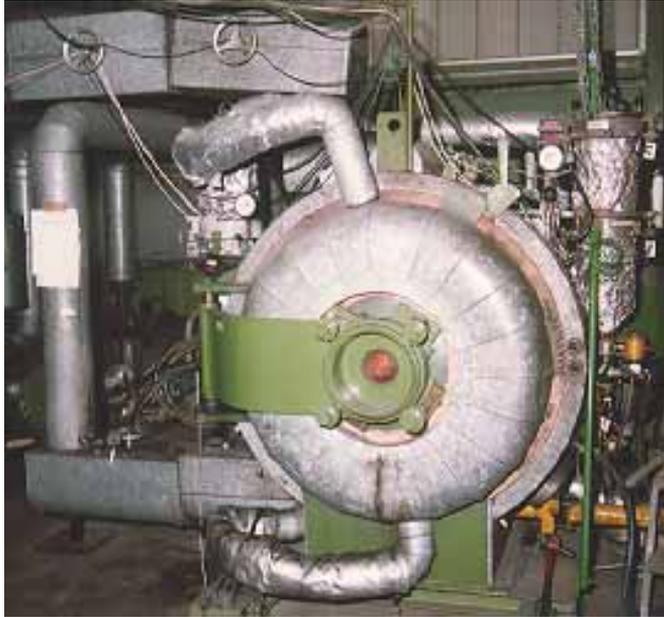


Figure 30. 1 m^3 vessel of DMT.

INERIS carried out tests at elevated pressure using an elongated vessel 1 m^3 in volume (Figure 31) described in the French standard AFNOR U54-540 but which was slightly modified to be able to disperse larger quantities of dust (elevated pressure tests).

The vessel is a cylinder 2.5 m long and 0.7 m in diameter. The dust was dispersed from a reservoir with a capacity of up to 30 litres which was equipped with a LECHLER¹ nozzle (opening of $1\,200 \text{ mm}^2$) and pressurised by the discharge of gas contained in a 8-litre cylinder. The gas is injected into the reservoir

¹ Christopher Proust, Ineris, France, personal information

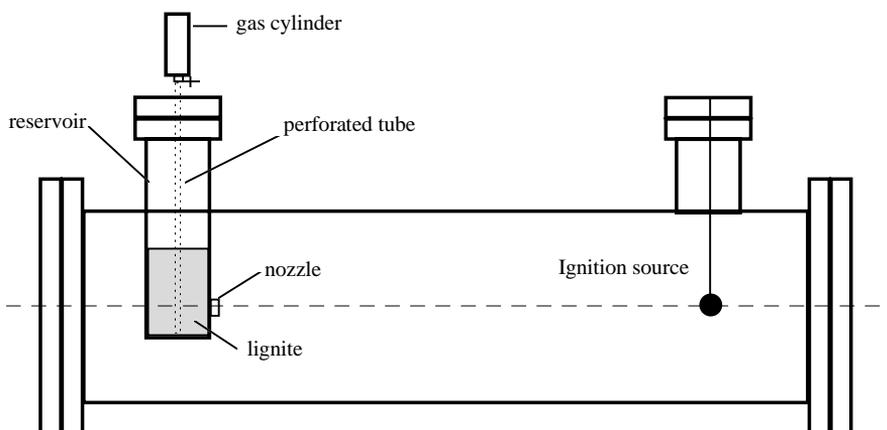


Figure 31. The elongated 1 m³ vessel of INERIS.

by a perforated tube. This reservoir was placed at a distance of 40 cm from one end of the explosion vessel and the nozzle was positioned on the central axis and pointing towards the other end. The ignition source was a 160 kJ pyrotechnical charge placed at 400 mm from the wall at the end opposite the nozzle.

The dust was placed in the reservoir, then the explosion vessel was slowly pressurised (1/2 hour to go from 0 to 10 bar). The dispersion cylinder was pressurised as well. The explosion was created by opening the dispersion cylinder for one second and the ignition source was set off 600 ms after the start of the dispersion process.

During the tests, several technical problems were encountered. With maximum pressures approaching 80 bar and the rate of pressure rise of up to 600 bar/s, mechanical components and seals suffered and required repair after each violent explosion. Another problem observed was that for very violent explosions, an explosion developed inside the dispersion system which was the ignition source for a secondary explosion involving the residual dust in the explosion vessel. As a result, nitrogen was used to pressurise the dispersion system.

The following sensors were used:

- piezoresistive transducer on the vessel wall (0 - 200 bar);
- piezoresistive transducer on the dispersion reservoir (0 - 200 bar)

- piezoresistive transducer on the tube between the gas cylinder and the reservoir (0 - 200 bar)
- precision pressure gages between the vessel and the gas cylinder
- photodiodes, end of the explosion vessel
- the data record one at each by a 16 channel digital datalogger (SMR Kontron).

8.3 Explosion tests at ambient pressure

Dust explosion characteristics were determined by LOM and TNO in the 20-litre sphere in normal ambient conditions (Table 17). All pressures are given as absolute pressures. Corresponding tests in the 1 m³-vessel were carried out at DMT (according to VDI-guideline 3673) and LOM (using the hyperbaric vessel and test procedure) with the chosen fuel samples wood, barley straw, German and Spanish lignites and mixture 2. Additionally, INERIS carried out one test series in the 20-litre sphere and the elongated 1 m³-vessel on German lignite. The maximum explosion pressure P_{max} and the rate of pressure rise described by the K_{st} -value are shown in Figures 32 and 33.

Table 17. Explosion properties of fuels and fuel mixtures.

Dust sample	P_{max} (TNO) bar(a)	P_{max} (LOM) bar(a)	K_{st} -value (TNO) bar·m/s	K_{st} -value (LOM) bar·m/s	LOC (TNO) vol%
Wood	8.6	8.8	115	87	10
Bark	9.0	9.7	132	98	10
Forest residue	8.6	9.1	87	84	10
Spanish pine	7.7	8.2	44	23	17
Barley straw	7.9	9.3	72	58	13
Miscanthus	7.8	8.1	53	31	20
Sorghum	7.3	8.2	41	28	20
Rapeseed straw	6.7	8.3	23	32	20
German lignite	8.6	8.7	146	105	9
Spanish lignite	8.6	8.8	164	107	8
Mixture 1	8.4	9.8	111	104	8
Mixture 2	8.4	9.1	137	103	9

Comparable results should in principle be obtained by all laboratories when using the standard 20-litre procedures or the 1 m³-vessel. Fairly consistent results were measured for the maximum explosion pressure in the 20-litre sphere, although the results obtained by LOM were in general somewhat higher than those of TNO. The woody dusts, the lignite dusts and the mixtures showed similar explosion pressures, while the explosion pressures of straw dust were usually lower. The explosion pressure measured in the 1 m³-vessel were significantly higher. In these tests LOM determined the highest values for barley straw, Spanish lignite and mixture 2. The explosion pressures measured by LOM and DMT are, however, not directly comparable due to different test procedures. The hyperbaric vessel of LOM was not evacuated before the test, resulting in somewhat higher initial pressures at the point of ignition, than allowed by the standard procedure. The value measured for German lignite by INERIS deviated substantially from the results obtained in the standard 1 m³ vessels.

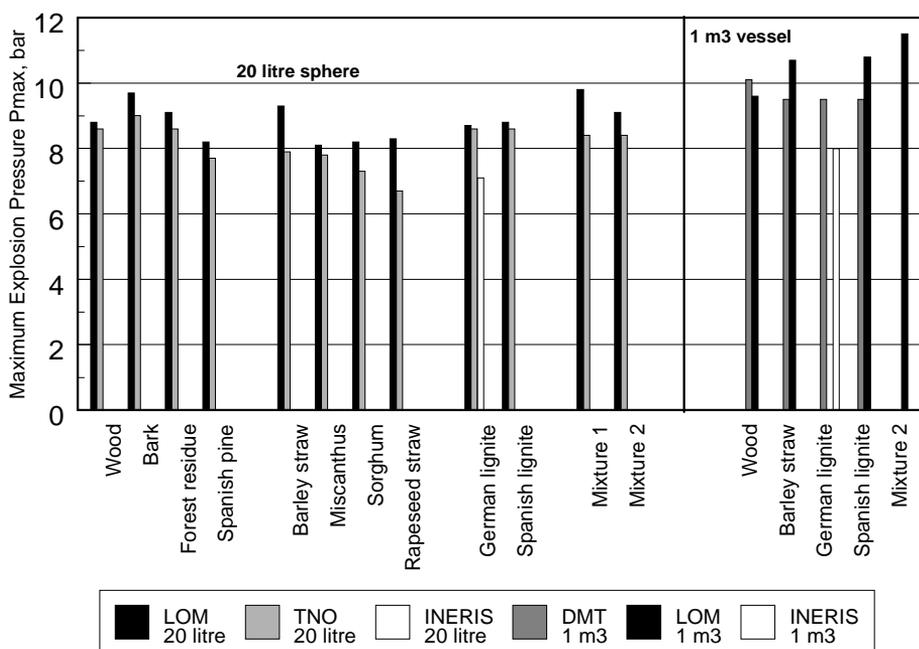


Figure 32. Comparison of P_{max} measured by different laboratories.

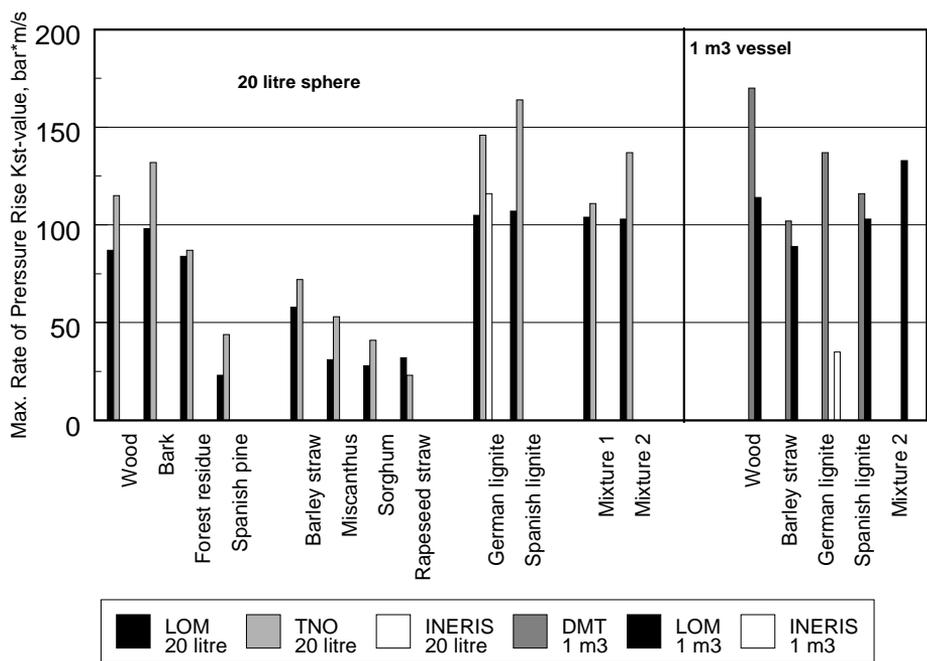


Figure 33. Comparison of K_{St} -values measured by different laboratories.

The dispersion of the dusts into the explosion vessel and the turbulence level inside the vessel at the moment of explosion greatly influence the rate of pressure rise. The variation of the K_{St} -values obtained by the laboratories reflect the difficult nature of the fuel samples in respect of uniform test conditions. However, significant differences were measured by LOM and TNO in the 20-litre sphere also for the more conventional lignite dusts. Also INERIS obtained a very low K_{St} -value for German lignite in their elongated vessel. In general, the straws again indicated the lowest values. The tests at ambient pressure demonstrated clearly the need of additional experience in testing difficult biomass-based fuel dusts. They also indicate the problems probably encountered when proceeding to dust explosion experiments at elevated pressure.

A substantial part of variations in the explosion data must also be ascribed the heterogeneous fuel samples. Classification of coarse and fine particles during handling and transportation and minor changes in the moisture content during storage of the rather voluminous dust samples at the different test facilities could

not be avoided. A more accurate comparison of the tests methodology would require a round-robin test procedure, which was out of scope regarding this research work.

