The Development of Smouldering Combustion in Combustible Building Insulation Materials

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Abstract:
This thesis is a study of smouldering combustion in combustible building insulation materials. The purpose was to find the necessary conditions for combustible building insulation materials to undergo a self-sustained smouldering combustion.

The main goal of the thesis was to find the temperature exposure needed to initiate a self-sustained smouldering combustion. If smouldering was obtained, the differences and similarities in the combustion of the different test materials would be studied, and connected to the chemical and physical properties of the materials.

The report is based on experimental testing, and a literature study is done in order to be used as a foundation for the discussion of the test results. Four different types of wood fibre insulation was tested. The samples was placed in an insulated steel chimney above a hotplate, and heated from underneath. The temperature development and mass loss was registered during the testing.

Two different types of smouldering behaviours were observed in the test samples. The distinction was made based on the registered mass losses and maximum temperatures. The one with highest maximum temperature and mass loss was found to be typical for materials undergoing a secondary oxidation of the char. The temperatures needed from external heating to obtain a self-sustained smouldering behaviour in the materials are: 275 °C for material T, 280 °C for material U, 225 °C for material V and 290 °C for material W. This indicates that an onset of a self-sustained smouldering combustion occurs at lower temperature in insulation material with smaller fibre size than in a material with larger fibre size, when the materials have the same added level of fire retardant. A higher level of added fire retardant gave an onset of a smouldering combustion at lower temperatures, when the fibre size of the material was the same.

The testing and results in this thesis is only indicative. Future work should include more tests with each material, but narrow down the factors to be studied. The existing test results can also be analysed with other methods than the ones used in this thesis, to look for more information.

Keywords:
1. Smouldering Combustion
2. Building Insulation Materials
3. Wood Fibre Insulation
4. Reaction to Fire
Acknowledgements

This master thesis marks the ending of my education at the Norwegian University of Science and Technology (NTNU). The thesis was carried out in the period from August 2015 to January 2016 at the Department of Civil and Transport Engineering (BAT), and covers 30 credits.

For the last 13 years, I have had a fascination for fire and fire safety, which has led me to the education I am now finishing. I am not the kind of person who likes to sit still in front of a computer for a long time, so when I got the opportunity to do a master thesis with testing in the laboratory of SP Fire Research AS (SPFR), I was very happy.

Working with this thesis has been challenging, but at the same time, very educative. I have learned a lot about the planning and execution of a project with experimental testing, from the very beginning to the end.

I want to thank my supervisor Harald Landrø at NTNU and supervisor Anne Steen-Hansen at SPFR, for the opportunity to do this thesis and for the guidance throughout the project. I would also thank Ragni Fjellgaard Mikalsen at SPFR. You have given me valuable guidance in the laboratory and feedback on my work.

Finally, I would like to give special thanks to the technicians at SPFR for practical help and keeping my motivation up and making me laugh, even at tough times.

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Ulla Eidissen Jensen
Abstract

Energy wastage due to little or bad thermal insulation is a problem. An increase in the quality of the building envelope with more and better insulation is therefore an initiative to reduce the emission of greenhouse gases.

Smouldering fire is a hazard in combustible insulation. A material can decompose and undergo a smouldering combustion at lower temperatures than needed to initiate a flaming fire, and cellulose insulation in combination with inadequate separation from electrical appliances and heat-producing equipment has led to smouldering combustions and building fires.

The purpose of this thesis was to find the conditions necessary for combustible building insulation materials to undergo a self-sustained smouldering combustion. The main goal was to find the temperature exposure needed to initiate a self-sustained smouldering combustion. If smouldering was obtained, the differences and similarities in the combustion of the different test materials was studied and connected to the chemical and physical properties of the materials. The thesis is based on experimental tests. A literature study was used as a foundation for the discussion of test results. Four different types wood fibre insulation was tested. The samples were placed in an insulated steel chimney above a hotplate, and heated from underneath. Temperature development and mass loss was registered during the testing.

The results show that one of the four test materials obtained a smouldering combustion at temperatures much lower than the rest. Two different types of smouldering behaviours were observed in the test samples. The distinction was made based on the registered mass losses and maximum temperatures. The one with highest maximum temperature and mass loss was found to be typical for materials undergoing a secondary oxidation of the char. The temperatures needed from external heating to obtain a self-sustained smouldering behaviour in the tested materials are: 275 °C for material T, 280 °C for material U, 225 °C for material V and 290 °C for material W. This indicated that an onset of a self-sustained smouldering combustion occurs at lower temperature in insulation material with smaller fibre size than in a material with larger fibre size, when the materials have the same added level of fire retardant. A higher level of added fire retardant gave an onset of a smouldering combustion at lower temperatures, when the fibre size of the material is the same.

The testing and results in this thesis is only indicative. Future work should include more tests with each material, but narrow down the factors to be studied. The existing test results can also be analysed with other methods than the ones used in this thesis, to look for more information.
Sammendrag

Energitap grunnet lite eller dårlig isolasjon er et problem i bygninger. En forbedring av bygningsskallets kvalitet med mer og bedre isolasjon kan derfor bidra til en reduksjon av klimagassutslipp.

Ulmebrann er en fare i brennbar bygningsisolasjon. Et material kan brytes ned og gjennomgå en ulmebrann ved mye lavere temperaturer enn det som er nødvendig for initiering av en flammebrann. Det har vært hendelser hvor celluloseisolasjon i tilknytning til utilstrekkelig elektrisk anlegg og varmeavgivende komponenter har ført til ulming og bygningsbrann.


Resultatene viser at ett av de de fire testmaterialene ulmet ved mye lavere temperatur enn de andre tre. Det ble observert to ulike typer ulmebrann, basert på målinger av massetap og makstemperaturer. Den med høyest massetap og temperatur viste seg å være typisk for materialer som gjennomgår en sekundær oksidering av det forkullete materialet. Nødvendige temperaturer for å få en selvgående ulmebrann i testmaterialene var: 275 °C for materiale T, 280 °C for materiale U, 225 °C for materiael V and 290 °C for materiale W. Dette viser at ulming ble oppnådd ved lavere temperatur i isolasjonsmateriale med mindre fiberstørrelse enn i material med større fiberstørrelse, gitt at de har samme mengde brannhemmer tilsatt. En større mengde brannhemmer i et materiale gav ulming ved lavere temperatur hvis to materialer med lik fiberstørrelse testes.

Testingen og resultatene i denne oppgaven er indikative. Forslag til videre arbeid er å gjøre flere tester, men å snevre inn hvilke faktorer som undersøkes. Mer avansert analyse av eksisterende testresultater kan også være en kilde til mer informasjon.
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<th>Abbreviation</th>
<th>Symbol</th>
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<td>Department of Civil and Transport Engineering</td>
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<td>°C</td>
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1 Introduction

This master thesis is carried out in connection with the Emerging Risks from Smoldering Fires (EMRIS)-project. EMRIS seeks to improve the knowledge about smouldering fire, and of the properties of materials prone to smouldering fire. In this thesis the conditions necessary to obtain a smouldering combustion in combustible building insulation materials will be studied and discussed.

1.1 Background

In 2009 the United Nations Environment Programme published a report (1) based on research done by the Sustainable Buildings & Climate Initiative and partners. Energy performance standards, skills-base and capacity to assess energy performance and implement energy efficiency policies is in the report said to be a part of the work to achieve a reduction in the emission of greenhouse gases from buildings, both new buildings and existing. Over 80 percent of the greenhouse gas emissions from a building is during its operational phase. One of the reports five major policy targets in order to reduce emissions from buildings is to increase the energy efficiency of buildings. This includes both the energy efficiency of the appliances and technical installations of the building, and the standard of the building envelope. A better building envelope decreases the energy needed to heat or cool the building. Energy wastage due to little or bad insulation and air leakages is a problem in existing buildings. Insulation and retrofitting of existing buildings is therefore an action in the work of increasing the energy efficiency of the building mass. (1)

The use of building materials with low energy consumption during the production phase, will contribute to keeping the greenhouse gas emissions low during the building time. Natural Resources Defence Council have compared three different insulation materials, where they found that it takes 10 times more energy to produce fiberglass insulation, than to produce cellulose insulation (2). Wood fibre- and cellulose insulation have another advantage as well: In addition to their energy efficiency, the materials have the advantage of being hygroscopic, which means that they contribute to controlling the moist balance in the building (3).

1.1.1 The hazard of combustible building insulation materials

Wood fibre- and cellulose insulation has a disadvantage that has to be addressed. Wood fibre and cellulose are combustible materials. When produced, fire retardant agents are added to the insulation in order to make the insulation fire safe. Still, there have been many cases of building
fires in retrofitted buildings with cellulose insulation. These fires have typically been smouldering combustions in connection with inadequate separation from electrical appliances and heat-producing equipment. (4–6).

