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# Recycling of Glass Wool Waste

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## Preface

This thesis was written at NTNU at the department of material science. The thesis was given by GLAVA, during spring semester 2018. I would like to first and foremost thank my supervisor Merete Tangstad for patiently guiding me through this semester with valuable advice and feedback. Equally important; I must thank the people at GLAVA in particular Lars Jørgen Krohg and Mads Rikardsen for giving me the opportunity to work on such an interesting challenge. I would also thank for the opportunity to work at GLAVA Askim during the summer. I would also like to thank everyone who have helped me through various experimental challenges during this semester, including, but not limited to: Dmitry Slizovskiy, Trygve Schanche and Sergey Khromov

## Abstract

The importance of environmentally friendly solution is an ever-increasing concern of industries all over the country. The glass wool industry has for a long time had a problem regarding waste from their production process. The solution today is to dump the waste at a landfill, which is regarded as an unsatisfactory solution. The most desired solution would be to throw the waste back into the furnace as raw material for the production process. However due to a certain amount of carbon based organic content, problems like excessive bubbles and reduction of surrounding glass will appear if the organic content reach the melt. In the production process, the melted glass is covered by a carpet of unbelted raw material. Waste added to the furnace would first be submerged in this carpet and be exposed to elevated temperatures, in an atmosphere low on oxygen, before reaching the melt.

This thesis has a twofold objective. First; develop a method to simulate the glass melting furnace to explore and analyze glass wool waste in the carpet layer of the furnace. Second; to explore the incineration of the waste at different temperatures and timeframes, both in atmospheric conditions and in oxygen lacking atmosphere like beneath the glass carpet.

The method developed succeeded in determining the temperature gradient in the carpet, and thus which temperatures the waste was subjected to beneath different depths. At a depth of 8 cm and lower, the lack of oxygen prevented the complete incineration of the waste, and carbon residue was observed. In atmospheric conditions the organic content was quickly removed at temperatures significantly lower and at much shorter time then for the waste in the oxygen lacking atmosphere.

## Sammendrag

Miljøvennlige løsninger er et stadig økende fokus for industrier rundt om i landet. Glassull industrien har i lengre tid hatt utfordringer angående glassull avfall fra produksjonsprosessene. Dagens løsning innebærer å dumpe avfallet på fylling, og en bedre løsning er ønsket. Den beste løsningen ville innebære å putte avfallet tilbake i smelteovnen, og bruke det som råmateriale. Avfallet inneholder i midlertid en viss mengde karbonbasert organisk bindemiddel, som i kontakt med smelten vil medføre skape store mengder gass. Smelten i ovnen er dekket med et teppe av usmeltet råvarer som er viktig for å opprettholde kontroll over temperaturen. Avfall som blir tilsatt i smelteovnen vil først være en del av dette teppet før det når smelten. Atmosfæren her vil være oksygenfattig og til tross for høye temperaturer er det usikkert hvorvidt bindemiddelet brenne bort før det når smelten

Denne oppgaven har to målsetninger. Først; å utvikle en metode for å simulere glassovnen brukt i industrien, for å kunne måle temperaturgradienten og analysere glassull avfallet i mengeteppet. Andre målsetning er å undersøke hvilke betingelser karbonet i bindemiddelet blir brent bort, dette innebærer forskjellige temperaturer, tid og hvorvidt avfallet ligger i en oksygenrik atmosfære. Metodeutviklingen var vellykket til å vise hvilke temperaturer glassullavfallet ble utsatt for og hvilken dybde i mengeteppet. Under atmosfæriske betingelser brente avfallet raskt bort, hvor nesten alt var fjernet etter 3 minutter på 500 °C. I mengeteppet var det fortsatt karbonrester på 8 cm dybde og dypere, til tross for å ha vært utsatt for høyere temperaturer over lengre tid.

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## 1. Introduction

Recycling of glass wool waste have been a challenge for the glass wool industry for many years. The industries at large experience an ever-increasing pressure for more environmentally friendly production processes. As a result, the demand for new solution for production related waste is increasing. Recycled glass as a raw material is in high demand for many industries, most importantly the glass wool industry itself. However due to the addition of an organic binder to the wool during production, the waste cannot easily be used as raw material. If glass wool waste is returned to the furnace along with the rest of the raw materials, undesired effects like gas bubbles might appear if the carbon is not removed before it reaches the melt.

This thesis has a two main goals. First; to develop a method to simulate the parameters in the glass furnace used for glass wool production. This development is will be explained by explaining every simulation performed an evaluate what worked and which improvements applied each time.

The second goal is to examine the incineration of the organic content in the glass wool, in both atmospheric conditions and in atmospheres with low oxygen. This is done by performing incineration experiments in atmospheric conditions as well as examine the samples from the simulation processes where the organic content have been incinerated in a low oxygen environment.



## 2. Theory

In this section the theory of glass production, and glass wool recycling is explained. The theory can be divided into two parts. The first part explains the broader field of glassmaking and glass microstructure, with special emphasis on soda-lime-silica glass. The other section emphasizes more particularly on the production process relevant in this thesis, and the recycling challenges that follows.

### 2.1 Theoretical groundwork on glass

This section will explain the basis of glass forming, the definitions of glass, network theory and the different role each component plays in the glass structure. An emphasis will be held on soda-lime-silica glass and the other oxides that are relevant in this scenario. A section will also discuss the heat transport in glass.