8.4 Explosion tests at elevated pressure

Explosion tests at elevated pressures between 1 and 16 bar were performed for selected fuel samples. Tests were carried out over a wide range of dust concentrations to establish the maximum values for explosion pressure and rate of pressure rise using the apparatus and procedures described in chapter 8.2. The results are presented below as a summary of test series carried out for separate fuel samples. In Figure 34 the maximum explosion pressures are compared as a function of initial pressure. All pressures are, as in previous chapters, expressed in absolute pressure. In Figure 35 the corresponding K_{St} -values are compared.

Figure 34 shows that a linear correlation exists between the maximum explosion pressure and the initial pressure. The maximum explosion pressure is directly proportional to the initial pressure. The results also show a fairly good conformity considering the different tests methods. Further, the explosion pressures of all fuels fall in the same order of magnitude.

Figure 35 shows that an approximately linear relation also exists for the K_{St} -values, except for the INERIS test with German lignite. There is, however, a large discrepancy in the slope of the lines for the same fuel samples. Of the spherical vessels, LOM seemed to obtain consistently lower values than TNO and DMT. For the different fuels, barley straw had lower K_{St} -values than the other fuels, which already was found at ambient pressure. INERIS tests with German lignite suggested a better dispersion of the dust at elevated initial pressure. Most differences in the measured K_{St} -values can probably be associated with diverse turbulence conditions inside the explosion vessel. Various turbulence levels may be ascribed to different equipment and methods, and generally to the inhomogeneous nature of the fuel samples.

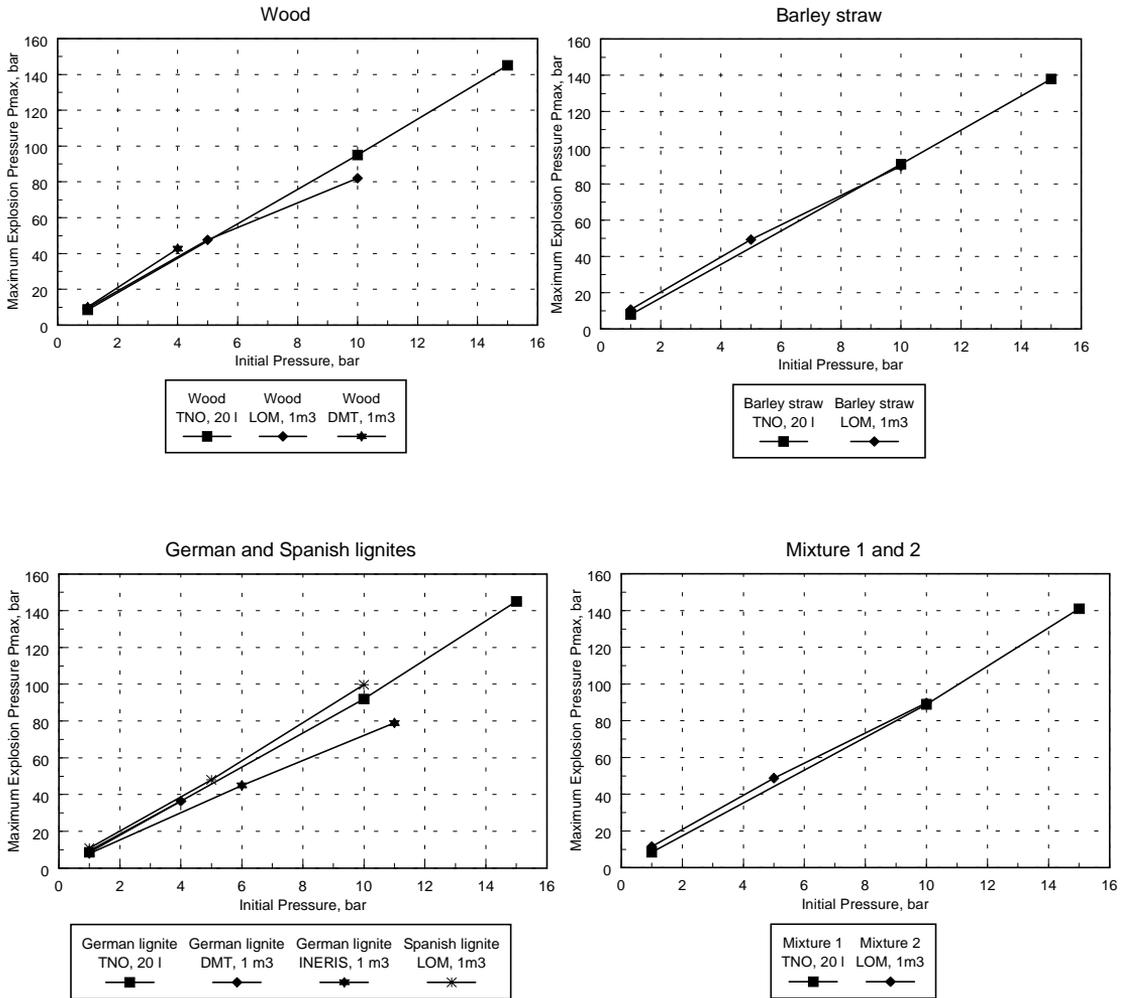


Figure 34. Maximum explosion pressure as a function of initial pressure for different fuel samples.

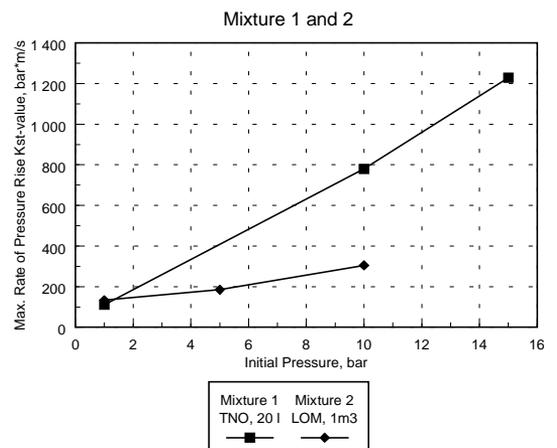
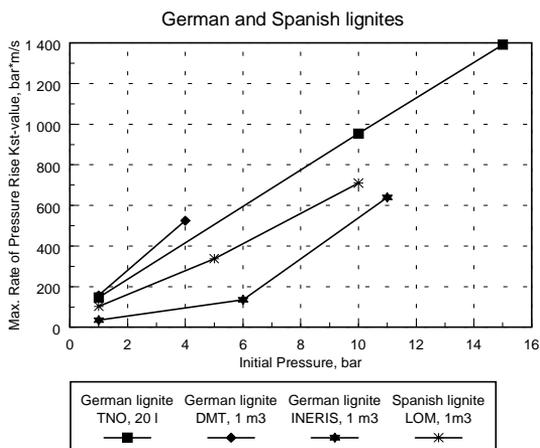
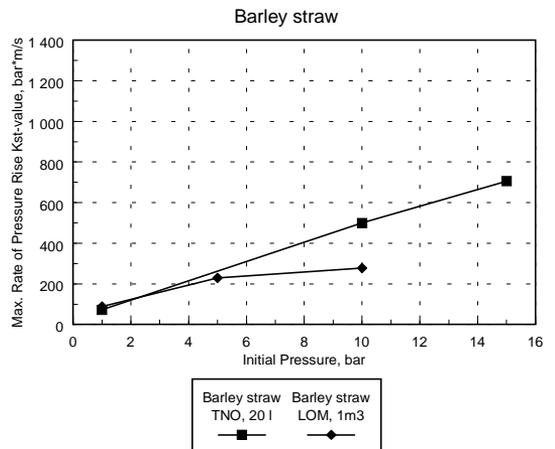
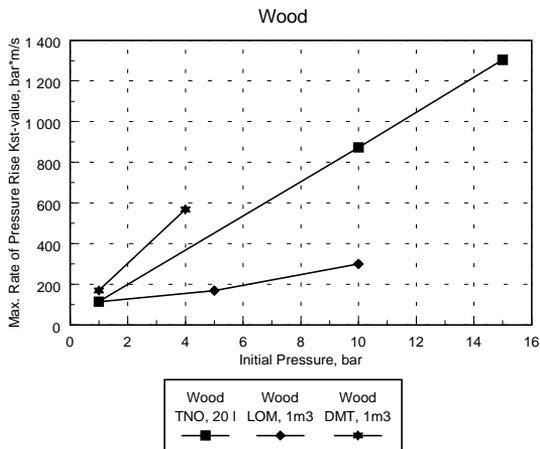


Figure 35. The K_{St} -value as a function of initial pressure for different fuel samples.

One of the main conclusions drawn from these results is that the use of different vessels, within the regulations, is still under discussion. Good comparison should be made between the different vessels and test procedures. Further, it is very likely that the linear relationship between K_{St} -value and initial pressure exists as long as the test procedure is kept the same.

Additional explosion tests at elevated pressure in the 1 m³ were performed by LOM to study the influence of turbulence on the development of the explosion. The sample selected for this work was mixture 1 (German lignite (75%) and wood dust (25%)). All tests were carried out with the same sample to allow the comparison of results, eliminating effects caused by the nature of the dust.

As mentioned earlier, the explosion parameters are strongly affected by the turbulence immediately before and during the explosion. Turbulence depends strongly on the injection process. Therefore, turbulence can be affected by different aspects such as the delay time between injection and ignition, the magnitude of the injecting pressure and the rate of mass discharged into the vessel, which can be closely related to the number of charging vessels from which sample is injected.

From the practical point of view, different turbulence levels were created by using one or two dust containers, at different initial pressures. The delay time was constant for all the tests, and the injecting pressure was constant for each initial pressure, following the “proportional pressure difference” method. Higher concentrations were reached with the use of two charging vessels. Due to the low density of biomass, the use of only one deposit had been a limitation for the previous tests. The range of initial pressure was increased up to 15 bar. The tests were performed by LOM at 5 and 15 bar of initial pressure.

Figures 36 and 37 summarise the maximum explosion pressures and K_{St} -values obtained at different initial pressures, divided in two groups: low turbulence tests (only one dust container used) and high turbulence ones (both containers used). The atmospheric values are taken from the 20-litre apparatus determinations, so they represent standard turbulence. The values presented correspond to the maximum at each pressure over a wide range of dust concentrations.

As can be seen, the increased turbulence had a more significant effect on the rate of pressure rise than on the explosion pressure, and this should be considered for safety measures design. The turbulence affects much more the rate of pressure rise than the explosion pressure itself, because an increase in turbulence produces a higher mobility of particles, the access of oxygen to the surface of the dust particles is promoted and the burning rate greatly increased.

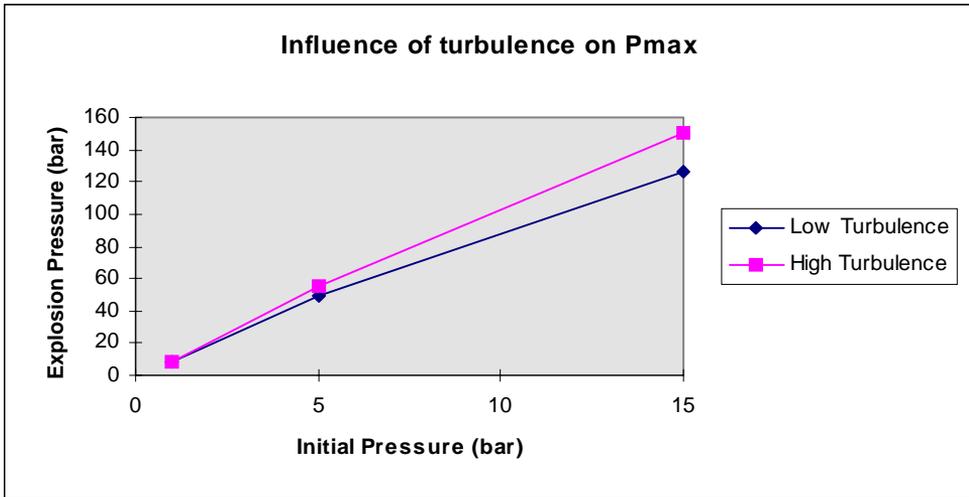


Figure 36. Maximum explosion pressure versus initial pressure at low and high turbulence. Mixture 2, LOM.