Smouldering is a problem in combustible insulation because a material can start to decompose and undergo a smouldering combustion at lower temperatures than what is needed to initiate a flaming fire, and the smouldering fire can transfer to a flaming fire. Another reason for smouldering combustion to be a hazard is that it leads to a higher level of toxic compounds from the fuel than a flaming fire. (7)

1.2 Problem
The goal of this thesis is to find the conditions necessary for combustible building insulation materials to undergo a self-sustained smouldering combustion. The focus will in particular be on finding the temperature exposure needed to initiate a self-sustained smouldering combustion in the test materials. If smouldering is obtained in any of the test samples, the differences and similarities in the combustion of the different test materials will be studied and connected to the chemical and physical properties of the materials.

1.3 Hypotheses
Many factors can influence the initiation of smouldering combustion. There is formed four hypotheses about factors that may influence the smouldering. In this thesis, hypothesis 2, 3 and 4 will be assessed.

1. Higher bulk density in the test will give lower temperature for onset smouldering, due to a higher heat conductivity.
2. A self-sustained smouldering fire will occur at lower temperature in the insulation material with smaller fibre size, than in material with larger fibre size, due to a larger surface/volume-ratio.
3. A higher level of added fire retardant chemicals in the insulation material will lead to the insulation material being more fire resistant, and a higher temperature exposure will then be necessary to obtain a self-sustained smouldering fire.
4. The material will undergo combustion faster with smaller fibre-size, due to a larger surface/volume-ratio.
1.4 Structure of the report

This report is based on experimental testing. A literature study is done as a part of the report, in order to give background information about smouldering combustion and other research results on smouldering in cellulosic materials. In section 2, the methods used will be explained. Section 3 is the theoretical background found by the literature study, while sections 4-0 is the experimental method, results, discussion and conclusion. In section 0, suggestions to further work will be presented.

1.5 Readers guidance on nomenclature

When «cellulose insulation» is denoted from now on and out in the report, it means insulation made from recycled paper, that is, processed wood. The other type of combustible insulation in this report is made directly from wood and will be denoted as «wood fibre insulation». Personal communication with a representative from a European cellulose insulation organisation made it clear that the mentioned nomenclature is the one used in the industry today.
2 Method

This section describes the methods used in this master thesis, as well as why and how the methods are used. The thesis is based on experimental work with material tests. The test results are assessed and discussed in comparison to theory found in a literature study.

Smouldering combustion is a complex field of work where the knowledge and understanding is limited (7). Both the literature study and the testing are done in qualitative manners, in order to increase the knowledge about the phenomena of smouldering combustion, and how it affects combustible building insulation materials.

2.1 Literature study

A literature study was conducted in order to find information about smouldering in combustible building insulation material. The theory found will be used as a base for the discussion of the test results.

It was chosen to search for information about wood fibre- and cellulose insulation. Other research and studies done on smouldering combustion and on cellulosic materials were also studied to get a general impression on the process of smouldering combustion in cellulosic materials.

When looking for literature, multiple search engines and databases have been used with the same keywords, such as cellulose, insulation, smouldering, combustion, fire and building insulation. This was done because different databases do not include the same library of literature. Basing a literature study on the results from only one database could therefore leave out useful information. The keywords were used in different combinations as well. For keywords that have different spelling in American English and British English, searches were done with both American and British spelling, e.g. both smoldering and smouldering were used as search keywords. The databases and search engines used was Oria, Scopus, Science Direct, Google Scholar and Google. Articles and rapports without online access were ordered through NTNU University Library.

After doing a thorough search for literature, the results showed that there were not much research done on smouldering fire in wood fibre and cellulose insulation, and the literature found was up to 60 years old. It must be considered that comparing test results of new materials with older literature can be difficult. This relies on whether there have been much development
in the building material industry, and how similar the products tested today is with the ones tested earlier.

2.2 Testing

Four different types of wood fibre insulation were tested in order to find the temperature exposure necessary for the materials to undergo a self-sustained smouldering combustion, and then to study how the differences and similarities in the combustion of the different test materials can be connected to the chemical and physical properties of the materials. The experimental work will be described in detail in section 4.

The test method used in this thesis, is the same that is in use in the EMRIS-project. This makes it easier to compare the data from the material tests run in this master thesis, with data from material testing done in the EMRIS-project. The test method is not a standard test method, which means that it may be difficult to compare the results directly to results from other test methods.
3 Theoretical Background

In this part, what smouldering combustion is and how it affects combustible building insulation materials will be looked into.

3.1 Smouldering

Smouldering is a type of combustion. When a material is exposed to sufficient temperature rise, it will start to decompose. This reaction, which is an endotherm reaction, is known as pyrolysis. Sufficient temperature to obtain pyrolysis differs, depending on the material exposed to heat. As long as the material is exposed to an external heating source with a sufficient temperature level, there will be a forced pyrolysis, and this can lead to ignition of the material and a combustion of the decomposed material. (7,8)

Smouldering must not be confused with flaming combustion. When a material is heated, the pyrolysis leave products in both the form of gases and as solids. These products can undergo oxidation, which is the combustion process. In a flaming combustion, the gases from the pyrolysis are the fuel for the combustion. In a smouldering combustion, the fuel is the solid char from the pyrolysis. Oxidation of the char is what gives the heat generation in a smouldering combustion, so instead of a combustion of volatiles, like with a flaming combustion, the smouldering combustion is a solid state combustion. (8,9) The oxidation is an exothermic reaction. If the oxidation releases more heat than what is lost from the pyrolysis to the surroundings, there is a self-sustained smouldering propagation. (7,8)

The temperature difference between a smouldering and a flaming combustion is noticeable. Maximum temperature in a smouldering combustion is typically found in the area around 500-700 °C (8,10–12), while a flaming combustion has values around 1500-1800 °C (11).

Smouldering is a slow combustion. It can propagate with a velocity as low as 0.01 mm/s in wood fibres (12). The rate of the smouldering propagation is controlled by the oxygen transport to, and the heat loss from the area where the oxidation is happening (7,11).

In a one dimensional system, which is a simplified situation, smouldering can be divide into forward smoulder and reverse smoulder. Forward smoulder is when the smouldering propagates in the same direction as the air flow, while reverse smouldering is when the smouldering propagates in the opposite direction of the air flow. (7)

The fuel of a smouldering combustion is typically porous materials with the ability to form char, and combustion is sustained by the heat of the oxidized fuel. A porous structure give
A material undergoing a smouldering combustion can typically be divided in three zones (10) as shown in Figure 1. This is a simplified example of forward smouldering, and the three zones, starting from the virgin cellulose, are:

- A pyrolysis zone where there is a fast temperature rise, an outflow of visible gases and discoloration of cellulose, denoted as «Discoloration of cellulose» in the figure.
- A charred zone where the temperature reaches a maximum, the evolution of visible gases stops and glowing occurs, denoted as «Black char», «Glowing char» and «Maximum temperature» in the figure.
- A zone of very porous residual char and/or ash, which is cooling of slowly and no longer glowing, denoted as «Residual ash/char» in the figure.

Figure 1: Simplified representation of a forward smouldering combustion. (8,10) The text in the figure is enhanced for readability.
Before the pyrolysis zone described above, there is the zone where the water is driven out of the material by condensation and evaporation. In reverse smouldering the pyrolysis and oxidation zones overlap, while the zones are distinct in the forward smouldering. (12)

It is not easy to identify a smouldering ignition, because it cannot be seen with the naked eye, like the ignition of a flaming fire. Krause et al. (13) used three different indications in their experiments to decide if the material ignited or not:

- A significant rise of temperature above the oven temperature
- A significant loss of mass
- A change of colour in the material

In addition to the oxidation of the decomposed material, there is a possibility for secondary oxidation of the char residue left by the first reaction. This secondary oxidation is an intense high-temperature reaction. (7,14) Temperature of secondary char oxidation in polyurethane is ~ 700 °C (7). The char residue can undergo this secondary oxidation when it receives a fresh supply of air (15). Transition to flaming will not be addressed in this thesis, but the secondary char oxidation is an important factor in the transition to flaming, because it provides the fuel, heat and sites (pores left by the oxidation) that is necessary for gas-phase ignition (15).

### 3.2 Wood fibre- and cellulose insulation

Natural cellulosic materials such as wood, and other products based on wood fibre, are composed of cellulose, hemicellulose and lignin (9,16). The proportions will vary with wood type. For most wood types there is ~40 % cellulose, ~25 % hemicellulose and 20-30 % lignin (17). The different components of wood decomposes at different temperatures. Hemicellulose at 200- 260 °C, cellulose at 240- 350 °C and lignin at 280- 500 °C (16).