#### 2.1.1. Glass structure

Several different definitions of glass have existed over the years, all with its own strength and weaknesses. One characteristic every glass share is that of the glass transformation behavior. This phenomenon occurs over a certain temperature range. In this range the glass will solidify without crystallizing. A glass can thus be defined as amorphous solid with only short range periodic atomic structure. Another definition would be *a frozen in undercooled liquid* [1]. Figure 1 illustrates how the volume decrease in when a liquid is cooled, for a crystal and a glass.

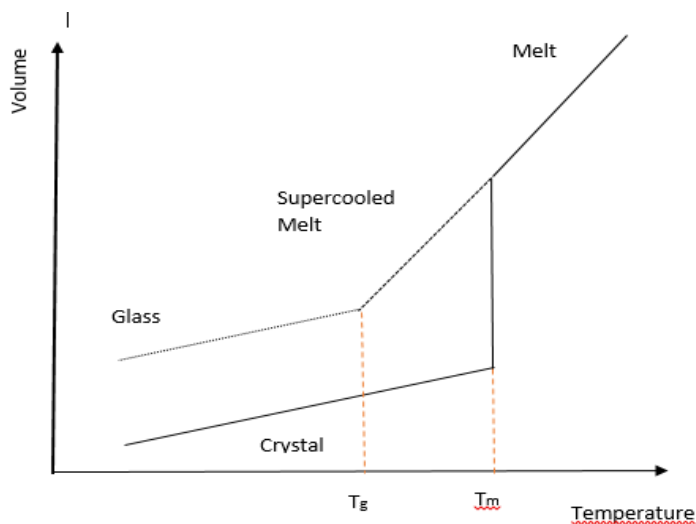


Figure 1: For a crystal, the volume will instantly decrease when solidification temperature is reached. For a glass it will continue to decrease at the same rate until transition temperature is reached [1]

Being an amorphous material, glass does not crystallize when cooled from a liquid like metals do. Instead, the melt will gradually increase in viscosity. At some point the viscosity is too high for equilibrium to occur and is thus regarded as a solid. Zachariasen [2] proposed that for a

glass to form the material had to (1) contain cations surrounded by oxygen tetrahedra or triangles, (2) the polyhedral must be connected only by their corners and not edges (3) some of the oxygen must be only connected to two cations.

The microstructure of glass and crystals does in fact have some similarities. In both cases the structure is organized as tetrahedrons. The difference however lies in how these tetrahedrons themselves are organized. In a crystal the tetrahedrons have a repeatable pattern, while in a glass they are randomly oriented. Figure 2 shows the difference between the microstructure in a glass and a crystal.

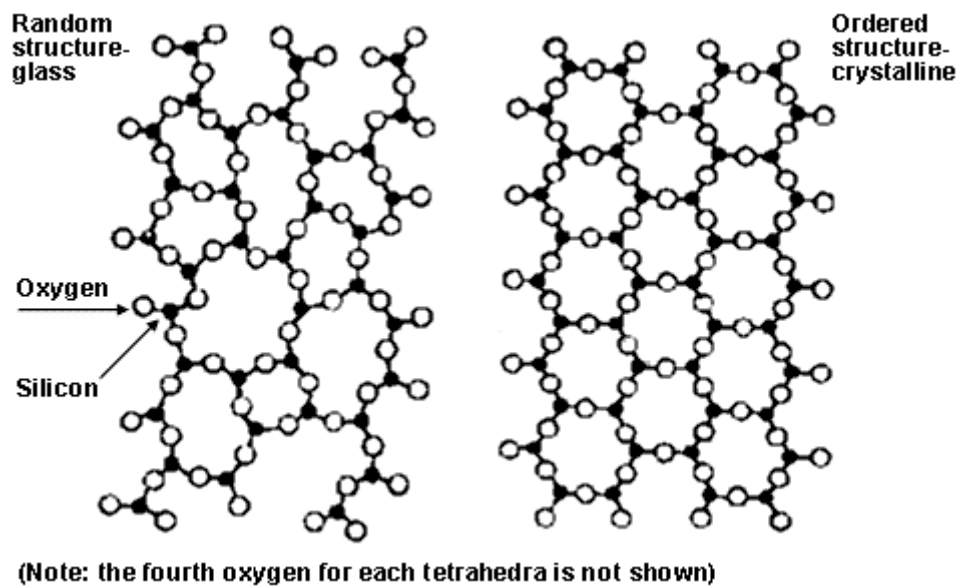


Figure 2: The difference between a crystal and a glass. The glass shows no repeated structure [3]

Silica glass as shown in figure 2 contains silicon and oxygen. Alkali oxide are however often added to the mixture which will change the properties of the glass. If the glass only contains silica, all the  $O^{2-}$  atoms will be connected to two  $Si^{4+}$  ions. For this reason, the oxygen atoms are referred to as *bridging atoms*, while the silicone atoms are called *network formers*. In the case of glass containing alkali oxide like  $Na_2O$  then the  $Na^+$  will open the connection between silicon and oxygen, leaving the  $O^{2-}$  ions connected to only one silicon atom. These oxygen atoms are referred to as *non-bridging atoms* [1].