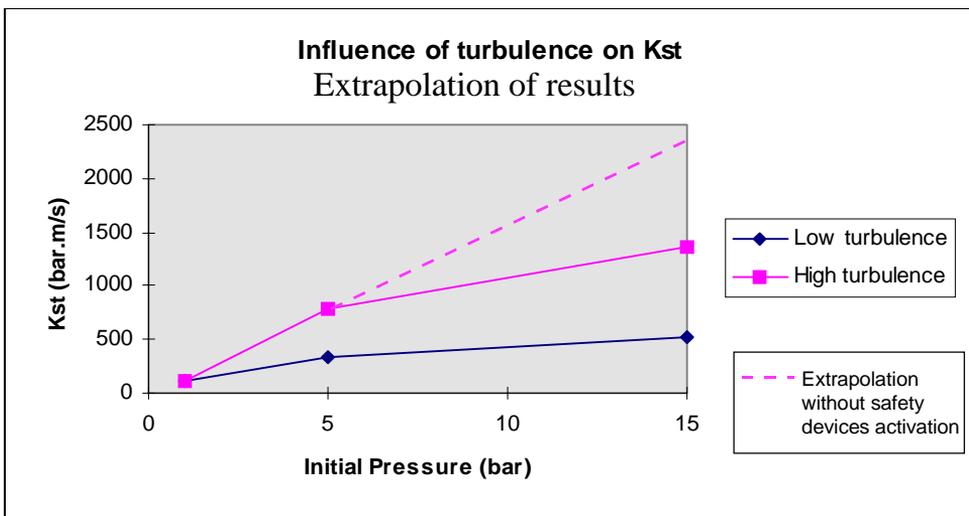


Figure 37. K_{St} -values versus initial pressure at low and high turbulence. Mixture 2, LOM.

The high difference between K_{St} -value at 15 bar at low turbulence (524 bar·m/s) and at high turbulence (1369 bar · m/s) can be seen in Figure 37. The high turbulence value is 2.5 times the value of low turbulence. This phenomenon is not shown to the full extent, because in the high pressure turbulence test at 15 bar, the sphere rupture disks burst out due to the great severity of the explosion, releasing the high rise of pressure. An extrapolation of the high turbulence tests without safety devices activation is shown in Figure 37. A K_{St} -value of about 2 400 bar · m/s could be reached at 15 bar initial pressure.

8.5 Explosion tests at elevated pressure and temperature

Most biomass gasification concepts require a dry fuel if the product gas will be utilised for power production. In many cases this means drying of the renewable fuel, and handling and feeding of the fuel at elevated temperatures. A high temperature, especially in combination with high pressure, influences the explosion risks significantly. To achieve an acceptable and required level of safety in these operating conditions it is necessary to determine the effect of elevated working temperature and pressure on the explosion characteristics.

8.5.1 Test in the 20-litre sphere

TNO performed explosion tests at elevated pressure and temperature in the strengthened spherical 20-litre vessel. It is constructed of stainless steel, which is capable of withstanding a constant pressure of 150 bar at 250 °C (Figure 28).

The following test series were performed: Determination of dust explosion effect at 10 bar and 15 bar initial pressure and 20 °C and 150 °C for the samples: wood and German lignite. For all test series, tests were performed over a wide concentration range in order to find the maximum values for maximum explosion pressure and maximum rate of pressure rise. The dust was placed on the bottom of the sphere and air from the injection vessel was used to disperse the dust inside the sphere by means of the specially designed nozzle (nozzle 2). A pressure ratio of 10 is used between the injection vessel and the sphere.

Air expands at rising temperatures. This means that at higher initial temperatures and the same initial pressure the absolute amount of air (oxygen) present inside

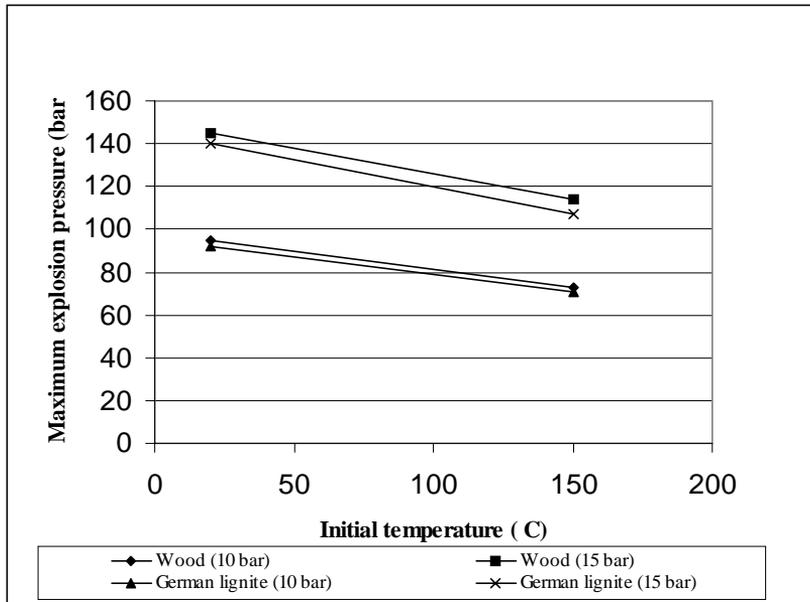


Figure 38. Maximum explosion pressure P_{max} as function of initial temperature, TNO.

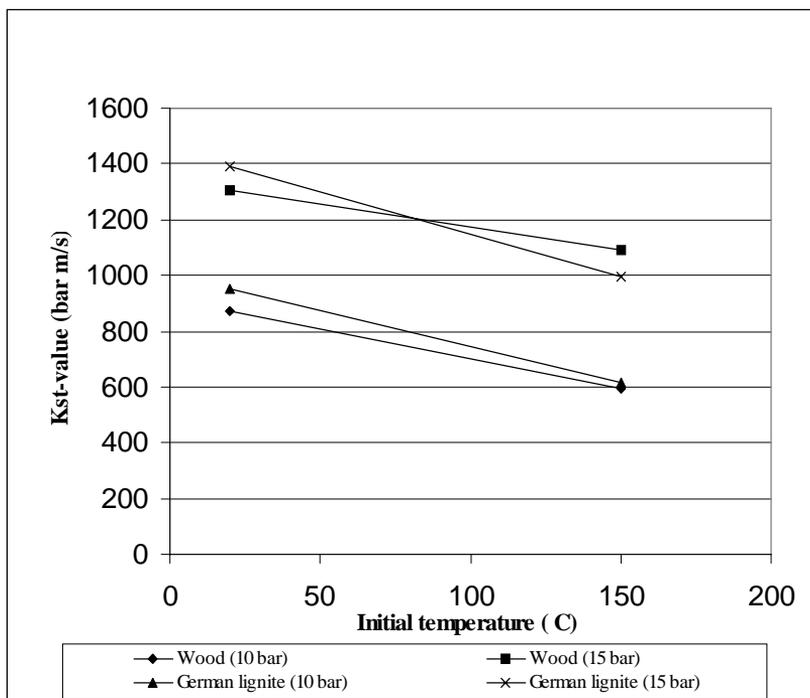


Figure 39. K_{St} -value as function of initial temperature, TNO.

Figures 38 and 39 show that the dust explosion effect (P_{\max} as well as K_{St} -value) decreased with rising initial temperature. Because air expands at rising temperature the absolute amount of air (oxygen) in the 20-litre sphere decreases at high temperature. Due to the smaller amount of oxygen present, a smaller amount of dust can be burned and a decreasing explosion effect is the result. The total amount of oxygen available at 1 bar and 150 °C is about 70% of the amount of oxygen at 1 bar and 20 °C. This is also roughly the ratio between the explosion effects for tests performed at standard conditions and tests at 150 °C.

8.5.2 Tests in the 1 m³ vessel

DMT carried out dust explosion tests at elevated pressure and temperature for two dust samples: wood and German lignite. The tests included determination of explosion indices at 1 bar and 4 bar initial pressure and temperatures of 20 °C and 150 °C. The explosion tests were performed in a heatable 1 m³ vessel. The tests results are presented in Table 18. The reduction of the explosion indices (25 - 30%) at ambient pressure in the temperature range 20 to 150 °C was of the same magnitude as in the high pressure tests carried out by TNO.

Table 18. Results of the explosion tests under elevated conditions in the 1 m³ vessel, DMT.

Dust sample	1 bar, 20 °C		1 bar, 150 °C	
	P_{\max} bar	K_{St} -value bar · m/s	P_{\max} bar	K_{St} -value bar · m/s
Wood	10.1	170	7.3	129
German lignite	9.5	157	7.2	129
Dust sample	4 bar, 20 °C		4 bar, 150 °C	
	P_{\max} bar	K_{St} -value bar · m/s	P_{\max} bar	K_{St} -value bar · m/s
Wood	38.7	568	32.3	562
German lignite	32.5	525	26.9	452

By plotting the results of all tests at elevated pressure and temperature as shown in Figure 40, a relation of the "normalised" explosion pressure (P_{\max}/P_i) and the inverse value of the absolute temperature ($1/T$) is obtained. The regression lines and values for wood and German lignite are indicated in the figure.

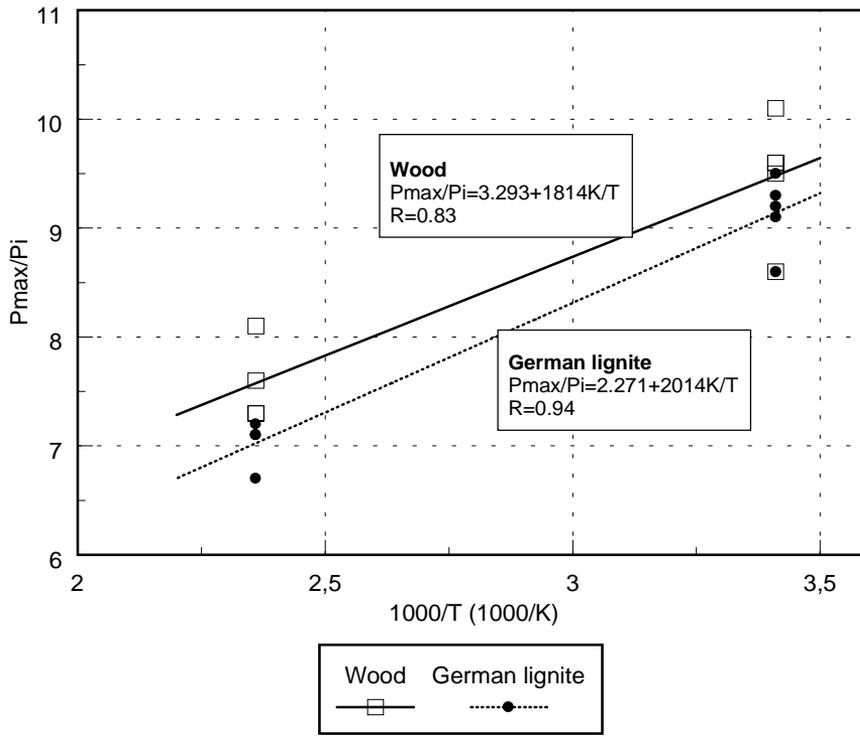


Figure 40. The relation of explosion pressure and temperature.

8.6 Explosion tests at reduced oxygen concentration

Knowledge of the explosion is a prerequisite for designing appropriate pressure relief devices for atmospheric handling systems. Pressure relief cannot always be applied and in these cases sufficient inertisation is required. With inertisation the oxygen concentration is reduced to such a level that an explosion no longer occurs. Inertisation and explosion suppression are the only means to prevent dust explosions in pressurised fuel handling systems. Suppression systems for high pressure applications are not yet commercially available. The objective of this task is to experimentally determine the required lower oxygen concentration (LOC) to inhibit a dust explosion in both ambient and elevated (high pressure and high temperature) conditions.

8.6.1 Tests in ambient conditions

Inerting tests to determine the lower oxygen concentration (LOC) were performed at normal pressure by TNO for all dusts (20-litre sphere), and by LOM (20 litre sphere), DMT (m³ vessel) and INERIS (1 m³ elongated vessel) for selected dusts. Nitrogen was used as inerting gas in all experiments. The results are shown in Figure 40. There is a considerable discrepancy between the LOC values obtained in the different vessels for the same fuel samples. The different procedures used and the nature of the fuel samples discussed before might be the most important factors for the variation of the results. Limiting oxygen concentrations for lignites and biomasses are usually known to vary between 11 and 13%. In this respect the values measured by TNO in the 20-litre sphere for the woody biomasses and especially for the lignites and the mixtures are surprisingly low. The measurements were checked by repeating the test with German lignite. The duplication test gave, however, the same result. A satisfactory explanation for the low LOC values could not be found.