#### 3.2.1 Thermal decomposition of wood

Moist in the wood is driven out of the wood when the wood is heated to 100 °C. The layer of the wood that has a temperature of 200-300 °C is the pyrolysis zone. Between the pyrolysis zone and the charred part there is a distinctive border, and this lies at approximately 300 °C. (8) The different zones are showed in Figure 2.
3.2.2 Building insulation material properties

Wood fibre- and cellulose insulation are hygroscopic materials. Insulation products based on wood fibre or cellulose have to be treated with mineral salts to make them resist mould, rot, bacteria and fungus, as well as fire. (3)

Properties of the insulation will vary with type of insulation. An example of this variation is the heat conductivity and heat capacity of two common types of insulation. Wood fibre- and cellulose insulation has a heat conductivity as low as 0.041 W/(mK). This is slightly higher than mineral wool which has 0.034 W/(mK). The heat capacity of cellulose insulation is 1600 J/(kgK), which is remarkable higher than the heat capacity of mineral wool which is 1030 J/(kgK). (3)

3.2.3 The use of wood fibre- and cellulose insulation

Wood fibre- and cellulose insulation can be made both in the shape of boards and as loose-fill insulation. Loose-fill insulation is delivered to the building site compressed in bags. At the building site, the insulation is filled into machines where it is loosened up from the compressed state. Then it is blown through hoses and into the building structure with the use of pressurized air. (3) The amount of insulation blown into a structure depends on the type of building structure and the recommendations from the insulation producer. Recommended density for loose-fill insulation in use were from personal communication with a supplier found to be approximately
32 kg/m³ when installing loose-fill insulation in an open attic floor, and 32-38 kg/m³ when installing loose-fill insulation in decks and other enclosed frame works.

3.3 Fire retardants

Different additives can be used as fire suppressors. In order to find the most used fire retardants in wood fibre- and cellulose insulation in Norway, Europe and the world today, searches in different channels were done. A rather new book about fire retardancy (20) was studied for fire retardants used in cellulosic materials. The list of retardants in the book was complemented with fire retardants found in the literature study about wood fibre- and cellulose insulation (4,9,21). Personal communication with a representative from a European cellulose insulation organisation and a researcher in a Norwegian fibre research institution was then used to shorten the list of additives down.

The most common fire retardants in wood fibre- and cellulose insulation today are:

- Ammonium phosphates
- Boric acid + Magnesium sulphate
- Boric acid + Aluminium hydrate
- Boric acid + Borax
- Ammonia
- Magnesium hydroxide

According to the representative from a European cellulose insulation organisation, the most widespread fire retardant in cellulose insulation in Europe is Boric acid in combination with Magnesium sulphate, while Boric acid in combination with Borax is the most common in the United States. The researcher in Norway mentioned Ammonium phosphates, Boric acid + Borax and Magnesium hydroxide as much used fire retardants.

The representative from the European cellulose insulation organisation also informed that the use of fire retardants in production of cellulose insulation today, is mainly to improve the fire safety in the factories during the production.
3.4 Smouldering fire in wood fibre- and cellulose insulation materials

There have not been found much literature about smouldering combustion in wood fibre- and cellulose insulation, so it is supplemented with literature on combustion in other cellulosic materials. This section is divided into three parts. The first part is about how air flow and geometrical factors influence the smouldering, the second is about the influence of fire retardants and the last is about degradation of the fire safety of fire retarded combustible insulation. Transition to flaming will not be covered in the testing done in this project, but literature where transition to flaming is mentioned in connection with factors that will be covered, will still be included. This is done because transition to flaming is one of the hazards of smouldering combustion.

3.4.1 Influence of air flow and geometrical factors

As mentioned in section 3.1, there are differences in the processes of forward and reverse smouldering. Ohlemiller tested cellulose insulation to study forced flow smouldering propagation and the transition to flaming combustion. The study showed big difference in how the smoulder velocity depended on change of air flow in forward and reverse smouldering. In the case of forward smouldering, an increased air flow leads to a considerable acceleration in the smouldering. For reverse smouldering, there was only registered a weak increase in smouldering as the air flow increased. According to Ohlemiller, the kinetic limitations of the smoulder front makes it sensitive to the differences in convective heat transfer implicit in forward and reverse configuration. Ohlemiller concluded by this that both the oxygen transport and the kinetic limitations of the smouldering participate in the determination of the velocity of the smouldering, but in different parts of the smoulder region. (22)

Already in 1956, Palmer did experiments to study smouldering combustion in dusts and fibrous materials. The dusts tested were of cellulosic materials. One of the shapes that the dusts were tested in was trains. This means that the dusts were moulded into the shape of a thick strip using moulds, in this case with cross sections that were segments of circles. A dependency between smouldering rate and airflow was registered. The smouldering rate was low in still air, and increased considerably by applied airflow. The experiments showed that the relation between air velocity and smoulder rate of dust trains was dependent upon particle size. This is illustrated in Figure 3. (23)
Figure 3: Variation of smouldering rate depending on air velocity and particle size. The particle size are sieve fraction according to British Standard, the train size dimensions are found in the table below. «f» indicates flaming. Particle type, particle size and dust train type are given in the label, which is added for clarity. (23)

Table 1: Dimensions of dust trains. The content of the table is from Palmer (23)

<table>
<thead>
<tr>
<th>Train</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>E</th>
<th>G</th>
<th>H</th>
<th>J</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base width [cm]</td>
<td>1.35</td>
<td>2.35</td>
<td>3.55</td>
<td>5.10</td>
<td>7.25</td>
<td>11.3</td>
<td>17.6</td>
<td>26.1</td>
</tr>
<tr>
<td>Vertical depth along centre [cm]</td>
<td>0.30</td>
<td>0.80</td>
<td>1.00</td>
<td>1.65</td>
<td>2.40</td>
<td>2.40</td>
<td>3.70</td>
<td>5.70</td>
</tr>
</tbody>
</table>

Figure 3 shows that even for an air velocity of zero, dust with smaller particle size that has slowest smouldering rate. The dust with largest particle size has the highest smouldering rate.

Palmer also saw that the smouldering combustion could turn into flaming if the dust was sufficiently coarse. The transition occurred at lower air velocities as the particle diameter was increased. (23)
Ohlemiller tested cellulose insulation with wood present to simulate a typical case of installed insulation. It was found that air flow affects transition to flaming, and that the critical air flow velocity for transition to flaming is depending on the fire retardant and wood present. Char tends to pull away from adjacent structures and form a cavity. This can lead to increased airflow to the char, and increased oxidation and heat directed towards the adjacent structure. The increased air flow lead to an acceleration of the smoulder and raised peak bed temperatures. Even though there was no transition to flaming in the insulation, the glowing char ignited the wood at the conditions showed in Figure 4. This happened at the same temperatures as the adjacent wood was ignited when testing both retarded and unretarded insulation, so with wood present, the flame suppression effect of Borax is lost. (22)

![Figure 4: Smouldering velocity depending on the air flow velocity for insulation with borax and boric acid. The insulation was tested with adjacent wood and it is marked which conditions gave flaming in the wood.(22)](image-url)
3.4.2 Influence of fire retardants

When looking into smouldering combustion of fire retarded combustible building insulation materials, the influence of fire retardants must be considered. An effective flame retardant will not necessarily prevent a smouldering combustion (9). Some additives used to improve the fire resistance of wood promote the char-forming process (8,9), while others prevent the oxidation (9).

Borax causes a minor increase in the acceleration of the char oxidation process for the fire retarded insulation, compared to unretarded insulation (22). Testing done by Day and Wiles (24) showed that the borax-boric acid formula that was recommended to give flame resistance to cellulose insulation was not sufficient to give smouldering resistance. They found that to satisfy both the flaming- and the smouldering criteria, it was needed a much higher part of boric acid than in the flame-retarding formula (24).

Kokkala ran a series of tests to evaluate the British Standard BS 5803: Part 4:1985, in order to propose a Nordtest method based on it. The materials tested were dry peat, sawdust, cellulose insulation with/without fire retardant, and glass wool. The propagation velocity was found to be 30% lower with retarded insulation than with unretarded insulation. (21)

3.4.3 Degradation of the fire resistance after installation

Wood fibre- and cellulose insulation are biological materials, and they will react to the environment they are placed in. In order for a building material product to be sustainable, it has to keep up performance after it is installed.

Dowds, Infante and Pentz tested the fire resistance of fiberglas, rockwool and cellulose insulation that had been installed for at least two years in attics. Of the 23 samples of cellulose insulation, 15 failed the criteria of the smouldering combustion test. This means that they have degraded after installation, because the criteria must have been fulfilled for the insulation materials to be sold. (25)

An American study of the flammability performance of cellulose insulation showed that a reduction in fire retardant “loading” in cellulose insulation over time occurs. Cellulose insulation that had been installed for twelve months or more may lose the ability to retard fire. (4)
4 Experimental

4.1 Materials

Four different types of loose-fill insulation materials were supplied by a producer of building materials. The insulation types are mechanical treated wood fibre, but they differ in wood type, grain size and additives.