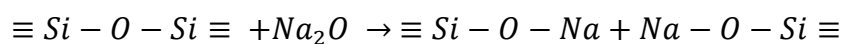
The kind of polyhedral that is formed is dependent on the radius ratio between the cation and the anion. With a ratio of 0,155 a triangle will be formed with one cation surrounded by three anions. As the size of the cation increases, the coordination number will increase, and at 0,225 a tetrahedron is formed, and even larger cations with a ratio of 0,414 will result in an octahedron.

The ratio between Si and O is 0,30, meaning that for SiO<sub>2</sub>, a tetrahedron is formed. For this to be, with respect to valences, each of the oxygen atoms must be connected to two silicon atoms at once, while the silicon form bond with four oxygen atoms each. Due to the large repellant forces between the silicon ions, the tetrahedrons are only connected where the distance between them is largest, that is at the corners through the oxygen. As a rule; all glasses with SiO<sub>2</sub> is structured this way [1].

The type of bond between the atoms are also of importance. Covalent and ionic bonds can appear in mixed proportions. Due to this mix, a range of bond strength exist, and thus a model has been designed by Dietzel. This model introduces field strength [1]. Field strength is expressed as:

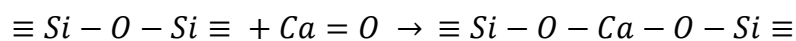
$$F = z_1 z_2 e^2 / a^2 \quad (1)$$

Where F is field strength, e is elementary charge and a is the sum of the ion radii, and z<sub>1</sub> and z<sub>2</sub> are the valences. In a system where all the anions are the same, as for example glass only containing oxides, the field strength is proportional with z/a<sup>2</sup>. Covalent bonds correspond to the highest bond strength, while ionic corresponds to the weakest. Mixed bonds correspond a whole range between them. For network formers, the ion radii is small and the coordination number is usually 3 or 4, which corresponds to an intermediate field strength. Network modifiers usually correspond to a low field strength. The influence of alkali elements like Na<sub>2</sub>O as previously, mentioned to break the bonds between the cation and the anion in the network former, as shown by reaction below [1]:



The new non-bridging oxygen bonds are mostly ionic, and they are much weaker then their network forming counterparts. The Na<sup>+</sup> ion favor a coordination number of 6. This is fulfilled in the more open places in the network, where it is bonded to the adjacent bridging oxygen atoms [1].

Alkali earth oxides is an addition found in most glasses. The most common being CaO, which incorporates in very much the same way as the alkali elements:



The bond between the  $\text{Ca}^{2+}$  and the oxygen is stronger than the bond between oxygen and  $\text{Na}^+$ , and thus the two non-bridging oxygen connected to  $\text{Ca}^{2+}$  will still maintain a bond. Systems only containing alkali oxide and  $\text{SiO}_2$  have a strong tendency to crystallize and thus are usually not used for glass making. On the other hand, adding a third alkali will considerably facilitate glass formation. Figure 3 shows the network of the soda-lime silica glass [1].

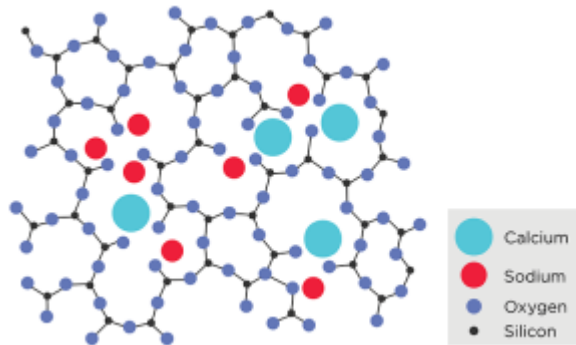


Figure 3: The network of soda -lime -silica glass. The network formers Ca and Na ions break the Si-O bonds [4].

Magnesium oxide,  $\text{MgO}$  can, due to its ion radius of 0,078 nm, function as either a network former or a network modifier depending on the amount of the amount of alkali ions present in the glass [1].

The influence of  $\text{Al}_2\text{O}_3$  is also of importance in soda-lime-silica glass. The radii of  $\text{Al}^{3+}$  is 0,057 nm resulting in it lying between coordination number 4 and 6. If added in pure silica glass, it will take on the form of  $\text{AlO}_6$  and thus function as a network modifier, however in soda-lime silica glass it will take on the form of  $\text{AlO}_4$  due to the opportunity to create coordination with the non-bridging atoms. The  $\text{Al}^{3+}$  ion is connected to the  $\text{Na}^+$  ion closing the separation, and thus strengthening the glass structure.  $\text{B}_2\text{O}_3$  functions the same way as  $\text{Al}_2\text{O}_3$  by closing the separation created by the  $\text{Na}^+$  ion [1].

### 2.1.2. Glass melting and composition

The transformation of a batch of raw materials into a glass melt, consists of several steps. The first step is the releasing of gases. Initially any moisture in the glass will be evaporate, however the decomposition of carbonates, sulfates and nitrates will release a far larger amount of gas. These gases will cause stirring, which helps to create a homogeneous melt, however the large amount of bubbles will have to be removed. If the transformation into a liquid is sufficiently rapid, enough gas can be captured to cause foaming at the top of the melt [5].