The LOC values of the most of the straws are also considerably higher. This is, however, in good correlation to the low reactivity properties and mild explosion indices of these fuels.

Different procedures were used by TNO and LOM in the 20-litre experiments. TNO used an ignitor with an energy of 2 kJ instead of 10 kJ in these experiments. LOM followed the procedure and criterion for the determination of the lower oxygen concentration in the 20-litre apparatus as described in the document of CEN/TC305/WG1 Method for the determination of the lower oxygen concentration for dust clouds. The equivalent values for the 1 m³, shown in Figure 41, were calculated according to the correlation equation given by Cesana and Siwek [19]:

$$\text{LOC (1m}^3 \text{ vessel)} = \text{LOC (20-litre apparatus)} \cdot 1.64 \quad (12)$$

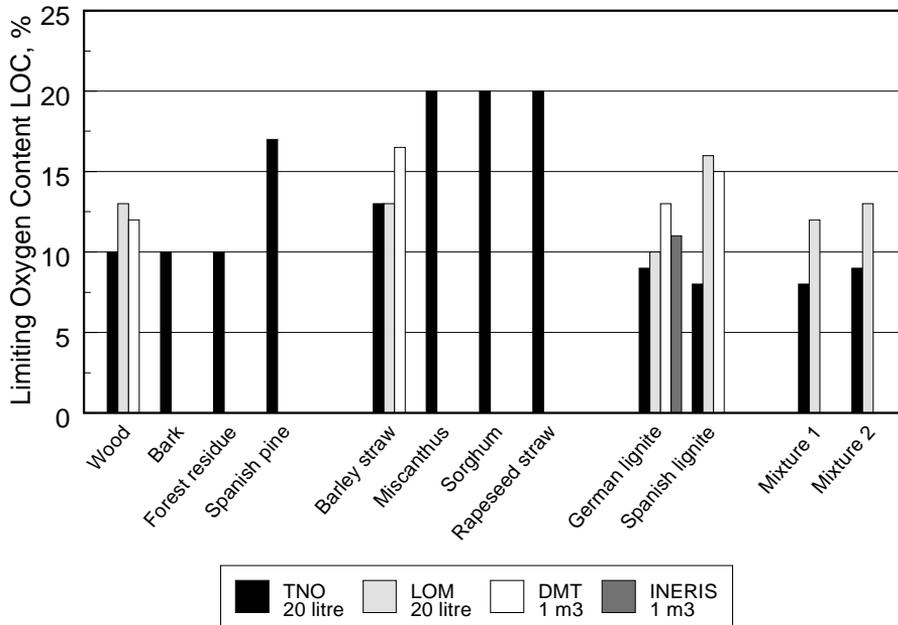


Figure 41. Lower oxygen concentration of different fuels at ambient pressure.

8.6.2 Tests in elevated conditions

Determination of lower oxygen concentration at elevated conditions (high pressure, high pressure and temperature) was carried out by TNO, LOM DMT and INERIS for selected fuel samples: wood, barley straw, both lignites and mixture 2. The results are shown in Figure 42 for wood and in Figure 43 for the lignites and mixture 2 (barley straw/Spanish lignite).

As shown in Figures 42 and 43 the LOC slightly increases for most of the cases with increasing initial pressure, even considering the low values measured by TNO in ambient conditions. The tendency is clear for wood but for the lignites and the mixture the effect of initial pressure is more irregular. Theoretically, an increase as well as a decrease of LOC with increasing initial pressure is possible. Experiments have shown that a decrease of LOC at higher initial pressures is expected when a decrease in lower explosion limit of the dust is observed with increasing initial pressure as well. When the lower explosion limit increases with increasing initial pressures also an increasing LOC is expected [11]. However, for some dusts (e.g. brown-coal) the opposite is also observed [20]. There is not yet an explanation for these contradictions.

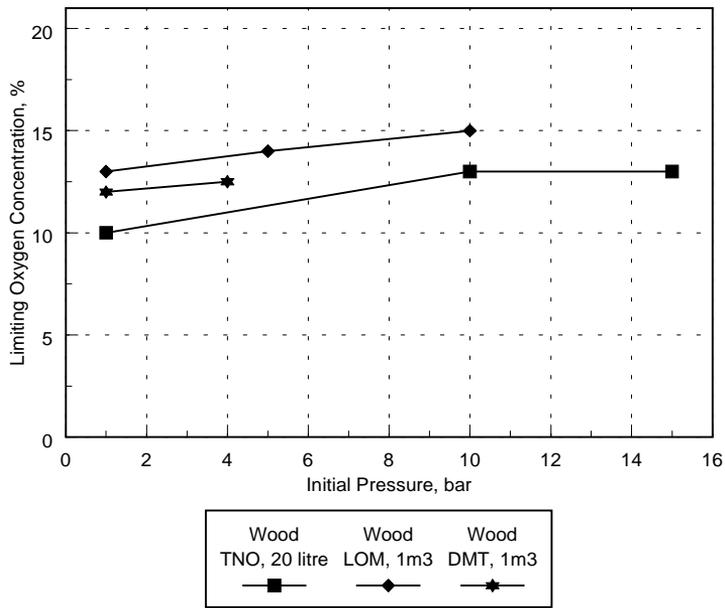


Figure 42. Lower oxygen concentration as a function of initial pressure, wood.

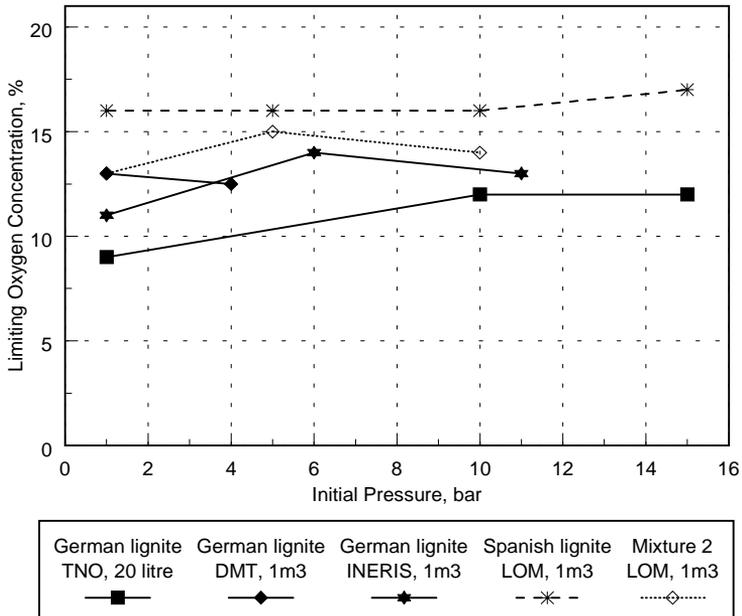


Figure 43. Lower oxygen concentration as a function of initial pressure, lignites and mixture 2.

Figure 44 shows a decrease of LOC at increasing initial temperature. Previous research [21] showed that the explosible range is widened with increasing temperature. The LOC therefore decreases with increasing initial temperature. Until an initial temperature of 200 °C and at atmospheric initial pressure Glarner found a linear decrease in LOC. For all powders tested this decrease was more or less the same, ca. 1.8 percentage units for 100 °C temperature rise. Regarding the accuracy of the test results and the fact that only gas mixtures with a full percentage of oxygen are tested this is, more or less, also in line with the results found within this project. In this study the corresponding reduction was 1 - 3 percentage units.

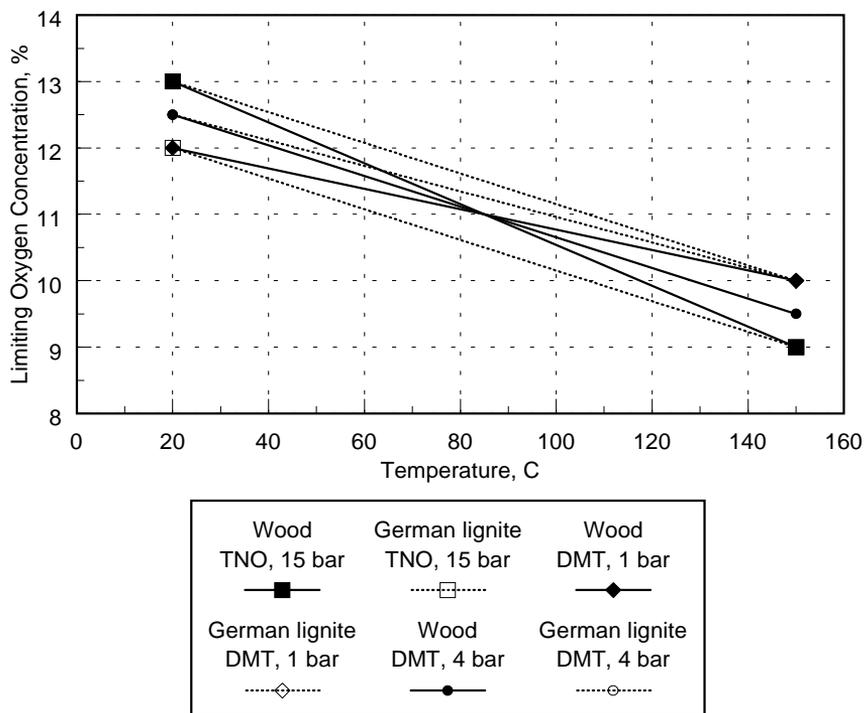


Figure 44. Lower oxygen concentration as a function of temperature, wood and German lignite.

8.7 Minimum explosive dust concentration

As mentioned before, dust explosion tests are carried out for several different dust concentrations. The minimum explosive dust concentration MEC represents the concentration below which no explosion occurs. The optimum concentration gives the highest explosion pressures and rate of pressure rises. In most cases two slightly different optimum concentrations can be detected corresponding to the two explosion indices. Figure 45 shows a typical explosion test carried out for a wide range of dust concentrations and four different oxygen concentrations.

The minimum explosive dust concentration influence of the dust concentration was measured for all fuel samples by TNO and LOM in ambient conditions in the 20-litre sphere. DMT performed the corresponding determinations for selected samples in the 1 m³ vessel at both ambient and elevated conditions. The results are presented in Table 19.

Higher minimum explosive concentrations were measured for the biomass samples compared to the lignites. The less reactive and generally coarser straw samples exhibit considerably high values. The optimum dust concentration at ambient pressure ranged from 750 to 1 500 g/m³ for biomass samples and from 375 to 750 g/m³ for the lignites and mixtures.

The influence of pressure and temperature on the minimum explosive concentration was identical for wood dust and German lignite (Table 19). With increasing pressure the MEC shifted from 50 g/m³ to 200 g/m³. This shift is proportional to the initial pressure (1 bar and 4 bar). The increase in temperature to 150 °C led to a drop in the MEC from 50 g/m³ to 30 g/m³. With 150 °C and 4 bar a stronger temperature effect was observed. The MEC dropped from 200 g/m³ to 80 g/m³, i.e. to less than 50% of the MEC at 20 °C.

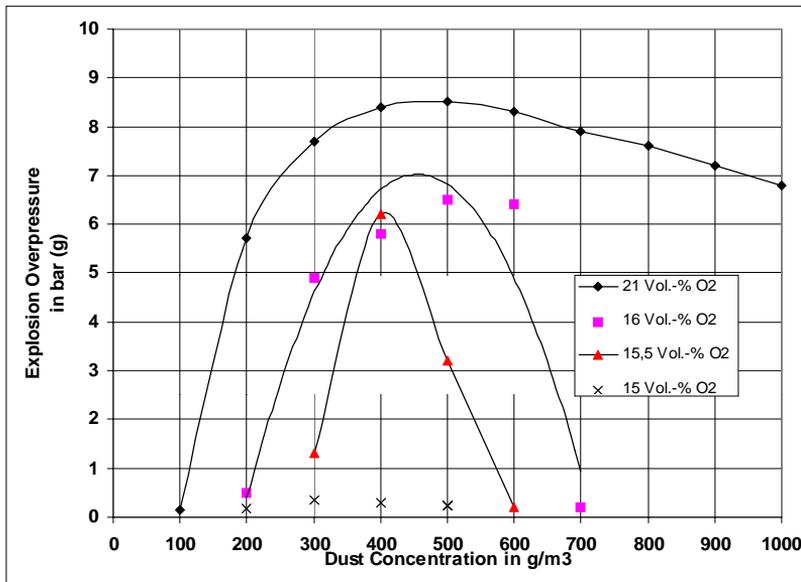


Figure 45. Explosion behaviour of Spanish lignite (1 bar, 20 °C) (DMT).