All insulation types are anonymised, and will in this thesis be described by their test-series name, as shown in Table 2. The insulation arrived compressed in plastic bags, and the packages were stored in the laboratory at ambient conditions before testing. Density in package from producer was calculated from the mass and volume of the plastic bags with insulation, found by weighing the bags and measuring the size of the bags before opening them. The moisture content was measured by drying insulation samples at 105 °C and weighing them every 24th hour until the mass change was lower than 0.1 % of the total mass. The mass after the drying was regarded as dry mass, and the difference between the mass before drying and the dry mass was regarded as the moisture content. Information on insulation type, additives and wood type are stated as given by the producer.

Table 2: Material information for the four different test materials

<table>
<thead>
<tr>
<th>Insulation type description</th>
<th>T-type</th>
<th>U-type</th>
<th>V-type</th>
<th>W-type</th>
</tr>
</thead>
<tbody>
<tr>
<td>Insulation type description</td>
<td>Wood fibre – the material is fibrous and connected together in small clusters</td>
<td>Wood fibre – the material is fibrous with long, thin fibres.</td>
<td>Wood fibre – the same type as U-type, but finer mass. The fibres are shorter and finer.</td>
<td>Wood fibre – the same type as U-type, but finer mass. The fibres are shorter and finer.</td>
</tr>
<tr>
<td>Density in package from producer [kg/m³]</td>
<td>130.3</td>
<td>106.8</td>
<td>104.3</td>
<td>105.7</td>
</tr>
<tr>
<td>Test density [kg/m³]</td>
<td>34.3</td>
<td>34.3</td>
<td>34.3</td>
<td>34.3</td>
</tr>
<tr>
<td>Wood type</td>
<td>Fir</td>
<td>Spruce</td>
<td>Spruce</td>
<td>Spruce</td>
</tr>
<tr>
<td>Additives</td>
<td>8 % ammonium polyphosphate; 0.8 % boric acid</td>
<td>9 % ammonium polyphosphate</td>
<td>9 % ammonium polyphosphate</td>
<td>4 % ammonium polyphosphate</td>
</tr>
<tr>
<td>Measured moisture content [%]</td>
<td>11</td>
<td>12</td>
<td>11</td>
<td>9</td>
</tr>
</tbody>
</table>
Figure 5: Material T after mixing. The picture shows approximately 80 grams of the material.

Figure 6: Material U after mixing. The picture shows approximately 60 grams of the material.

Figure 7: Material V after mixing. The picture shows approximately 60 grams of the material.

Figure 8: Material W after mixing. The picture shows approximately 80 grams of the material.
4.2 Test equipment

4.2.1 Test setup

The test setup used is an insulated steel pipe on a hot plate. Test samples are placed in the insulated steel pipe and heated from underneath. Figure 9 illustrates the cross section of the test setup with a test sample inside the insulated steel pipe.

![Figure 9: Illustration of the test setup](image)

Figure 9 and Figure 10 show the different elements of the test setup. The bottom part is a scale, type Systec IT1000 with a display that shows the mass in grams. On top of the scale there is placed a hotplate, which in this case is a Wilfa cooking plate, type WCP1, with a power of 2000W. The hotplate is turned on/off manually. When the hotplate is on, it is controlled by a HENSEL thermostat.

A 28 mm thick aluminium plate, aluminium type 6082-T6, is placed on top of the cooking plate. It has a width and depth of 28 cm x 28 cm. This aluminium plate has milled channels on both top- and bottom side for placement of thermocouples, as shown in Figure 14. The thermostat controls the cooking plate based on temperature readings from the thermocouple (TC) that is placed in the milled channel on the bottom side of the aluminium plate. The thermostat will keep the heat on until the TC under the aluminium plate reaches a given set point temperature. Since the thermostat works on data from beneath the aluminium plate, the test sample will not necessarily reach the set point temperature for the thermostat. The thermostat has a hysteresis,
which is a value that the temperature has to change with from the set point temperature, before the thermostat switches the heat on/off.

On top of the aluminium plate, the insulated steel pipe is placed. It is a *Jøtul* steel pipe made of 1 mm stainless steel. The insulation is 60mm mineral wool with a density of 140 kg/m³. The steel pipe is 33 cm high, has an inner diameter of 15 cm, and is open at the top. The test setup is placed under an extractor hood as shown in Figure 11.

![Figure 10: The test setup consisting of, from the bottom, the scale, cooking plate, aluminium plate and the insulated steel pipe.](image1)

![Figure 11: Test setup placed under the extractor hood. On the table to the right the thermostat and the equipment for data logging is placed.](image2)

4.2.2 Thermocouples

All temperatures are logged using TCs type K with 0.5 mm encapsulation.

During the tests, there were eight TCs inside the steel pipe logging temperatures in the test sample, one TC underneath the aluminium plate logging the temperature in the aluminium plate, and one TC on top of the aluminium plate also logging the temperature in the aluminium plate, all as shown in Figure 9. The TCs underneath and on top of the aluminium plate are placed in milled channels in the plate which can be seen in Figure 14, so that they measure aluminium temperature, and not the temperature of the cooking plate underneath or the material sample above. The cross section illustration in Figure 9 shows how the two TCs are placed close to the surface of the bottom- and top side of the aluminium plate.
The TCs inside the steel pipe are mounted on a «ladder» made from steel, illustrated in Figure 12. The vertical parts of the «ladder» is made from stainless steel 316 band ½” x 0.4 mm. The horizontal steps is made from stainless steel band 1/8” x 0.2 mm. Having the TCs fixed to the “ladder” ensures that the TCs will stay in place during the test, and that they will have the same position in all the tests that are being run. The ladder is 15 cm wide, to fit in the middle of the steel pipe as seen in Figure 13. The TCs are mounted on steps that separate them vertically with 2 cm. The lowest step is 2 cm above the aluminium plate, and there are three TCs mounted on this step, left, centre and right, separated by 3.75 cm horizontally. The rest of the TCs inside the steel pipe are mounted centred on the ladder at 4 cm, 6 cm, 8 cm, 10 cm and 12 cm above the aluminium plate. This is illustrated both in Figure 9 and Figure 12.
Figure 12: The “thermocouple-ladder” with horizontal steps where the TCs are mounted. The steps are at every two centimetres.

Figure 13: The “thermocouple-ladder” as seen from above, when lowered into the steel pipe.

Figure 14: The aluminum plate with the milled channel topside for placement of thermocouple.

Figure 15: The thermostat and logging equipment: Thermostat, scale display, logger and laptop.
4.2.3 Data logging

The TCs were connected to a KEYSIGHT 34972A LXI logger. A laptop with the registered number NBL2874 and the software Agilent BenchLink Data Logger 3 was used to register the data at intervals of five seconds. This equipment is shown in Figure 11 and Figure 15.

The mass of the test samples was logged together with the temperature loggings. The values from the scale was logged in Volt(V), and experimentally determined scaling values was used in the software to get the logged mass in kilograms. 11 channels were used, connected to the TCs and scale as shown in Table 3.

Table 3: Placement of the measurement points per channel

<table>
<thead>
<tr>
<th>Channel</th>
<th>Placement/measurement point</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>In a milled channel under the aluminium plate</td>
</tr>
<tr>
<td>2</td>
<td>In a milled channel topside of the aluminium plate</td>
</tr>
<tr>
<td>3</td>
<td>2 cm above the aluminium plate - left</td>
</tr>
<tr>
<td>4</td>
<td>2 cm above the aluminium plate - centred</td>
</tr>
<tr>
<td>5</td>
<td>2 cm above the aluminium plate - right</td>
</tr>
<tr>
<td>6</td>
<td>4 cm above the aluminium plate - centred</td>
</tr>
<tr>
<td>7</td>
<td>6 cm above the aluminium plate - centred</td>
</tr>
<tr>
<td>8</td>
<td>8 cm above the aluminium plate - centred</td>
</tr>
<tr>
<td>9</td>
<td>10 cm above the aluminium plate - centred</td>
</tr>
<tr>
<td>10</td>
<td>12 cm above the aluminium plate - centred</td>
</tr>
<tr>
<td>21</td>
<td>Scale</td>
</tr>
</tbody>
</table>
4.3 Material preparation

Before each test, a sample of 100 grams of the chosen test material was weighed up. The insulation material was placed in a flat-bottomed bucket and mixed to a lower density, using a power drill with a paint mixer. The equipment used is shown in Figure 16. The mixing time varied from sample to sample, depending on the initial density, but was approximately one minute. Insulation at the edges was harder compressed at more than one side, and needed a bit more mixing before all compressed lumps were loosened up.

![Figure 16: The equipment used for mixing the insulation with air: A power drill with a paint mixer and a flat-bottomed bucket.](image)

4.4 Test procedure

All the tests were run with the same settings of the thermostat, and these were checked before each test. The set point was 370 °C and the hysteresis was 0.2 °C.