Several compounds often are present in the glass, each serving their own purpose. Five categories based on their purpose is; *Glass formers, flux, property modifier, colorant and fining agent*. Glass formers are the main component of the glass, its role is being a network builder and makes up the largest portion of the glass. A common glass former is silica, however due to its high melting point, at 1720 °C its often desired to lower cost by lowering the melting temperature. For this reason, a flux is added to the mixture. The flux usually consists of alkali oxides and will significantly lower the melting temperature of glass; however, it will also have a detrimental effect of the mechanical properties of the glass. To remedy this effect, a property modifier is added, usually alkali earth metal oxides like CaO. Al<sub>2</sub>O<sub>3</sub> can also be used [5].

Colorants are used for giving the glass a specific color, while fining agents are used to remove bubbles from the melt by creating huge quantities of glass. Smaller bubbles will attach to these larger ones which will quickly be carried to the top [5]. Table 1 lists the different categories

*Table 1: The different purpose for different compounds in glass [5].*

<b>Category</b>	<b>Purpose</b>	<b>Common composition</b>
<b>Glass former</b>	Main network former.	SiO <sub>2</sub> , B <sub>2</sub> O <sub>3</sub> , P <sub>2</sub> O <sub>5</sub>
<b>Flux</b>	Reduce melting temperature.	Na <sub>2</sub> O (Soda), PbO
<b>Property modifier</b>	Improve mechanical and chemical properties of the glass.	Alkali earth, transition metal oxides, Al <sub>2</sub> O <sub>3</sub>
<b>Colorant/Decolorants</b>	Add or remove color from the glass	Fe <sub>2</sub> O <sub>3</sub> , SO <sub>3</sub> , Gold, Silver, Copper.
<b>Fining agents</b>	Removing bubbles from the melt.	NaCl, CaF <sub>2</sub> , NaF, Na <sub>3</sub> AlF <sub>6</sub>

### 2.1.3. Heat transport

There are two different ways heat can be transported in glass; either through heat conduction or heat radiation. Which one is the most dominant is dependent on temperature. Heat conduction is most dominant at low temperature below 300 °C. At higher temperatures radiation becomes the most dominant [1]. Total heat transportation is expressed through the equation (2):

$$\lambda_{\text{eff}} = \lambda_c + \lambda_r \quad (2)$$

Where  $\lambda_{\text{eff}}$  is total effective thermal conductivity,  $\lambda_c$  is pure thermal conductivity and  $\lambda_r$  is heat radiation. Specific heat will obviously be influenced by the different components in the glass.

Formula (3) from Sharp and Ginther can be used to determine the specific heat capacity in a temperature range between 0 and 1300 °C [1].

$$c_{p,0-r} = \frac{T \sum a_i p_i + \sum c_i p_i}{0,00146T+1} \quad (3)$$

Where T is temperature,  $c_p$  is specific heat capacity.  $P_i$  is the fraction of each component. The two consonants  $a_i$  and  $c_i$  is shown I table 2 for different compounds

Table 2: a and c consonants for different compounds [1]

Compound	a*10 <sup>8</sup>	C*10 <sup>6</sup>
SiO <sub>2</sub>	468	1657
Na <sub>2</sub> O	829	2229
CaO	410	1709
MgO	514	2142
Al <sub>2</sub> O <sub>3</sub>	453	1765
B <sub>2</sub> O <sub>3</sub>	598	1935
Fe <sub>2</sub> O <sub>3</sub>	380	1449

## 2.2. Glasswool production and recycling

This section will explain the melting process of glass in glass wool production, as well as the challenges regarding recycling of glass wool waste. First part will explain what glass wool is and how the glass wool waste differs from the ordinary glass wool. While the second and third part will explain the recycling and production process.

### 2.2.1. Glass wool and glass wool waste

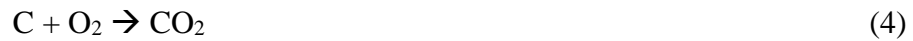
Glass wool contains thin fibers of soda-lime-silica glass. The fibers are used for insulation material in buildings. An carbon based organic binder is added to the fibers to give better mechanical properties, in addition a small amount of oils is added to reduce dusting. The normal amount of organics in the wool usually constitute to around 5-10wt%. The glass wool is white before the addition of the organic binder and turn yellow/brown after the binder has been added and cured [6].

During the production process a large amount of glass wool waste is produced. This waste has a significantly larger amount if organic content then the normal glass wool product, usually



ranging between 15-20 wt%. In addition, a large amount of water is present in the waste, and despite being pressed, the waste can contain between 30-70 wt% water [6].

If the glass wool waste is exposed to elevated temperatures the organic content will start to combust. This reaction will release heat into its environment. The carbon will burn after the reaction (4) and release heat and CO<sub>2</sub> gas into the environment:



One gram of carbon will release 92 kJ of energy if fully combusted [7].

Before the waste can be incinerated or melted, any moisture must be evaporated. This will significantly increase the amount of heat necessary to melt the waste. If 100 gram of glass wool containing 50 wt% water is heated, 37kJ of energy needed to remove all the moisture.