Table 19. Minimum explosive dust concentrations.

Dust sample	TNO 1 bar, 20 °C g/m ³	LOM 1 bar, 20 °C g/m ³	DMT 1 bar, 20 °C g/m ³	DMT 1 bar, 150 °C g/m ³	DMT 4 bar, 20 °C g/m ³	DMT 4 bar, 150 °C g/m ³
Wood dust	100	30	50	30	200	80
Bark	100	30				
Forest residue	150	60				
Spanish pine	350	90				
Barley Straw	250	90	200			
Miscanthus	450	120				
Sorghum	1 150	120				
Rapeseed straw	750	210				
German lignite	60	60	50	30	200	80
Spanish lignite	60	90	100			
Mixture 1		90				
Mixture 2		150				

9. Predictive criterion

9.1 Introduction

The objective of this task is to create a predictive criterion based on simple tests and analyses for evaluating the reactivity of the fuels and fuel mixtures with regard to their safety technical characteristics.

The method followed has been the statistical treatment of the results obtained during the initial stages of the project, in order to find some representative relations between some physical parameters that are accepted as indicators of the fuel reactivity and the chemical analyses of such fuels. The construction of those relations is aimed at the prediction of the theoretical reactivity of a determined fuel as a function of its chemical composition.

9.2 Methodology and results

All the tests results produced by all the partners during the former stages of the project were collected and compiled in a database. The parameters, including physical and chemical characteristics, are listed below, showing in brackets the laboratory that produced the data. The parameters names were given by LOM for the mathematical treatment (Table 20).

Some of the mentioned parameters had been determined only over a reduced number of samples, losing weight and significance in the mathematical treatment of the data. Parameters represented by few cases were eventually taken out of the database.

Some significant relations and transformations over the measured chemical values were introduced:

- Chemical canonical variables: LOM developed some years ago a method to evaluate the explosion risk [22], based on the fact that the chemical parameters are correlated with the parameters that characterise the explosibility, grouping these two sets of variables in two canonical variables. The chemical canonical variable takes into account the proximate and ultimate analysis of the samples. The chemical canonical variable V_{chem} , widely proved by LOM along the years, was introduced into the database for each sample.

Table 20. The parameters names given by LOM for the mathematical treatment.

Parameter	Identification
IDE	Identifying number of the sample
SAMPLE	Description of the sample
d50INERIS	Mean diametre (INERIS)
densINERIS	Real density (INERIS)
T50INERIS	The temperature $\Delta\Theta$ at which $\Delta T=50^{\circ}\text{C}$. DTA-TGA (INERIS)
WprelossINE	Sample weight before large weight loss. DTA-TGA (INERIS)
Residuoine	Residue weight after combustion. DTA-TGA (INERIS)
LosstempINE	Temperature at which there is a large weight loss. DTA-TGA (INERIS)
T8cm3ine	Critical temperature 8 cm ³ cell. Isothermal oven. (INERIS)
T120cm3ine	Critical temperature 120 cm ³ cell. Isothermal oven. (INERIS)
T340cmine	Critical temperature 340 cm ³ cell. Isothermal oven. (INERIS)
T1000cm3ine	Critical temperature 1000 cm ³ cell. Isothermal oven. (INERIS)
S%lom	Ultimate analysis. Sulphur content. (LOM)
C%lom	Ultimate analysis. Carbon content. (LOM)
H%lom	Ultimate analysis. Hydrogen content. (LOM)
N%lom	Ultimate analysis. Nitrogen content. (LOM)
Humlom	Proximate analysis. Humidity content. (LOM)
Ashlom	Proximate analysis. Ash content. (LOM)
Vollom	Proximate analysis. Volatiles content. (LOM)
Heatcomblom	Heat values. (LOM)
D50lom	Mean diametre (LOM)
Apardenslom	Apparent density (LOM)
Denslom	Real density (LOM)
MITlom	Minimum ignition temperature in layer. (LOM)
MITclom	Minimum ignition temperature in cloud. (LOM)
MEClom	Minimum explosion concentration (LOM)
MIElom	Minimum ignition energy (LOM)
MEPlom	Maximum explosion pressure (LOM)
MRPRlom	Maximum rate of pressure rise (LOM)
Kstlom	K_{St} -value. (LOM)
D50tno	Mean diametre (TNO)
RelHumtno	Relative humidity (TNO)
Meptno	Maximum explosion pressure (TNO)
Kst-tno	K_{St} -value (TNO)
MEC-tno	Minimum explosible concentration (TNO)
LOC-tno	Limiting oxygen concentration (TNO)
Meptno	Maximum explosion pressure (TNO)
Kst-tno	Kst value (TNO)
LEL-tno	Lower explosion limit (TNO)
LOC-tno	Limiting oxygen concentration (TNO)
Pmaxdmt	Maximum explosion pressure 1 m ³ (DMT)
Kstdmt	Kst value 1m ³ (DMT)
LELdmt	Lower explosion limit 1m ³ (DMT)

- Correction of the hydrogen content determined through the ultimate analyses, due to the humidity content given by the proximate analyses.
- Correction of the carbon content determined through the ultimate analyses, due to the CO₂ content obtained in the proximate analyses.
- The volatiles were also corrected, due to the CO₂ error induced by the carbonates.

The hydrogen to carbon ratio (H/C) was also introduced in the database. This relation is used in the typical experiments for the rank evaluation of coals.

The first step of the mathematical analysis over the database results was a multiple variable correlation study. Although this work is focused mainly on reactivity, some correlations between the different tests carried out by all the laboratories were found very significant:

- The Minimum Ignition Temperature on a layer (MIT₁) test developed by LOM with critical temperatures obtained in the isothermal oven test carried out by INERIS, and also with the large weight loss temperature and the temperature $\Delta\Theta$ at which $\Delta T = 50\text{ }^{\circ}\text{C}$ ($\Delta\Theta$) given by the DTA-TGA tests from INERIS (see Figure 4).
- The weight loss temperature and $\Delta\Theta$ from the DTA-TGA (INERIS) with the volatiles and hydrogen content (LOM) of the samples.
- The ashes content given by LOM with the residue obtained in the DTA-TGA tests from INERIS.
- The K_{St} values obtained by LOM with the K_{St} and MEP values obtained by TNO.
- The apparent density (LOM) of the samples was found inversely correlated to the critical temperature of the isothermal oven test (INERIS).

Since there are no standards defining accurate expressions for the reactivity of a material, it is usually evaluated through the measurement of some characteristic parameters, widely accepted by the laboratories involved in such analyses.

The parameter selected as a reference for the reactivity in ambient pressure of the samples was the temperature $\Delta\Theta$ at which $\Delta T=50^{\circ}\text{C}$ obtained through the DTA-TGA analysis by INERIS.

Several multiple regressions were obtained over the $\Delta\Theta$ variable as a function of the chemical analyses measured by LOM. The most significant ones are showed below:

$$R_1 = 205.53 - 1.6742 \cdot C + 1.4498 \cdot Vol \quad (13)$$

$$R_2 = 15.783 - 0.7298 \cdot C + 30.792 \cdot H + 0.09921 \cdot Hum + 2.7973 \cdot S + 0.80602 \cdot Vol \quad (14)$$

$$R_3 = 24.72 \cdot H + 11.2471 \cdot Vchem \quad (15)$$

$$R_4 = 91.2688 + 20.2587 \cdot Vchem \quad (16)$$

where C is C%lom (carbon)

H H%lom (hydrogen)

S S%lom (sulphur)

Hum Humlom (humidity)

Vol Vollom (volatiles)

$Vchem$ chemical canonical variable = $f(C,H,S,Hum,Vol,Ash)$.

The values obtained for each sample for the described regressions are drawn in Figure 46, comparing them with the results measured in practice through the $\Delta\Theta$ in the DTA-TGA analyses. The regressions appear in order of predicting goodness.

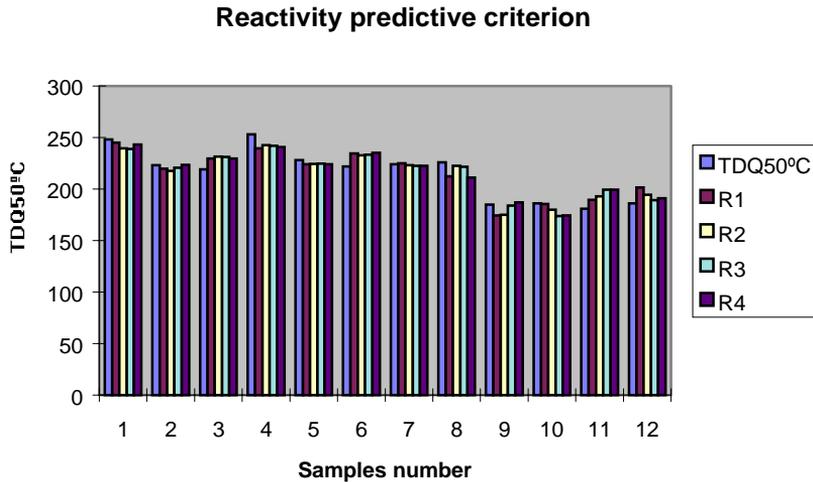


Figure 46. Results of measured and calculated reactivity data. $\Delta\Theta=TDQ50^{\circ}C$ (INERIS). Sample number referring to chapter 5.1.

All the calculated regressions show an statistically significant relationship between the variables above the 95% confidence level, and have a high percentage of explanation of the variability in $\Delta\Theta$ (above 82%).

9.3 Discussion of results

As described before, the conventional tests used in the assessment of self-combustion of materials, such as MIT_i, TGA and isothermal oven have showed a high correlation.

As stated by INERIS, there are three groups regarding fuels reactivity:

- the most reactive samples (the two lignites)
- the moderately reactive samples (agro samples, forest residue and bark)
- the least reactive samples (wood dust and Spanish pine)

The mixtures are very close in reactivity to coal, which is their main component.

The calculated indicators of the reactivity of these samples are a good tool to evaluate the reactivity magnitude of a sample based only in chemical characteristics of fuels, being perfectly valid to differ which group of reactivity a sample pertains to.

However, the low number of data existing for each test restricts the accuracy of the calculated regressions and the number of dependent variables able to enter the fitted models. The complementation of the database with a higher number of results would offer the possibility of refining the models.

10. Dust explosion suppression

10.1 Introduction

Explosion suppression is a procedure whereby the incipient explosion is detected and extinguished, with a suitable suppressant, before the explosion pressure exceeds the vessel pressure shock resistance. In the suppression system, the dust explosion is suppressed immediately after ignition, before the explosion pressure reaches the design pressure of the equipment or the bin. Thus like explosion venting, the design criteria for suppression require that

$$p_{\text{red}} < p_s \quad (17)$$

where p_{red} is the reduced (suppressed) explosion pressure and p_s is the pressure load corresponding to the minimum yield strength of the vessel.

The pressure rise due to dust explosion is indicated by a sensitive pressure detector based on pressure difference or on the rate of pressure rise, the valves of the extinction vessels are released at pressure p_A , and the dust explosion is suppressed at its initial stage. The overpressure p_{red} formed in the vessel is controlled either by designing the construction sufficiently strong or, if an atmospheric system is concerned, by depressurizing through explosion vents.

As an explosion suppression system is an active system, its effectiveness depends on the reliability of the components used in the detection control and release of chemical suppressants. A typical system would normally consist of pressure detectors, suppressors, and a control unit to provide a process/system interface. The choice of suppressant is critical. The suppressant must have free flow capabilities and efficient heat absorption characteristics so that, when introduced into the flame kernel, the chemical removes the heat energy quickly and effectively. The selection of suppressant is often undertaken in consultation with the user and depends on the type of process to be protected. In the food and pharmaceutical industries, for instance, it would be beneficial to select a soluble or chemically clean agent, such as sodium bicarbonate, to reduce the time spent for cleaning down following an activation. In areas where contamination is not

a problem it is more appropriate to select a high-efficiency suppressant such as monoammonium phosphate.