A test matrix, as shown in Figure 17, was made before the first test. It was decided that the external heating would be turned off when two out of the three TCs placed 2 cm above the aluminium plate reached the temperature chosen from the matrix. The test temperature for the first test of the first material was according to the matrix 300 °C. The next test would be at a higher temperature if smouldering was not initiated, and at a lower temperature if smouldering was initiated. For the next materials, the results from the tests done earlier was used to choose temperature for the first test. Already after the first test ($T_1$), it became clear that the matrix
would not be followed to the details. The matrix was used only as a pointer when choosing test-
temperature for the tests after T1, for all four test-series.

Figure 17: The original test matrix showing start temperature at 300 °C and the following
temperatures to be tested, depending on whether the last test did obtain a self-sustained
smoulder or not. If smoulder, a lower temperature would be chosen for the next test. If non-
smoulder, a higher temperature would be chosen for the next test.

The “TC-ladder” was lowered into the steel pipe, and the prepared insulation was carefully
packet by hand in to the pipe and around the TCs, in order to get an even distribution of the
insulation. All the tests were run with the same bulk density as shown in Table 2. 100 grams of
test material filled 16-17 cm of the steel pipes height.

The total mass on the scale was read off the scales display before the test started, and the
software was set to log data. Mass and temperature was logged for two minutes before the
cooking plate was turned on maximum effect.
When the temperature inside the test sample reached the set limit, the external heating was turned off. Logging continued until all the TC’s in the steel pipe had dropped below 100 °C. This was done both for tests which appeared to smoulder, and for those who did not.

After the test had been stopped, the remaining material in the steel pipe was carefully removed from the steel pipe. It visually inspected and registered how much of the remaining material were black, brown or virgin. The sample was then soaked in water to make sure that any smouldering reaction in it was put out. The test setup was let to cool down completely before the next test.
5 Results

Four different materials have been tested. The four different test series are named $T$, $U$, $V$ and $W$, and each test is numbered. The goal of the testing was to find the temperature conditions, which leads to smouldering, and see which other factors may influence the possible variations in the temperature needed to obtain a self-sustained smouldering. When smouldering is obtained, it is a goal to study the differences and similarities in the combustion of the different test materials, and how this can be connected to the chemical and physical properties of the materials.

The results are displayed in a variety of figures. Separate diagrams for all the tests can be found in Appendix A.

Test $T1$ was stopped after observations that indicated that a self-sustained smouldering combustion was initiated. It was decided after that test that also the tests that showed signs of smouldering, should be let running until all the TCs showed temperatures below 100 °C. $T2$ is not in the data set because it was carried out with different parameters on the thermostat. $T3$ has a negative mass loss and the mass change of $T3$ is therefore not given considerations in the later discussion, because it is assumed to be an error in the logging.

The results show two variations of smouldering reactions, and these have been denoted as Smouldering behaviour type 1 and Smouldering behaviour type 2.

5.1 Test list

Table 4 is a list of tests carried out and the corresponding external temperature applied to the test material before the external heating was turned off.

*Table 4: Test names and the corresponding temperatures to which the materials were heated to by the external heating source*

<table>
<thead>
<tr>
<th>Test</th>
<th>Temperature [°C]</th>
<th>Test</th>
<th>Temperature [°C]</th>
<th>Test</th>
<th>Temperature [°C]</th>
<th>Test</th>
<th>Temperature [°C]</th>
</tr>
</thead>
<tbody>
<tr>
<td>T1</td>
<td>300</td>
<td>U1</td>
<td>275</td>
<td>V1</td>
<td>275</td>
<td>W1</td>
<td>225</td>
</tr>
<tr>
<td>T3</td>
<td>200</td>
<td>U2</td>
<td>300</td>
<td>V2</td>
<td>250</td>
<td>W2</td>
<td>250</td>
</tr>
<tr>
<td>T4</td>
<td>250</td>
<td>U3</td>
<td>250</td>
<td>V3</td>
<td>225</td>
<td>W3</td>
<td>300</td>
</tr>
<tr>
<td>T5</td>
<td>275</td>
<td>U4</td>
<td>290</td>
<td>V4</td>
<td>200</td>
<td>W4</td>
<td>275</td>
</tr>
<tr>
<td>T6</td>
<td>265</td>
<td>U5</td>
<td>280</td>
<td>V5</td>
<td>215</td>
<td>W5</td>
<td>285</td>
</tr>
<tr>
<td>T7</td>
<td>300</td>
<td>U6</td>
<td></td>
<td>V6</td>
<td></td>
<td>W6</td>
<td>290</td>
</tr>
<tr>
<td>T8</td>
<td>275</td>
<td>U7</td>
<td></td>
<td>V7</td>
<td></td>
<td>W7</td>
<td></td>
</tr>
</tbody>
</table>

25
5.2 Explanation of charts

The testing showed three different variations of material reaction to heating. The charts of all tests are in appendices. Pay attention to the differences in the y-axis for the different tests. The charts are scaled to fit the maximum temperatures registered during testing, and they varied significant for the three different behaviours seen. A detailed description of the test setup can be seen in section 4.2.

5.2.1 Non-smouldering behaviour

The charts of the non-smouldering behaviour will be described here, using the chart from one of the tests as an example.

![Chart showing a typical case of non-smouldering behaviour both in temperature/time-curves and in mass loss. This example is from test W1.](image)

Figure 18: Chart showing a typical case of non-smouldering behaviour both in temperature/time-curves and in mass loss. This example is from test W1.

Figure 18 shows the typical results of a test that did not undergo a self-sustained smouldering combustion. The different graphs in the diagram are all denoted W1 because this is the results of test W1. All the graphs marked with a centimetre placement are temperature measurements. The centimetre placement tells how high above the aluminium plate in Figure 9 the TC is placed. The letter c, l or r behind the centimetre placement tells if the TC is placed centered, left or right in the steel pipe, when seen from the front. The graph denoted Under hotplate is also a temperature measurement, and the hotplate mentioned in the charts is the heated aluminium plate, and not the cooking plate. The black dotted line marks when the external
heating source was turned off. On the secondary axis one can see the mass change during the test time. The mass change starts at zero, and has negative values due to the mass loss.

In Figure 18, it can be seen that the temperature in the test material increased as the temperature in the aluminium plate increased. When the external heating source was turned off, the temperature in the aluminium plate started to decrease. The temperature in the test material increased some more. When the temperature in the test material started to decrease as well, the temperature in the lower part of the insulation crossed above the temperature of the aluminium plate for a short while. The temperature in the test material then continued to decrease until it went below 100 °C, where the logging was turned off and the test was stopped.

5.2.2 Smouldering behaviour type 1

Figure 19 is an example of one of the two smouldering behaviour seen in the tests, with a description of what is seen.

![Figure 19: Chart showing a typical case of smouldering behaviour type 1, both in temperature/time-curves and in mass loss. This example is from test U4.](image)

In Figure 19, the denotation of the graphs are the same as in Figure 18. The test sample U4 was heated until the sample reached 290 °C, measured by the TCs 2 centimetres above the aluminium plate. This took 38 minutes and 48 seconds, which gives a rate of external heating of 485 °C/h. It can be seen in Figure 19 that when the external heat source is turned off, and the temperature in the aluminium plate decreases, the temperature in the insulation increases above the temperature in the aluminium plate. There is a mass loss in the beginning of the heating,
which stabilizes slightly when the external heating source is turned off. When the temperature starts to increase in the insulation again, the mass loss increases as well, before stabilizing again when the temperatures starts to drop. Maximum temperature for $U4$ was 397 °C, and it was reached after three hours and nine minutes. The mass showed no sign of further change as the temperature dropped. When the temperature dropped to the limit of 100 °C the test was stopped. The total mass change was 44 grams, which is 44 % of the total mass of 100 grams.

5.2.3  Smouldering behaviour type 2

The other type of smouldering behaviour seen in the tests is shown in Figure 20. The samples undergoing this type of smouldering behaviour reached higher temperatures and had a much higher mass loss, than the ones undergoing the first type of smouldering behaviour, described in section 5.2.2. Note that the scale of the $y$-axis is not the same as in Figure 19.

![Figure 20: Chart showing a typical case of smouldering behaviour type 2, both in temperature/time-curves and in mass loss. This example is from test V3.](image)

The denotations in Figure 20 are, as in Figure 19, the same as in Figure 18. Sample V3 was heated until the temperature measured by the TCs 2 centimetres above the aluminium plate was 225 °C. This took 28 minutes and 45 seconds, which gives a rate of external heating of 498 °C/h. For the first 2-3 hours of the test time, the temperature development is similar to the temperature development in smouldering behaviour type 1, after which it changes. In Figure 20 it can be seen that the temperatures continue to increase after 2.5 hours, and the temperature increase is significant. The same can be seen in the mass change. When the temperature
increase, the change of mass is larger. The mass loss continues until the temperatures reach their peaks. The maximum temperature was 697 °C, and it was reached after 3 hours and 55 minutes. When the temperature starts to drop, the mass change stabilizes, as seen with the first type of smouldering behaviour. The total mass change for test V3 was 88 grams of a total of 100 grams, which gives a mass loss of 88%.