The glass wool is as mentioned made of soda-lime-silica glass. In addition, other oxides including; Al<sub>2</sub>O<sub>3</sub>, MgO and BO<sub>3</sub> is added in small amounts to modify its properties. A table of the raw materials used to make soda-lime-silica glass is shown in table 3

*Table 3: Raw materials used for soda-lime-silica glass*

<b>Name</b>	<b>Composition</b>
<b>Sand</b>	SiO <sub>2</sub> , Al <sub>2</sub> O <sub>3</sub> , Fe <sub>2</sub> O <sub>3</sub> , MgO, K <sub>2</sub> O
<b>Soda</b>	Na <sub>2</sub> CO <sub>3</sub>
<b>Lime</b>	CaCO <sub>3</sub>
<b>Dolomite</b>	CaMg(CO <sub>3</sub> ) <sub>2</sub>
<b>Neobor</b>	Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub>
<b>Feldspar</b>	NaAlSi <sub>3</sub> O <sub>8</sub>

A typical composition of soda-lime silica glass is found in table 4

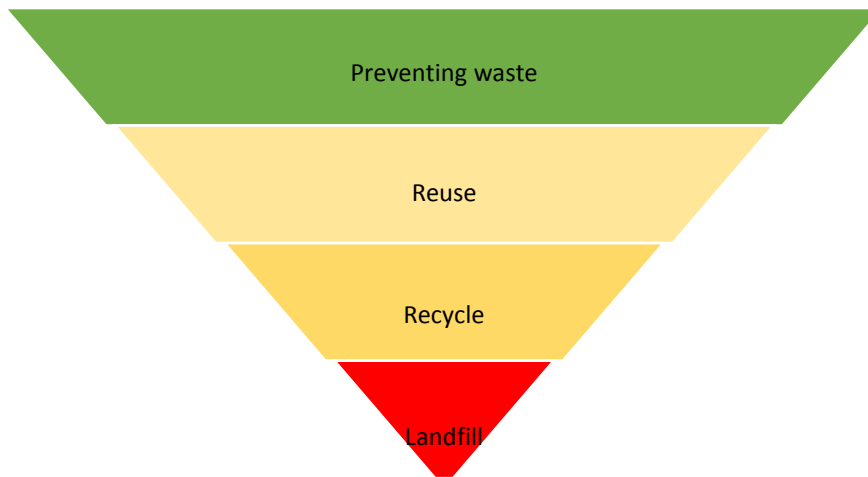
Table 4: Typical composition for soda-lime-silica glass

	Min. Wt %	Max Wt%	Wt% Range
<b>SiO<sub>2</sub></b>	66,2	74,7	66,2-74,7
<b>Na<sub>2</sub>O</b>	12,88	17,30	12,88-17,30
<b>CaO</b>	9,16	13,40	9,16-13,40
<b>K<sub>2</sub>O</b>	12,88	17,30	12,88-17,30
<b>Al<sub>2</sub>O<sub>3</sub></b>	1,25	2,5	1,25-2,5
<b>MgO</b>	0,91	1,51	0,91-1,51
<b>Fe<sub>2</sub>O<sub>3</sub></b>	0,11	0,12	0,11-0,12
<b>BaO</b>	0,8	0,17	0,8-0,17
<b>SO<sub>3</sub></b>	0,08	0,22	0,08-0,22

As can be seen in both table 3 and 4, in addition to the oxides already mentioned, the soda-lime-silica glass usually contains small amounts of trivalent iron oxides and sulphite. This usually is unintentionally brought into the mix by the sand, and is regarded as an impurity. If the iron oxides reacts with a reduction agent, like carbon, it will result in both an additional release of CO<sub>2</sub> as well as the presence of divalent iron oxide FeO in the glass which can have detrimental effect on the parts melt by lowering the conductivity in comparison with the rest of the melt, causing inhomogeneous melting [13].

### 2.2.2. Glass wool recycling

Recycled glass is in demand as a raw material for many production processes, in particular the glass wool industry itself. However, for the glass to be of any use, any organic content must be removed prior to melting. Several strategies have been developed to counter this challenge. Figure 4 shows a pyramid illustrating the preferred ways to deal with waste from the production process [8].



*Figure 4: The Pyramid shows the preferred ways to deal with waste. Preventing waste being the optimal route, while dumping at landfill is the least desired solution [10].*

Preventing waste to occur in the first place is unsurprisingly the most desired solution. The second most preferred way is to reuse the waste without significant processing like remelting the waste. This is done to a certain degree with other types of waste that does not contain the same amount of organic content. This is however, not an option for the waste in focus in this thesis, since the fibers are not of uniform size, and the amount of organic content is too large [6]. The strategy that will be discussed in this paper, is the recycling of the waste. This option refers to the strategy where the waste is to be used as raw material in either fiber production or in the production in some alternative process. To use the waste for fiber production is the more preferred option when possible, since outside industry might be reluctant to change their raw material batch. Thus, the strategy that this thesis will focus on is the recycling of glass wool waste by placing it back in the glass melting furnace along with the rest of the raw materials.

### 2.2.3. Glass melting process

In the industry, the glass is melted in a furnace. The raw materials are gradually added as fines at the top of the melt. A carpet of unmelted raw material is always present at the top of the melt. New raw material is added at the same pace as the glass at the bottom of the layer is melted and move out of the furnace, preserving a continuous thickness of the carpet. Figure 5 shows a sketch of the melting furnace with the glass powder carpet at the top.