The pressure/time curves of suppressed/unsuppressed explosions are shown in Figure 47. The reduced explosion overpressure depends on the activation overpressure p_A created by the explosion until the explosion suppression system is activated. The rate of pressure rise at that time is $(dp/dt)_{t=t_A}$.

In plants operating at atmospheric conditions protected by explosion suppression system, it is common practice to activate the explosion suppression system at an activation overpressure less than 100 mbar above the normal working pressure. This prevents a false activation of the explosion suppression system caused by pressure peaks. Normally this kind of plants must have a design strength p_s equal to twice of the working pressure (i.e. 1 bar (g)).

As a first approach the design strength/working pressure ratio can also be applied in systems operating at elevated working pressures. In these systems the use of a detector based on pressure difference may be limited. At elevated working pressure a dynamic pressure detector, which triggers the explosion suppression system at a certain rate of pressure rise dp/dt within a certain time window could be an adequate means of detecting an explosion in an early stage.

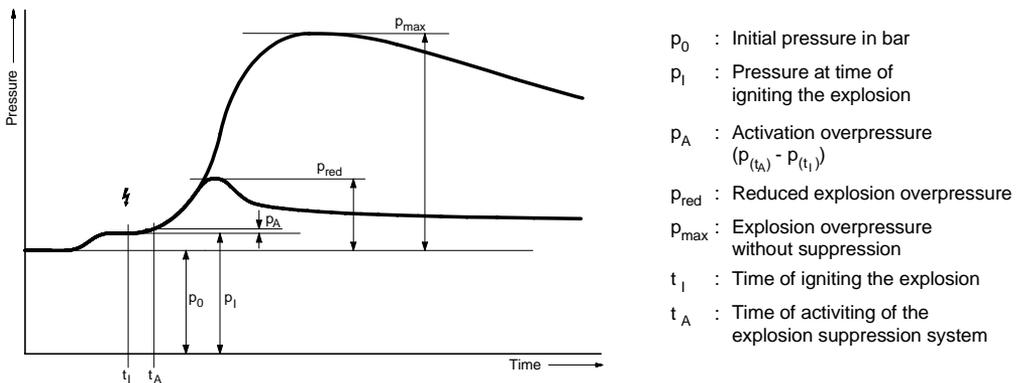


Figure 47. Principle of explosion suppression.

10.2 Explosion suppression tests

The explosion suppression tests were conducted by DMT in the heatable 1 m³ vessel. The explosion suppression system consisted of an 12.3 litre HRD (high rate discharge) container filled with monoammonium phosphate (4 kg and 8 kg), that was pressurised with nitrogen (p=120 bar(g)), and a static pressure sensor. After detecting the explosion at the activation overpressure p_A , monoammonium phosphate was blown into the 1 m³ vessel through two nozzles with a diameter of 1 inch. Figure 48 shows the experimental set-up. The explosion suppression tests were started with the standard amount of 4 kg suppressant agent for protecting a vessel of 1 m³ and were continued with a mass of 8 kg.

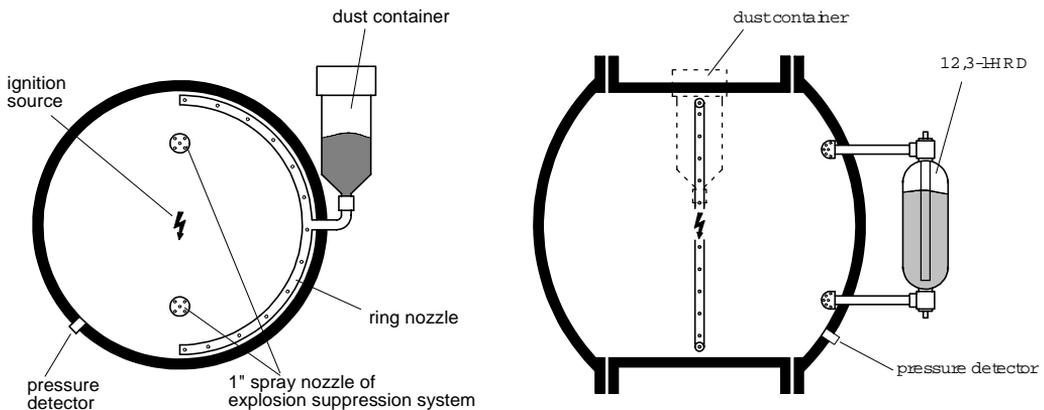


Figure 48. Test equipment for the explosion suppression tests, DMT.

The explosion suppression tests were performed on wood dust and German lignite. Tests were carried out at 20 °C and 150 °C. The optimum dust concentration of 2000 g giving the highest K_{St} value for these dusts at 20°C was used. The K_{St} values are summarised in Table 21.

The reduced explosion overpressure p_{red} was determined in respect of the activation pressures p_A of the explosion suppression system. Additionally, the rate of pressure rise was determined at the time of activation $(dp/dt)_{t=t_A}$.

Table 21. Explosion characteristics of the fuel samples for the explosion suppression tests at 4 bar and 20 °C/150 °C.

Sample	Dust concentration g/m ³	K _{St} value at 20 °C bar · m/s	K _{St} -value at 150 °C bar · m/s
Wood dust	2000	568	562
German lignite	2000	525	452

10.3 Combined protection tests

By using a partly inert atmosphere it should be possible to increase the efficiency of the explosion suppression in systems. This is important especially in elevated conditions, which otherwise may restrict the use of explosion suppression. The suppression test programme included tests with wood dust at 4 bar and 150 °C in reduced oxygen concentration. The oxygen concentration was 17 vol%, which is 7 percentage units above the limiting oxygen concentration of wood dust at 4 bar and 150°C.

10.4 Results and discussion

In Figures 49 and 50 the reduced overpressure p_{red} is shown as a function of the activation pressure p_A for wood dust and German lignite. The initial pressure was 4 bar. If the requirement on the design strength is two times the working pressure, the reduced overpressure should be at most 4 bar(g).

At 4 bar initial pressure and normal temperature with a standard amount of 4 kg of suppressant and a static activation overpressure of 100 mbar the reduced explosion pressures determined for wood dust explosions were greater than 20 bar(g). By doubling to 8 kg of suppressant agent the reduced explosion pressures were higher than 10 bar(g). To achieve reduced explosion pressures equal to double working pressure the static activation overpressures must be lower than 15 mbar respectively 40 mbar for an amount of 4 kg respectively 8 kg mono-ammonium phosphate.

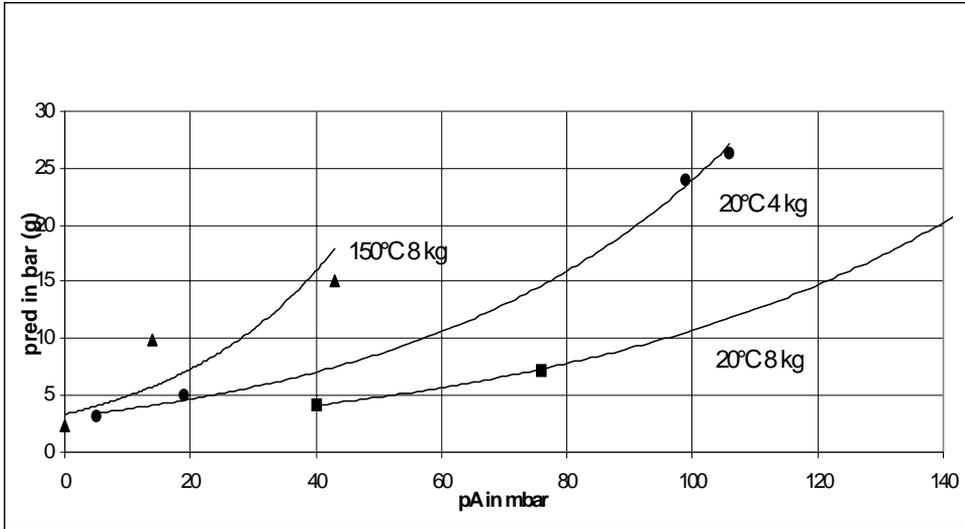


Figure 49. Explosion suppression tests with 2000 g/m^3 wood dust at 4 bar and $20^\circ\text{C}/150^\circ\text{C}$, 4 kg/8 kg monoammonium phosphate.

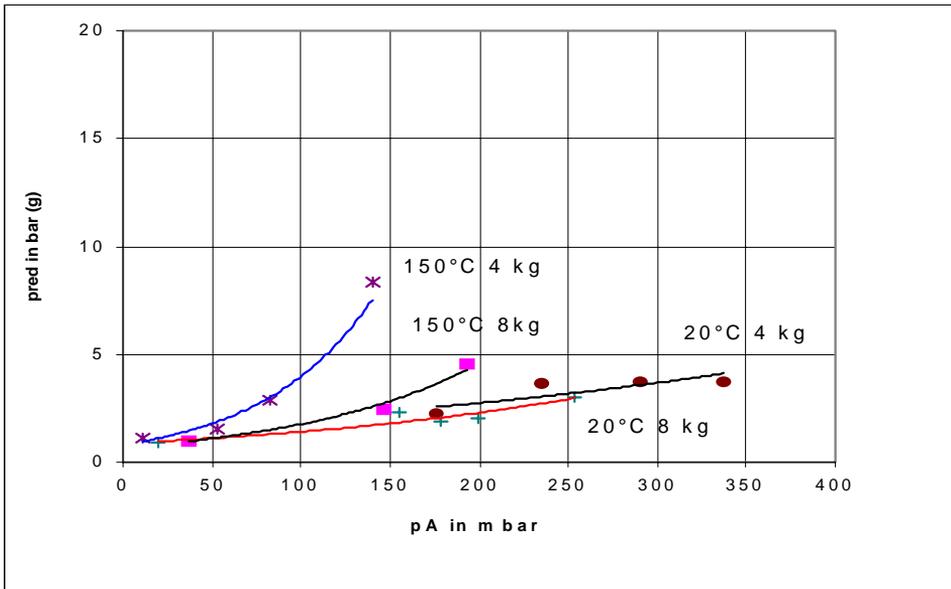


Figure 50. Explosion suppression tests with $2\,000 \text{ g/m}^3$ German lignite at 4 bar and $20^\circ\text{C}/150^\circ\text{C}$, 4 kg/8 kg monoammonium phosphate.

Increasing the temperature to 150°C a reduced explosion overpressure below 4 bar(g) was achieved only with 8 kg of suppressant and a static activation overpressure lower than 10 mbar. With a static activation overpressure of 40 mbar the reduced explosion overpressure was higher than 16 bar(g).

The rate of pressure rise was determined at the time of activation. For a rate of pressure rise lower than 5 bar/s respectively 2 bar/s the reduced explosion overpressure was below 4 bar(g) with 8 kg monoammonium phosphate at 20°C respectively 150°C.

At an initial pressure of 4 bar and a static activation overpressure of 100 mbar the reduced explosion overpressure for German lignite was lower than 4 bar(g) with 4 kg and 8 kg monoammonium phosphate at normal temperature and at 150 °C. For dust explosions with a rate of pressure rise lower than 10 bar/s the reduced explosion pressures were lower than 4 bar(g).

During the explosion suppression tests in air the highest reduced explosion pressures were observed for wood dust at 4 bar and 150°C. Therefore the tests in reduced oxygen concentration were performed with this fuel at the pressure and temperature conditions mentioned. The results are presented in Figure 51. With a reduction of the oxygen concentration to 17 vol% the reduced explosion overpressure was lower than 3 bar(g) at an activation overpressure of 100 mbar. The reduction of the oxygen concentration by only 4 percentage units led to an increased efficiency of the explosion suppression system compared to normal conditions.

Dust explosion suppression at elevated conditions should be feasible in the above described circumstances at least for German lignite. Suppression of wood dust explosion appears more demanding, requiring lower activation pressures and probably larger amounts of suppressant. Elevated temperature, typically experienced in drying processes, hampers the use of suppression system significantly. Rapid cooling of the heat energy released during the early stage of explosion is of great importance, and an high initial temperature seems to affect the efficiency of the suppressant. In these conditions a combined protection system appears feasible. Mild inertisation of the surrounding atmosphere in combination with suppression may be a solution for dust explosions at elevated conditions.