5.3 Distribution of test results
The tests have been divided in the three different categories as mentioned in section 5.2 and this can be seen in Figure 21. The dividing is done based on the typical behaviour in the three different categories when it comes to mass change and maximum temperatures. Which tests were run at which temperature can be seen in Table 4. Test T1 is not included in the figure, because the early stop of the test made it impossible to know if it was smouldering behaviour type 1 or smouldering behaviour type 2 in the test. Test T7 was run at 300 °C like T1, in order to try to replicate T1, but T7 did not smoulder. T8 was an attempt to replicate T5, but T8 did not smoulder like T5. This can be seen in Figure 21 with the two different markers at 275 °C for the T-series.
Figure 21: Distribution of the test results in the three categories non-smouldering behaviour, smouldering behaviour type 1 and smouldering behaviour type 2. The figure shows which behaviour was seen for the different test series and test temperatures.

From Figure 21 it can be seen that none of the samples in the U-series did undergo smouldering behaviour type 2. One of the three smouldering samples in the V-series did undergo smouldering behaviour type 1, while the other V-tests did undergo smouldering behaviour type 2.

T-series had both samples that smouldered and that did not, at 275 °C and 300 °C. The distribution shows that T-, U- and W-series all had a boundary between smouldering and non-smouldering in the same area. This boundary was in the area of external heating up to 270-290 °C measured in the test sample 2 centimetres above the aluminium plate, before the external heating was turned off. V-series stands out from the other three test series, with obtaining a smouldering combustion at a much lower level of external heating. For V-series, the external heating necessary is 215-225 °C measured in the test sample 2 centimetres above the aluminium plate.


5.4 Pictures of tested samples

In this section photos will illustrate the three different types of reaction to external heating that is described in section 5.2. The chosen examples are representative for the amount of residue and the distribution of different coloured residue seen in the three different alternatives.

5.4.1 Sample that did not undergo a self-sustained smouldering

*Figure 22: Sample W1 as seen from above before the test was started.*

*Figure 23: Sample W1 seen from the side after the test was finished.*

*Figure 24: The cross section of sample W1 seen from the side after the test was finished.*
5.4.2 Sample that did undergo smouldering behaviour type 1

Figure 25: Sample U5 as seen from above at 3 hours and 19 minutes into the test.

Figure 26: Sample U5 seen partly from the side and partly from the top after the test was finished.

Figure 27: The cross section of sample U5 seen from the side after the test was finished.
5.4.3 Sample that did undergo smouldering behaviour type 2

Figure 28: Sample V1 as seen from above at 3 hours into the test.

Figure 29: Sample V1 as seen from above after the test was finished.

Figure 30: Sample V1 seen from the side after the test was finished.

Figure 31: The cross section of sample V1 seen from the side after the test was finished.
5.5 Mass observations after test

After the tests was finished, observations of the remains of the test materials were done.

5.5.1 Total mass loss in context with maximum temperature

The total mass loss has been plotted together with the maximum temperature for all the tests, in Figure 32. Colours are used to divide the results into the three categories described in section 5.2, non-smouldering, smouldering behaviour type 1 and smouldering behaviour type 2.

![Figure 32: Total mass loss and the corresponding maximum temperatures. The total mass loss of the tests are divided by colour into the three different categories mentioned earlier.](image)

From Figure 32 it can be seen that there is a correlation between the maximum temperature and the total mass loss. The samples that did not smoulder all have a mass loss below 20 grams and a maximum temperature between 240 °C and 310 °C. The samples that did undergo smouldering behaviour type 1 all have a mass loss of 40-50 grams and a maximum temperature between 380 °C and 440 °C. The samples that did undergo smouldering behaviour type 2 clearly stands out in the chart. They have a mass loss of 80-90 grams and maximum temperature in the area of 660-700 °C.
5.5.2 Residue distribution

The percentage distribution of residue is based only on visual observations and surface measurements. The material in the T-series fell apart when the remaining mass was removed from the steel pipe. The distribution of residue in the T-tests is based only on visual observations of the remains after they were removed from the steel pipe and placed in a larger steel bucket. Most of the tests in the U-, V- and W-series kept an cylindrical shape after they had been removed from the steel pipe, so the distribution of residue could also be found by using a ruler and measure how much of virgin material, brown- or black residue the remaining mass consisted of.

Due to the similarities in total mass loss shown in Figure 32, the distribution of residue is displayed in charts for each of the three categories mentioned in 5.2. Figure 33 show the residue distribution of all the samples that did not smoulder. There are some variations in the distribution, but the overall observation is that the residue distribution is very similar for the samples in the same category, even though they are of different materials.

The residue distribution of the samples with smouldering behaviour type 1, is displayed in Figure 34. From the figure it can be seen that most of the residue is black. The same can be seen for the residue of the samples with smouldering behaviour type 2, in Figure 35. What makes the big difference in the residue between the two types of smouldering behaviour is seen when the residue is seen in context with the mass loss. Figure 32 shows that the total mass loss for the samples with smouldering behaviour type 2 is more than double the total mass loss for the samples with smouldering behaviour type 1. The pictures in section 5.4.2 and 5.4.3 supports this. The sample with smouldering behaviour type 1 on page 32 has kept its shape, but most of it is black, while the sample with smouldering behaviour type 2 on page 33 has shrunk substantially, and the aluminium plate underneath the sample can be seen in Figure 29. The pictures in Figure 26 and Figure 30 illustrates how big difference there is in the total mass loss between the two different smouldering behaviours. In the first case, the cylindrical shape fills out the shape of the steel pipe, but for the second case, the residue seems to cling to the «TC-ladder» and there are big gaps all around the sample.
Figure 33: Residue distribution of samples that did not smoulder

Figure 34: Residue distribution of samples with smouldering behaviour type 1

Figure 35: Residue distribution of samples with smouldering behaviour type 2
5.6 Rate of external heating

The rate of external heating in context with total mass loss can be seen in Figure 36. Figure 37 displays the rate of external heating in context with maximum temperature. There are no obvious correlations between neither the rate of external heating and the total mass loss, nor the rate of external heating and the maximum temperature.

Figure 36: Rate of external heating in context with total mass loss

Figure 37: Rate of external heating in context with maximum temperature
5.7 Time to ignition, ignition temperature and total combustion time

Figure 38: Test temperature and ignition temperature of all the samples with a smouldering behaviour

Figure 38 displays the test temperature and the ignition temperature. In this case, the ignition temperature is the temperature measured by TC that first reaches 20 °C above the temperature in the aluminium plate, after the external heating source is turned off. Test temperature is the temperature that the materials were heated to by the external heating source before the external heating was turned off, as described in section 4.4.

For T-, U- and V-series, the ignition temperature was found from the TC at 4 cm. For W-series, the first TC to have a rise of 20 °C after the cooking plate was turned off, was the TC at 6 cm. W-series have a higher test temperature than ignition temperature, while the other three test series have a slightly higher ignition temperature than test temperature.

It seems like the ignition temperature relates to the test temperature for the first three test series. Lower test temperature leads to lower ignition temperature. It can also be seen in Figure 38, that for the three test series, the difference between the test temperature and the ignition temperature is smaller with a higher test temperature.
Figure 39: Time to ignition, total combustion time and ignition temperature of all samples with a smouldering behaviour

In Figure 39, the time to ignition and total combustion time are displayed together with the ignition temperature. Time to ignition is the time from the cooking plate is turned on to the time where the ignition temperature is found. The total combustion time is the time from ignition and until all the TCs have values below 100 °C.

The time to ignition does not differ much for the four test materials. It seems like there is a correlation between the ignition temperature and the total combustion time.
6 Discussion

6.1 Procedure and choice of methods

In this section, choices done in association with the test materials and test procedure will be assessed.

6.1.1 Materials

In order to run as many tests as possible and have test results that are comparable, it was chosen to test only wood fibre insulation and not cellulose insulation. The materials tested were chosen because they are typical representatives for the materials on the market today, at the same time as the four materials have some common features. This makes it easier to compare the test results of the different materials, and study how the different factors influence the development of a smouldering combustion. The fire retardants used in the test materials are not the one most commonly used in Europe, according to the information from the representative from a European cellulose insulation organisation, as described in section 3.3.

6.1.2 Test equipment

The insulated steel pipe was chosen as «sample container» to avoid heat loss to the sides during tests. This gives an approximately «one-dimensional» system, with airflow in primarily one direction and a minimum of exterior influence along the boundaries of the test samples. There will not be any discussion of airflow, because all tests were run at the same approximately «one-dimensional» system. The down side of using an insulated steel pipe is that visual observations during the tests is limited. It is only possible to observe visual changes from the top of the sample. Unless there is changes in the top of the sample, visual changes will not be seen until the sample is removed from the steel pipe.

The aluminium plate was placed between the insulated steel pipe and the cooking plate to help give an even heat distribution to the material in the steel pipe.