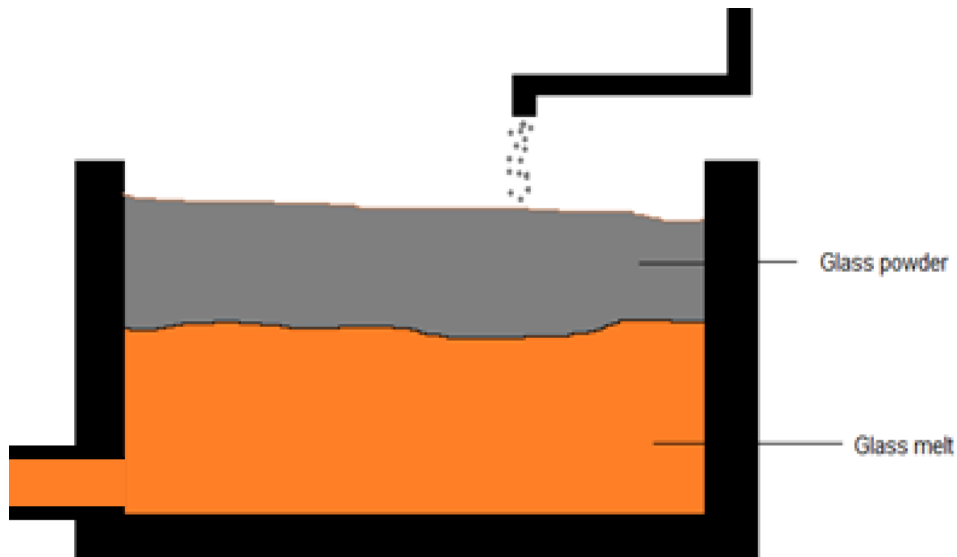


Figure 5: Glass melting furnace with a powder carpet at the top. New powder is continuously added as the bottom part of the carpet melts.

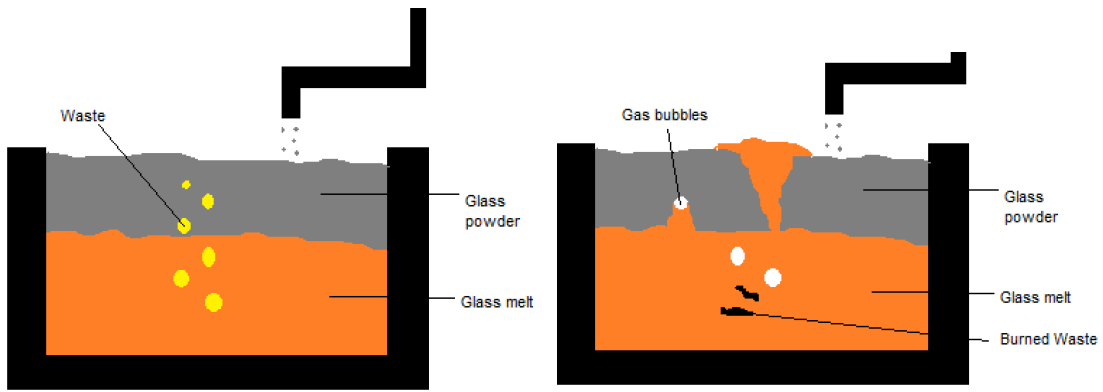
The carpet plays an important role in preserving the heat of the melt. The thickness of the carpet is used to controlling the effect of the furnace. A thicker carpet will preserve more heat and thus increase the effect [9]. However, if a sufficient amount of gas is released in the melt, the bubbles can burst through the carpet, creating small volcanoes at the top of the melt, disturbing the control of the heat in the melt [6]. This is illustrated in figure 6. This phenomenon is apparent when large amount of glass wool waste is added directly to the melt. If carbon come in contact with the melt it will react with CO<sub>2</sub> gas from the carbonates [10]:



The CO will further react with oxygen from iron oxides:



Excess amounts of gas can have a detrimental effect on the carpet. If the waste reaches the melt before the organic content is burned off, then the release of CO<sub>2</sub> can damage parts of the carpet, as shown in figure 6.



*Figure 6: a) Glass wool waste is added in the melt b) Released gas from the waste creates bubbles, destroying the carpet*



### 3. Experimental setup & Method development

This section will describe the equipment used as well as the set up for the experiments performed. First a list of the equipment used will be shown. Then it will be explain how the experiments was conducted, as well as explain the equipment used. The biggest part of this chapter is the simulation experiments, where the whole method development is explained in detail. Temperature measurement is also included for the simulations for convenience since it is closely related to how the experiments was conducted. The relevant measurements will still be included in the result and discussion chapter.

#### 3.1. Equipment

This section will describe all the equipment used in the experiments and the relevant theory on how they operate. This includes furnaces, microscopes, and raw materials.

- LabStar calcining furnace
- Struers Tegramin-30; polishing mashine
- Zeiss Ultra scanning electron microscope
- Epoxy
- IF 75 induction furnace
- Graphite crucible
- Graphite tubes
- Thermocouples
- Soda-lime-silica glass raw materials
- Carbon coating machine
- Glass wool waste

#### 3.2. Experimental procedure

In this section the experimental procedure is explained. The first section describes the incineration experiment in atmospheric conditions, while the latter will go into detail about the glass furnace simulation experiments. Since the simulation was partly about developing a

method, each experiment was done differently, hence the need to describe what was done in each experiment where the procedure has changed. The result of the temperature measuring is included as well, since it is closely related to how the experiments was performed.

All the glass wool waste used in experiments was dried beforehand in an oven at 80-100 °C, for 48 hours. 5 Batches of waste was put in the furnace and their weight was measured before and after to determine the amount of water initially in the samples. When a sample in this thesis is described as an untreated sample, while it implies that it has not been exposed to any furnace experiments, it still has been removed from any water.