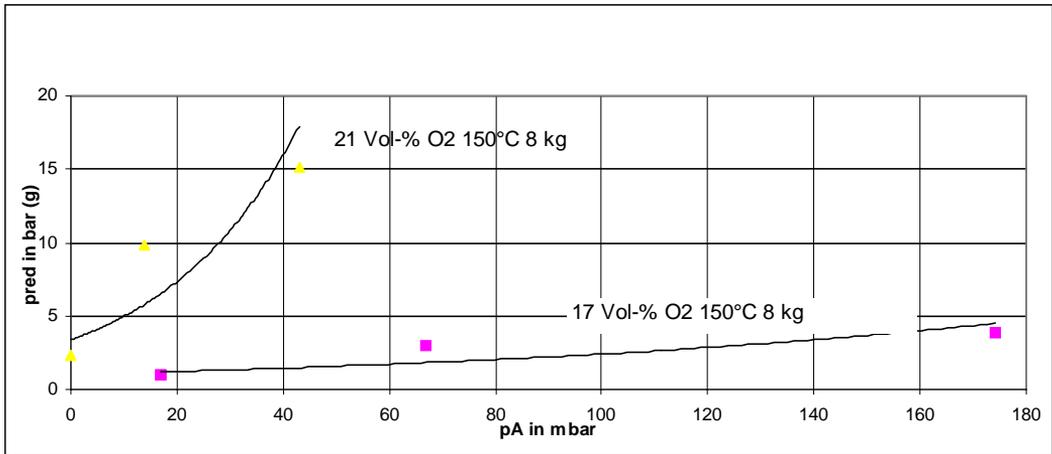


Figure 51. Explosion suppression tests with 2000 g/m^3 wood dust in air and 17 vol% O_2 at 4 bar and 150°C , 8 kg monoammonium phosphate.

11. Conclusions and recommendations

General conclusions and recommendation of the research work are summarised below. A more detailed elaboration of results are presented in this report at the end of each chapter.

Bulk density and particle size distribution are the physical properties of fuels most frequently needed for design of feeding and handling equipment. Bulk density is particle size dependent and should therefore be determined for a known particle size distribution. A compacted density value should be used for silos and intermediate storage bins while normal bulk density measurements are usually more adequate for the design of screws and conveying equipment. Generally low bulk densities were measured for the biomass samples involved in this study, and the compactibility of the biomasses, and the biomass/lignite mixtures, was quite significant compared to that of pure lignites.

The minimum ignition temperature of a biomass layer is between 300 and 340 °C and a little lower, 230 - 40 °C, for lignites. Corresponding ignition temperature of a dust cloud is higher, 400 - 460 °C for both biomasses and lignites. These ignition temperatures should not be confused with the self-ignition temperature of these fuels, which are considerably lower.

Thermogravimetric analysis was used to study the reactivity of the selected fuel samples. Classification of the dust samples indicates that the low-grade coals are clearly more reactive than the biomass samples. The mixtures also show a higher reactivity than the pure biomass fuels. Increasing the pressure leads to a higher oxygen partial pressure, increasing the aggressiveness of the oxidation. This results in a higher reactivity of the sample at elevated pressures. Thermoanalysis appeared to be a fast and convenient means of classifying fuels according to their reactivity. Much knowledge of fundamental fuel properties can also be extracted from these measurements. To obtain more quantitative information regarding the self-ignition behaviour of fuels by means of thermogravimetric analysis require extensive experimental work and development of the analysing methods. In addition, thermoanalytical data provides information for modelling self-heating, which could be the topic of a continuation work.

A statistical treatment of the results was used to create a predictive criterion based on simple tests and analyses, aimed at the prediction of the theoretical reactivity of a determined fuel as a function of its chemical composition. The statistical treatment of the developed data base resulted in four significant multiple regressions with a confidence level above 95 %. To have a more comprehensive assessment and a more reliable result, a “standard” definition for the reactivity would be needed and testing of considerably more fuel samples.

Low-temperature reactions of organic substances with atmospheric oxygen lead to self-heating. It is well-known that the self-ignition temperature of a dust deposit is a function of the size of the deposit; i.e., the self-ignition temperature falls when the size of the deposit (or storage volume) increases. Self-ignition can occur even in relatively thin dust layers at temperatures well below the ignition temperature on a hot surface. This must be observed for instance when biomass fuels are dried with hot air in low temperature bed dryers. Extended drying time and failing in removing all dust layers from the dryer might cause unexpected fires inside the dryer. These may consequent in violent dust explosions.

Self-ignition properties of the fuels were studied at normal and elevated pressure (1 - 25 bar). The results of the self-ignition tests for the fuel samples are mainly in line with the reactivity tests carried out by thermoanalysis. The lignites are the most reactive fuels both at ambient and elevated pressure. The pure wood fuels, wood and Spanish pine, were least reactive with regard to spontaneous ignition. The wood wastes, bark and forest residue are more reactive than the agricultural straw residues. The different straws showed very similar self-ignition temperatures. The mixtures of lignites and biomasses were in reactivity comparable to the most reactive pure lignites.

The elevated pressure had a significant effect on the self-ignition temperature of all fuel samples: the self-ignition temperature of the fuel fell when the pressure was elevated. Partial inerting, i.e., decreasing the oxygen concentration of the ambient air atmosphere, increased the self-ignition temperature, but to a rather low degree. To establish significantly safer conditions in fuel storage, an inert atmosphere with an oxygen content well below 7% is obviously required.

The explosion tests are usually performed in ambient conditions in a 1 m³ pressure vessel according to ISO 6184/1 standard procedure, or in a smaller 20 litre

laboratory sphere. At high initial pressure and for “difficult” dust it is necessary to define and develop new explosion test procedures. The work done within this project is a first step to study the influence of dust injection methods on the severity of the explosion behaviour of biomass fuels. A multifactorial experimental design was used to optimise the experimental work. As a conclusion two different experimental approaches were adopted for the high pressure test programme. In the 20-litre sphere the dust was dispersed with a specially design nozzle when it was already on the bottom of the sphere. Regarding the 1 m³ vessel the more conventional method of introducing the dust from an external dust container with a rebound nozzle was chosen.

Dust explosion tests were performed both in normal conditions and at elevated temperature and pressure. A linear correlation was found between the maximum explosion pressure and the initial pressure. The maximum explosion pressure is directly proportional to the initial pressure. The results also show a fairly good conformity considering the different tests methods. Further, the explosion pressures of all fuels fall in the same order of magnitude.

An approximately linear relation also exists for the the rate of pressure rise, the K_{St} value. There is, however, a large discrepancy in the slope of the lines for the same fuel samples. Most differences in the measured K_{St} -values can probably be associated with diverse turbulence conditions inside the explosion vessel. Various turbulence levels may be ascribed to different equipment and methods, and generally to the inhomogeneous nature of the fuel samples. The influence of turbulence on the K_{St} -value and more generally the violence of the explosion phenomenon should be further investigated, especially with regard to standardisation work.

In most cases, the lower oxygen concentration LOC slightly increased with increasing initial pressure. The tendency is clear for wood but for the lignites and the mixture the effect of initial pressure is more irregular. The results suggest that for most cases safe conditions can be obtained by decreasing the oxygen content of the surrounding atmosphere to a level of approximately 11 - 15 vol%. In practice a safety margin of 2 percentage units is usually recommended. LOC decreases, however, with increasing initial temperature. For all powders tested this decrease was more or less the same, around 1 - 3 vol% per 100 °C temperature rise.

The knowledge of the explosions parameters at high initial pressure and temperature is fundamental for the design of safety measures at IGCC and PFBC power plants. The dust explosion indices are essential for the design of protective measures like relief venting, inerting and explosion suppression systems. Generally very little information exists in the literature regarding explosion properties of biomass fuels and especially of explosion indices measured at elevated conditions. Therefore the knowledge created within this project can be considered unique. Further experimental work is, however, recommended and required to complement the current database and to develop a broader basis for the future standardisation work on explosion test methodology at elevated pressure and temperature. It would also be useful to create a theoretical model for comparing experimental data against predictions.

On the basis of the very high explosion pressure and rate of pressure rise measured at elevated initial pressures, it is quite obvious, that designing feeding bins for total explosion containment is not feasible. Neither can relief venting be used due to the large required vent area and high reduced pressure. Inerting offers a safe alternative, but involves usually high investment and operation costs. Explosion suppression systems are frequently used in industry as a measure of explosion protection. The technology is, however, not yet commercial for pressurised systems.

Explosion suppression tests were conducted in a heatable 1 m³ vessel. The reduced explosion overpressure P_{red} was determined in respect of the activation pressure P_A at 4 bar initial pressure and temperatures of 20 °C and 150 °C. Wood and German lignite were used as dust samples. Increasing the temperature makes the suppression of the dust explosion more demanding (low activation pressure). Use of partly inert atmosphere, e.g. by reducing the oxygen concentration from 21% to 17%, led to an increased efficiency of the explosion suppression system.

The development of the explosion suppression system for high pressure applications requires still extensive experimental work. A combination of mild inert conditions with explosion suppression appears to be a feasible alternative at elevated pressures.

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Appendix A

Scanning electron microscope photos of the dust samples

Magnification 71,5x

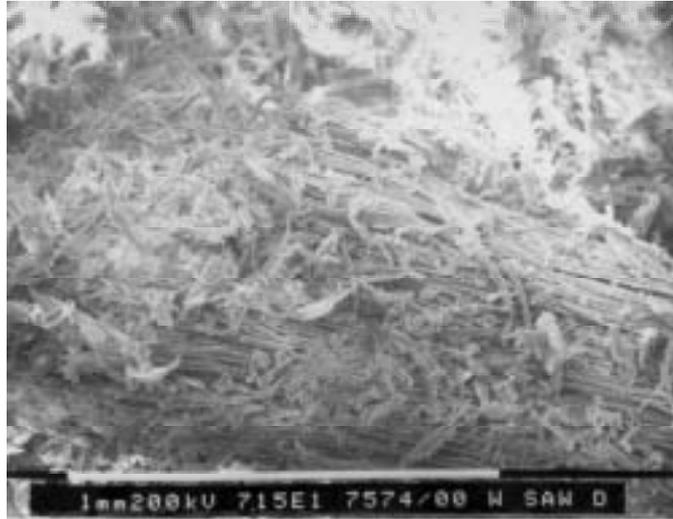


Figure 1. Wood dust.

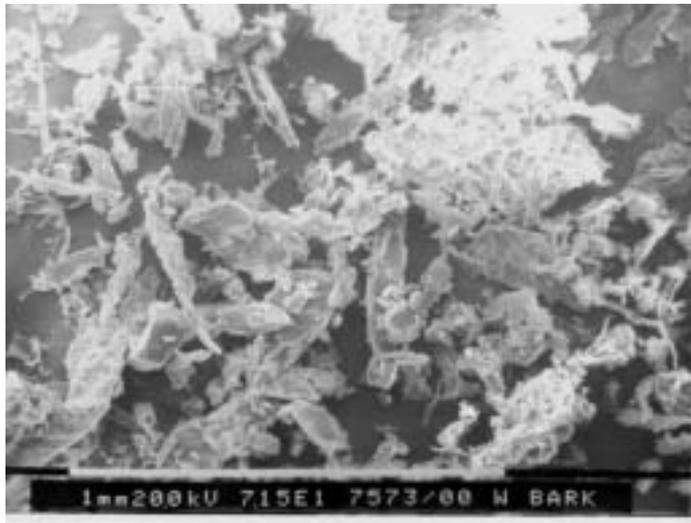


Figure 2. Bark dust.



Figure 3. Forest residue dust.