The «TC-ladder» was equipped with TCs before the size of the test samples was decided, and this was not reconsidered before the testing started. There were only TCs up to 12 centimetres above the aluminium plate, while the test samples had a height of 16-17 centimetres, thus leading to no data for the temperature development in the top 4-5 centimetres of the test samples.
6.1.3 Material preparation

The density of the test sample used in the test was in the range recommended by the supplier, in order to test a density relevant to the daily use of the materials. Samples of 100 grams in a 16.5 centimetres high cylinder gave a density of 34 kg/m$^3$. This is in the interval recommended by the supplier in section 3.2.3.

For professional use, the entrepreneurs have machines that loosen the material up before it is blown into place, as mentioned in section 3.2.3. This process had to be simulated in the laboratory in order to get the structure of the material as similar as possible to materials installed in a building. The machinery used could not have sharp knives or edges that would lead to a modification of the fibres, but it had to be strong enough to loosen the material up. A power drill with a paint mixer attached was chosen, as the paint mixer has round edges, so it would not cut the fibres during mixing. A flat-bottomed bucket was used to make sure that the paint mixer could reach all of the sample during mixing, so that the test sample got thoroughly mixed.

6.1.4 Test procedure and observations after test

The first test temperature, at 300 °C, was chosen on the basis of the temperatures for thermal decomposition of wood in section 3.2.1. The border between the pyrolysis zone and the charred wood is approximately 300 °C, and if wood fibre behaves like wood, this would be a suitable temperature for the first test. A set point on the thermostat of 370 °C was chosen, because this temperature was assumed higher than the temperature to start a smouldering combustion in the insulation. It was not known before the testing started if, how and for how long the sample would burn if it ignited. The decision to stop the tests when all TCs showed values below 100 °C was also done based on the results from the literature study in section 3.2.1, which stated that it is first at 100 °C the processes of thermal decomposition of wood starts.

The test setup was let to cool down completely before next test, so that all tests would start at approximately the same temperatures. If the tests had not started at the same temperature, the time to ignition would not have been comparable for the different tests.

Three of the test materials had a structure that made the material sample hold its cylindrical shape when removed from the steel pipe, but for one material, the T-series, it did not. This made the visual observations of the rest mass more difficult to visually quantify. The estimates of
mass residue after test in the three groups *virgin, brown and black*, is therefore for the T-series rougher than for the other three test series.

A reproduction of the tests *T1* and *T5* was not successful. The reason for this is unknown. All the preparation and procedures had been done in the same way for all tests. There are two known factors that may have had an influence. In the time area from the original test and until the reproduction, the test area under the extractor hood had been built in with a wall and a door to make it more enclosed. This could have affected the composition of the air surrounding the tests, and air supply to the combustion. It is a well known fact that air flow have influence on smouldering combustion (22,23). The other factor is that the test material was taken from different places in the package. The first tests were run with samples from the outer edge. The later tests samples were taken from the same opening in the package, but deeper into the package. As mentioned in section 3.4.3, it has been shown in former studies (4,25) that the fire safety of cellulose insulation degrades and may lose its ability to retard fire. During the storage time from the first test to the reproduction test, material degradation may have caused the problems with the reproduction of the test.

### 6.2 Discussion of results

In this section, the results found from testing will be discussed and compared to the literature in section 3. This is done in order to find the conditions necessary for initiating a smouldering combustion in wood fibre insulation, and to study the smouldering combustion of the different insulation materials tested.

The diagrams in section 5.6 will not be discussed because of the lack of correlations in the diagrams. There is a possibility that another method for analysing data could be more suitable for finding correlations between the rate of external heating and other factors, but for now, there was not found anything of significance there.

#### 6.2.1 Initiating of smouldering combustion in test samples

The three indications of smouldering used by Krause et al. (13) have been used to decide if the test samples did or did not have an onset of self-sustained smouldering combustion. All the tested materials did have a change of colour, but the materials that are denoted as *non-smouldering* had a significant residue of virgin material as showed in Figure 33. This can also
be seen in the picture in Figure 24. A small mass loss and no significant rise of temperature above the temperature of the external heating, makes it clear that these samples did not smoulder. Even though there were no self-sustained smouldering in these tests.

The test samples denoted with *smouldering behaviour type 1* and *smouldering behaviour type 2* had a change if colour in most of the test sample. This can be seen in the distribution of residue in Figure 34 and Figure 35. There were big variations between the two different types of smouldering behaviour mentioned, but all the samples had a temperature rise well above the temperature of the external heating source, and a significant mass loss, so they all did smoulder. Figure 26 and Figure 27 are good illustrations of the different zones in a cellulosic material undergoing a smouldering combustion, as described in section 3.1 and Figure 1.

### 6.2.2 Different smouldering behaviours

Figure 32 illustrates the maximum temperatures and total mass loss of the tests. The maximum temperature seen in the samples undergoing *smouldering behaviour type 2* was close to 700 °C, which is in the same area as the temperature of a secondary char oxidation of polyurethane (7), as mentioned in section 3.1. The pictures in section 5.4.3 show that it was a major mass loss. The mass had shrunk so much that there were big cavities between the sample residue and the steel pipe, which can be seen in Figure 29. The samples undergoing *smouldering behaviour type 1* had a significant mass loss, of 40-50 %, but not as severe as the one of the samples undergoing *smouldering behaviour type 2*, which had a mass loss of 80-90 %. The *smouldering behaviour type 1*-samples had kept their cylindrical shape, filling the whole steel pipe as before the test started. This indicates that the samples denoted *smouldering behaviour type 2* have gone through a second round of combustion. This assumption is backed up by the results showed in the diagram in Figure 20, where it is clear that the temperatures are on the way down, before they start to increase heavily. The behaviour of the samples denoted *smouldering behaviour type 2* appears to be typical for a material with a secondary oxidation of the char (7,14,15), described in section 3.1.

In other words, the results show that there is a clear distinction between the two different types of smouldering behaviour, and this can have implications of the fire safety when choosing building insulation materials. Materials undergoing a secondary oxidation leave large cavities between the residue of the insulation and the surrounding structure, in this case the steel pipe.
Cavities can lead to an increased airflow to the char, which then can lead to an increased oxidation, higher temperature and the ignition of adjacent wood (22).

Let us assume that this also is the case when the insulation is installed in buildings. If a smouldering combustion occur in a timber frame building insulated with a wood fibre insulation that can undergo a secondary oxidation of the char, it may lead to a very dangerous situation. The formation of cavities between the timber frame and the insulation can in the worst-case lead to an ignition and flaming fire of the timber frame. This factor can therefore be important to consider when choosing building insulation materials, in should be taken into account in future work on the topic.

The main goal of the thesis was to find the condition necessary for a smouldering combustion to be initiated. The external heating necessary to initiate a smouldering combustion in the four different test materials, can be seen in Figure 21 in section 5.3. There is a significant difference in the temperatures necessary to obtain a smouldering combustion between material V and the other three materials. A smouldering combustion is initiated in material V at much lower temperatures than in the three other materials. The onset temperature found was 275 °C for material T, 280 °C for material U, 225 °C for material V and 290 °C for material W. From Table 2 it can be seen that material V and material W have the same fibre size, but different amount of fire retardant added. Material V and material U have the same amount of fire retardant added, but material V have smaller fibre size that material U. The results indicates that both the fibre size and the level of fire retardant added may have a significant influence on the temperature necessary to obtain a smouldering combustion.

6.2.3 Influence of geometrical factors and flame retardants in the smouldering combustion

Material T has a different fibre structure than the other three, but it resembles material U the most in size, and has approximately the same level of fire retardant added as material U and material V. Figure 38 shows that material T and material U have a similar correlation between test temperature and ignition temperature as well, even though they are made from different types of wood. This indicates that the use of fir or spruce in the wood fibre insulation does not have an influence on the fire safety of the material.

Figure 21 shows that material W, which has the same fibre size as material V but only half the amount of fire retardant added, needs a much higher temperature to obtain a smouldering combustion, than what is needed for material V. This indicates that the fire retardant added to
the materials, can make the initiation of a smouldering combustion easier in a material with smaller fibre size. In section 3.4.2, it can be seen that flame retardants will not necessary prevent a smouldering combustion (9), and some additives even promote the char-forming process. The background in section 3.1, states that it is the oxidizing of the char that gives the heat generation in a smouldering combustion (8,9), and more char means more fuel to the combustion.

In other words, both the fibre size and the retardants can affect the smouldering combustion, and both factors must be considered when choosing building insulation materials. It cannot be expected that all building entrepreneurs know how fibre size and fire retardants may affect the fire safety of the materials. It is therefore important that the producers and sellers of building material have good insight into the properties and can guide the entrepreneurs to the right material for their use. The testing done in this thesis is only indicative. More testing is needed in order to support these results, and to do further studies on how fire retardants and fibre size affect the smouldering combustion in building insulation materials. This will be described in section 8.