### 3.2.1. Incineration in atmospheric conditions

Glass wool waste was obtained from the production plant at GLAVA Stjørdal. As with all the glass wool, it was made from soda-lime -silica glass with a certain amount of organic content. The glass wool was weighted before and after drying to estimate amount of moisture in the wool. A picture of dried waste is shown in figure 7.



*Figure 7: Glass wool waste after being dried in an oven for 48 hours.*

16 samples of glass wool were placed in a steel container and heated at different temperatures at different times. The temperatures used was 500, 600, 900 and 1000 °C, and the times used was 3, 7, 15 and 20 minutes. For example; four samples were heated at 500, each at a different amount of time, with the same procedure for the other temperatures. The samples were weighted before and after to determine the amount of organic matter burned away in the furnace. Table 5 show the different samples and their respective heat treatment. The samples exposed to the heat treatment for 20 min as well as the one subjected to 500°C for 3 minutes, and an untreated sample were sent to SintefMolab for LECO analysis to determine the amount of carbon left in the samples.



Table 5: The sample number and heat treatment in the incineration experiments

Sample number	Temperature (°C)	Time (min)
1	500	3
2	500	7
3	500	15
4	500	20
5	600	3
6	600	7
7	600	15
8	600	20
9	900	3
10	900	7
11	900	15
12	900	20
13	1000	3
14	1000	7
15	1000	15
16	1000	20

### 3.3. Glass furnace simulation & Method development

The general procedure of the glass furnace simulation is described below. Variations were made from experiment to experiment, but all followed the same general layout. The goal was to have a create a melt with a carpet of unmelted raw material on top, as in the furnace in the industry, and find a technique for measuring the temperature gradient in the carpet layer.

A graphite crucible with a diameter of 15cm and height of 40 cm was filled with 5 cm with raw material for soda-lime-silica glass production. The raw material used was acquired from the production plant at GLAVA Stjørdal. The material had a particle size ranging from the size of a sand corn to approximately with a diameter of 1 cm. A picture of a bucket containing the sample material is shown in figure 8.



*Figure 8: Raw material as a powder from the production plant in Stjørdal.*

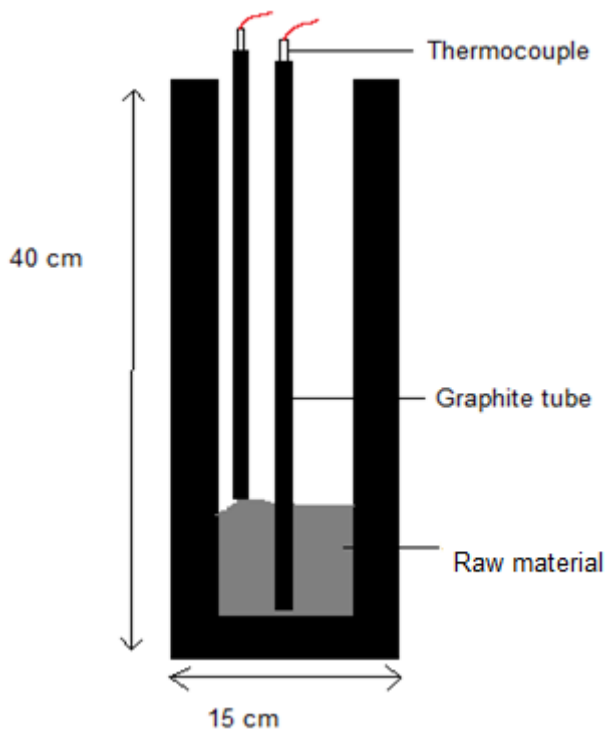
Two thermocouples placed in graphite tubes was placed in the crucible. The raw material was melted in an IF 75 open induction furnace. This melt was supposed to represent the melt in the glass furnace. A picture of the furnace is shown in figure 9.



*Figure 9: A picture of the IF 75 induction furnace.*

After stable melting temperature was obtained, 20 cm of raw material was added in the crucible. This layer was to represent the carpet layer in the glass furnace. The first thermocouple stood still at the bottom of the melt, while the second was gradually moved upwards to measure the temperature at different places in the carpet layer above the melt. The temperature was measured with 2 cm intervals to get the temperature gradient. The procedure can be explained more easily by separating it to several steps shown in figure 10-12:

*Step 1:* Place a graphite tube in the middle of the crucible, and one closer to the crucible walls. Add 5 cm of raw material in the crucible.



*Figure 10: Step 1; Add powder raw material in the crucible*

*Step 2:* Heat the raw material to a temperature of approximately 950 °C and keep the temperature stable.