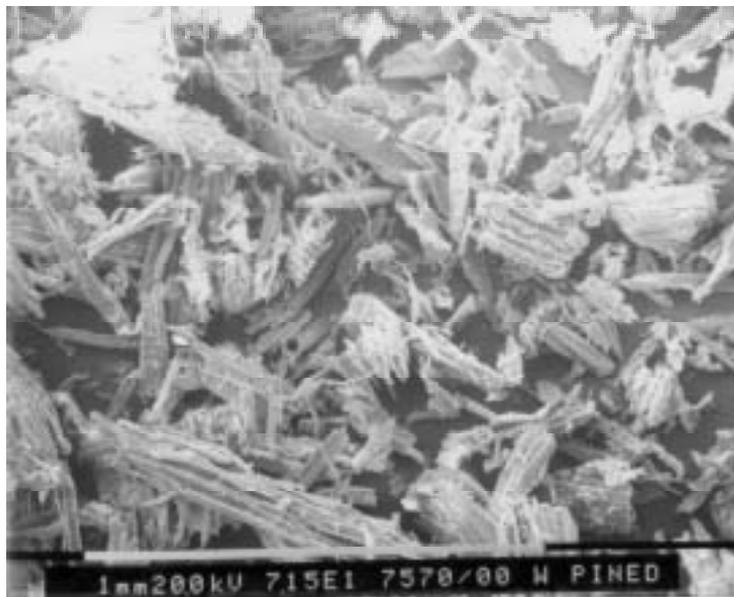


Figure 4. Spanish pine dust.



Figure 5. Barley straw dust.

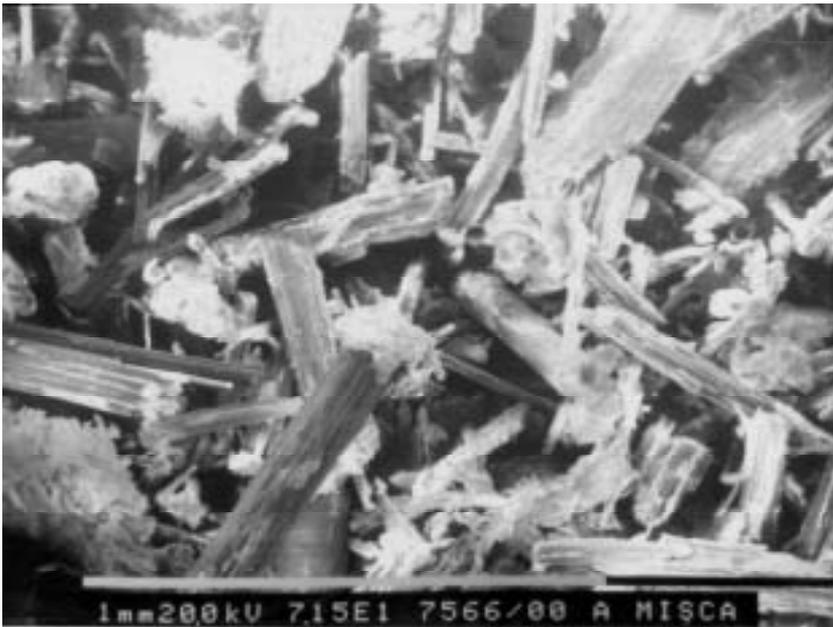


Figure 6. Miscanthus dust.



Figure 7. Sorghum dust.



Figure 8. Rapeseed straw dust.

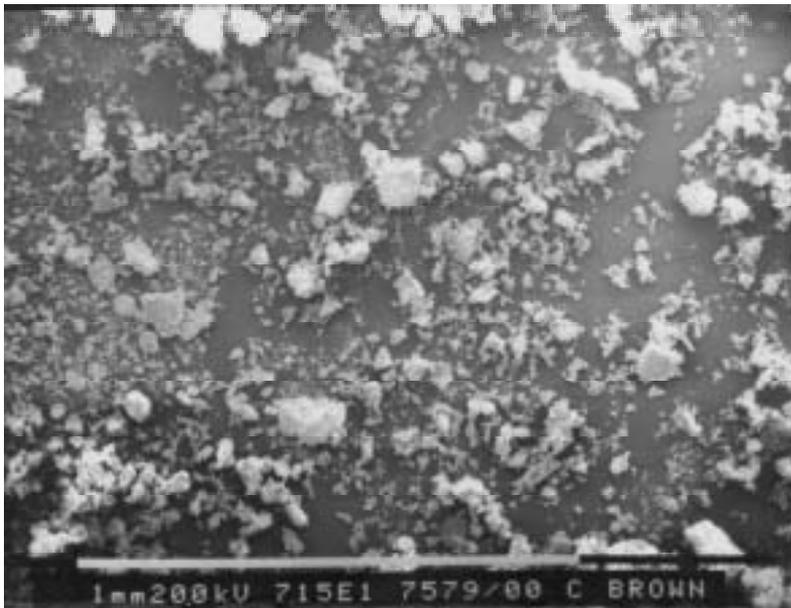


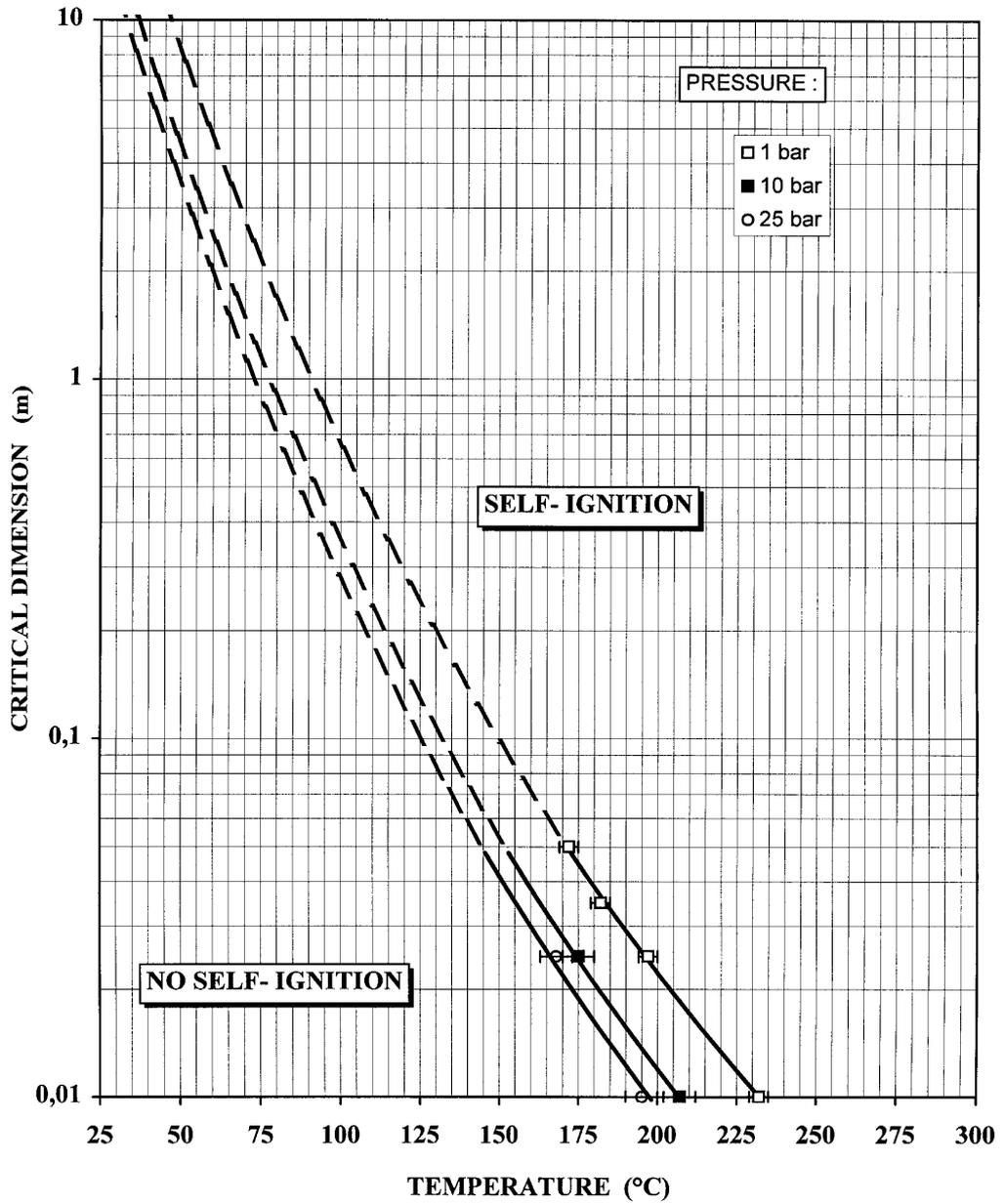
Figure 9. German lignite dust.



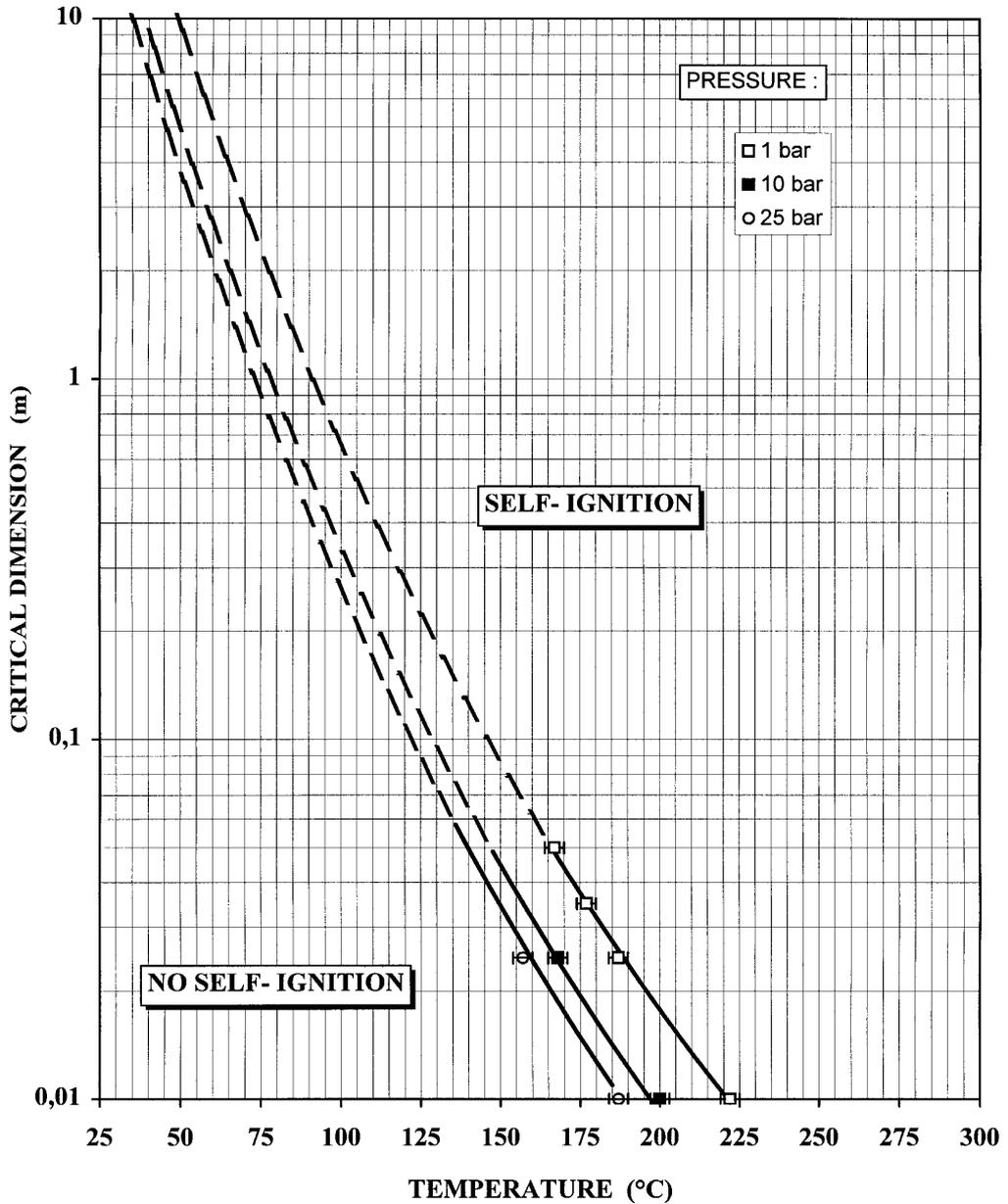
Figure 10. Spanish lignite dust.

Appendix B

EXTRAPOLATION OF THE CRITICAL DIMENSION AS A FUNCTION OF TEMPERATURE WOOD



EXTRAPOLATION OF THE CRITICAL DIMENSION AS A FUNCTION OF TEMPERATURE BARLEY STRAW



EXTRAPOLATION OF THE CRITICAL DIMENSION AS A FUNCTION OF TEMPERATURE GERMAN LIGNITE