In Figure 39 it can be seen that when material V is ignited at low temperatures, the combustion time is longer, but this is not consequent. When the ignition temperature continues to drop, the combustion time drops again. The total combustion time varies a lot between the tests of material V and W, even though they have the same fibre size, so the fibre size does not seem to be the major influence on the correlation between the total combustion time and the ignition temperature. This is not in correlation with the results of Palmer (23), which showed that particle size of the dusts influences the smouldering rate. The factor separating material V and material W is the amount of fire retardant. Results from Kokkala (21) showed that the difference in smoulder velocity for retarded and unretarded cellulose insulation, was 30 %. Smouldering velocities have not been determined in this thesis, but the total combustion time is lower for material W than for material V, and this can indicate that when a smouldering combustion is initiated, the fire retardant used in these materials, can slow the combustion. Even though the fire retardants can slow the combustion, an onset of smouldering combustion with temperatures from external heating as low as 225 °C is a hazard that has to be taken serious. An example of areas where increased temperatures can be experienced, is areas with technical installations and machinery. These are not necessarily the ones most often controlled, nor easiest to monitor due to cramped spaces and partly hidden installations. Obtaining a smouldering combustion at low external heating can therefore develop into a very dangerous situation, even though the fire retardant slow the smouldering down.
In Figure 39, it can be seen that for material $U$ and material $W$, there is a correlation between the ignition temperature and total combustion time as well. For both of the materials, low ignition temperature give long total combustion time, and a higher ignition temperature leads to a shorter total combustion time. Material T has a total combustion time in the same time interval between 2.6 - 3.1 hours as material $U$ and material $W$, and an ignition temperature between the two. The total combustion time for test samples of material $U$ is slightly longer than the total combustion time for the samples of material $W$. If this is due to the difference in fibre size, it is the opposite of the results Palmer (23), showed in Figure 3, which shows a higher smouldering rate for a coarser material. It must also be considered that the samples of material $W$ not only had a smaller fibre size than material $U$, it also had only half the amount of fire retardant added.

Material $V$ had the same amount of fire retardant as material $U$, but smaller fibre size. For test $V2$, which only obtained smouldering behaviour type 1 like the $U$-samples, the total combustion time seen in Figure 39 was significantly higher. This is in correlation to Palmers (23) results. Test $V1$ and $V3$ had a shorter total combustion time, but they obtained a secondary oxidation of the char, which material $U$ did not, and this may influence the comparison of the results of the two materials.

**6.2.4 Discussion of hypotheses**

Up until now, the results from the testing has been discussed. In this section, the results will be summarized in order to see if they support or falsify the hypotheses.

There were four hypotheses in this project:

1. Higher bulk density in the test will give lower temperature for onset smouldering, due to a higher heat conductivity.
2. A self-sustained smouldering fire will occur at lower temperature in the insulation material with smaller fibre size, than in material with larger fibre size, due to a larger surface/volume-ratio.
3. A higher level of added fire retardant chemicals in the insulation material will lead to the insulation material being more fire resistant, and a higher temperature exposure will then be necessary to obtain a self-sustained smouldering fire.
4. The material will undergo combustion faster with smaller fibre-size, due to a larger surface/volume-ratio.
Hypothesis 1, which involves variations in bulk density, is not discussed because all testing was done with the same bulk density. This is due to the time limitations of the project.

Hypothesis 2, which stated that a self-sustained smouldering fire will occur at lower temperature in insulation with small fibre size is supported, but this depends on the level of fire retardant added, as discussed in section 6.2.3. For two materials with the same level of fire retardant added, smouldering will occur with lower external heating as the fibre size decrease.

Hypothesis 3 is falsified. In Figure 21 it is showed that for two test samples of the same material and fibre size, but different levels of fire retardant added, it is the material with the highest level of fire retardant that ignites at the lowest temperature. This is an important factor to remember when choosing building insulation materials. The material that look the safest on paper is not necessarily the safest in use.

Hypothesis 4 deals with the total combustion time as a function of fibre size. In Figure 39 there are indications that a material with smaller fibre size will combust more slowly than a material of larger fibre size and the same level of added fire retardant, but the hypothesis is not fully supported. The ignition temperature is very different for tests of material \( U \) and material \( V \) that had a big difference in total combustion time. For test \( V1 \) that has an ignition temperature closest to the ignition temperature of material \( U \), the total combustion time was in the same area as the tests of material \( U \). This leads to ignition temperature being a factor to consider as relevant for the total combustion time.
7 Conclusions

Fibre size and level of added fire retardant both influence the onset of a self-sustained smouldering combustion in wood fibre insulation. There are indications that a higher level of fire retardant added may slow down the smouldering combustion after it is initiated.

The temperatures from external heating necessary to obtain a self-sustained smouldering fire in the test material are: 275 °C for material T, 280 °C for material U, 225 °C for material V and 290 °C for material W.

The testing has showed that two different types of smouldering behaviour can be obtained in wood fibre insulation. Smouldering behaviour type 1 have maximum temperatures in the area of 380 °C to 440 °C, and a total mass loss of 40-50 %. Smouldering behaviour type 2 has maximum temperature in the area of 660 °C to 700 °C, and a total mass loss of 80-90 %. Smouldering behaviour type 2 is found to be typical for materials undergoing a secondary oxidation of the char.

A self-sustained smouldering combustion occurs at lower temperature in insulation material with smaller fibre size than in a material with larger fibre size, when the materials have the same level of fire retardant added. It is not determined whether a material of smaller fibre size will undergo a smouldering combustion faster than a material with larger fibre size.

A higher level of fire retardant added gave an onset of a smouldering combustion at lower temperatures, when the fibre size of the material is the same. There are indications that the material with the lowest level of added fire retardant needs the highest external temperature exposure to obtain a self-sustained smouldering fire, out of all tests in the project. Of the two materials with the same fibre size and different level of added fire retardant, the one with the lowest amount of fire retardant has the shortest total combustion time, but time to ignition and ignition temperature may also be factors influencing this.
8 Suggestions for future work

The testing done in this project is indicative. Going further into specific parts of the smouldering combustion of combustible building insulation materials and doing more testing in those specific areas, will give results that say more about the average behaviour of combustible building insulation materials. Specific suggestions to further testing is to study what influences the external heat necessary exposure to obtain a self-sustained smouldering behaviour, and how it affects the smouldering of the material:

- Doing tests of one given material with several different levels of the same type of fire retardant added.
- Doing tests of one given material with several different fibre sizes and the same level of fire retardant for all the different fibre sizes.
- Doing tests of one given material with the same fibre size and level of added fire retardants in all tests, but varying the bulk density in the tests.
- Doing tests with wood present to resemble an insulated timber frame construction.

For all tests, it would be interesting to, at the same time, test a sample without fire retardant added. Comparing materials with and without fire retardant added would give valuable insight in how the fire retardant influence the material.

There is a possibility that the use of more advanced methods of analysing data, could give insight to more correlations and results from the data sets in this project. That would be a way to continue working on the topic without doing more testing.
9 References


10 Problem Description

The main goal of this thesis is to find the conditions required for a smouldering fire to occur in combustible building insulation materials, e.g. cellulose based insulation materials, and to study the development of the smouldering in the materials. Factors to be considered are type of material, material shape and density, different temperature influence, and the level of fire retardant additives. The thesis will consist of a literature study of smouldering fire in combustible insulation materials, followed by the planning and execution of laboratory testing of different combustible building insulation materials.
11 Appendices

A Test charts

Figure A1: Temperature-time-curves and mass change curve for test T1
Figure A2: Temperature/time-curves and mass change curve for test T3
Figure A3: Temperature/time-curves and mass change curve for test T4
Figure A4: Temperature/time-curves and mass change curve for test T5
Figure A5: Temperature/time-curves and mass change curve for test T6
Figure A6: Temperature/time-curves and mass change curve for test T7
Figure A7: Temperature/time-curves and mass change curve for test T8
Figure A8: Temperature/time-curves and mass change curve for test U1
Figure A9: Temperature-time-curve and mass change curve for test U2
Figure A10: Temperature/time-curves and mass change curve for test U3
Figure A11: Temperature/time-curves and mass change curve for test U4
Figure A12: Temperature/time-curves and mass change curve for test U5
Figure A13: Temperature/time-curves and mass change curve for test V1
Figure A14: Temperature/time-curves and mass change curve for test V2
Figure A.15: Temperature/time-curves and mass change curve for test V3
Figure A16: Temperature/time-curves and mass change curve for test V4
Figure A17: Temperature/time-curves and mass change curve for test V5
Figure A18: Temperature/time-curves and mass change curve for test W1
Figure A19: Temperature/time-curves and mass change curve for test W2
Figure A20: Temperature/time-curves and mass change curve for test W3
Figure A21: Temperature/time-curves and mass change curve for test W4
Figure A22: Temperature-time-curves and mass change curve for test W5
Figure A23: Temperature/time-curves and mass change curve for test W6