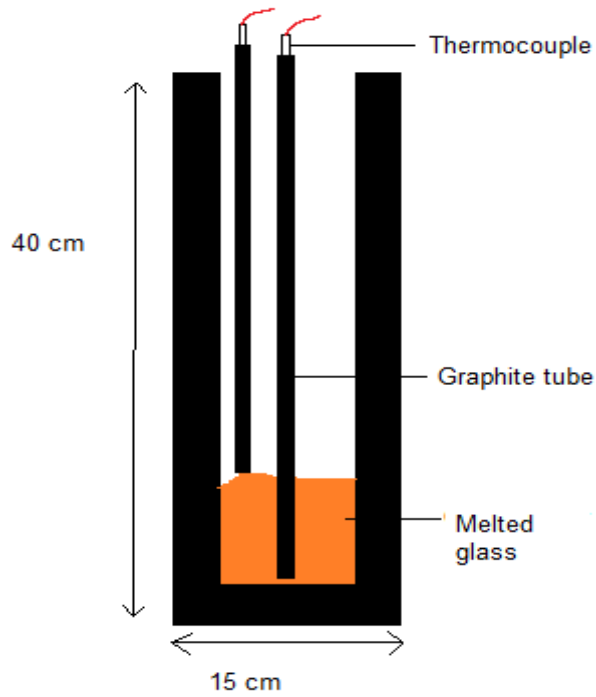


Figure 11: Step 2; melt the raw material and keep the temperature stable

Step 3: Add 20 cm of raw material to the crucible.

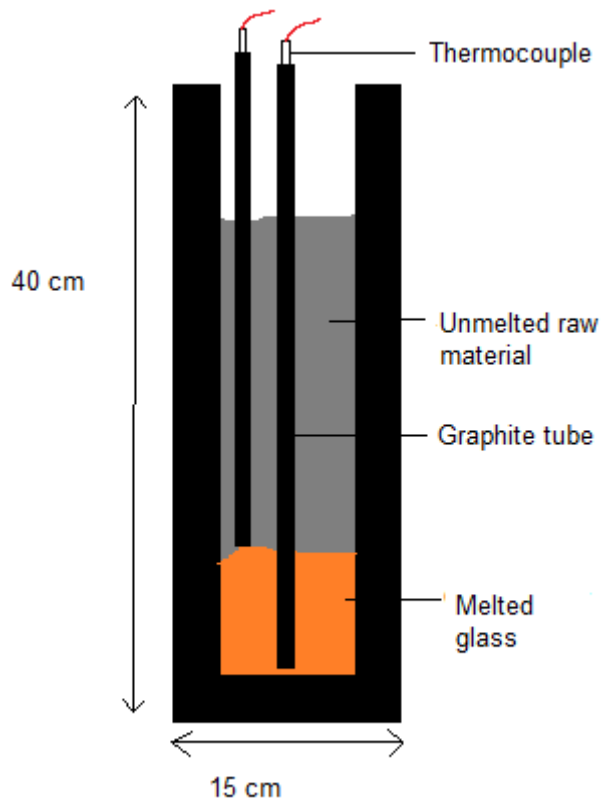


Figure 12: Step 3; Add raw material over the melt

*Step 4:* Keep the temperature in the bottom stable and measure the temperature with 2 cm intervals in the top layer of the crucible.

*Step 5:* Let the sample cool down, before further analysis.

The details of the procedure change over time, and thus each experiment is explained separately. Table 7 shows every simulation performed. For convenience, and for easier understanding of the explanation of the process, a table of the expression and their meanings are presented in table 6. A total of approximately 5kg of raw materials was used in each simulation.

*Table 6: The name and expressions used for the different parts in the simulations.*

<b><u>Name</u></b>	<b><u>Meaning</u></b>
<b>Raw material</b>	The raw materials used for glass production, in the form of fines as shown in figure 8
<b>Melt</b>	The first 5 cm layer of raw material added at the bottom. The part where the temperature is kept stable at ca. 950°C
<b>Carpet layer</b>	The 20 cm of raw material added after the melt is at stable temperature. This is the part where the temperature gradient is measured.
<b>Waste</b>	Glasswool waste as shown in figure 7, dried beforehand.
<b>Thermocouples</b>	used for measuring temperature
<b>Alumina tubes</b>	Thin tube holders for thermocouples
<b>Graphite tubes</b>	Tubes used to contain thermocouples and alumina tubes.

Table 7: A list of the simulations performed

NUMBER	NAME	DATE	WITH GLASS WOOL	COMMENT
1	Simulation 1	02.03.2018	No	
2	Simulation 2	06.03.2018	No	
3	Simulation 3	13.03.2018	Yes	
4	Simulation 4	23.03.2018	No	
5	Simulation 5	06.04.2018	No	Reused crucible
6	Simulation 6	06.04.2018	Yes	No measurment
7	Simulation 7	17.04.2018	No	
8	Simulation 8	17.04.2018	yes	No measurment
9	Simulation 9	19.04.2018	No	
10	Simulation 10	19.04.2018	Yes	
11	Simulation 11	02.05.2018	Yes	
12	Simulation 12	02.05.2018	Yes	
13	Simulation 13	23.05.2018	Yes	No epoxy
14	Simulation 14	23.05.2018	Yes	No epoxy

### 3.3.1. Simulation 1&2

Stable temperature was obtained at approximately 1000 °C, before the second batch of glass raw material was introduced. The first thermocouple was placed in the center of the crucible, and the second was placed near the crucible wall. The temperature gradient was only measured once. The graphite tubes containing the thermoelement was moved, instead of only moving the thermocouple. Figure 13 shows the temperature gradient. It can be seen that as the thermocouple being moved upwards from the melt, the temperature decreases. The point when the temperature gradient measurement starts is highlighted by a red line. A green line represents the time when the carpet layer was added. The temperature is from 892 °C right above the melt and gradually decrease to 361 °C at the top.

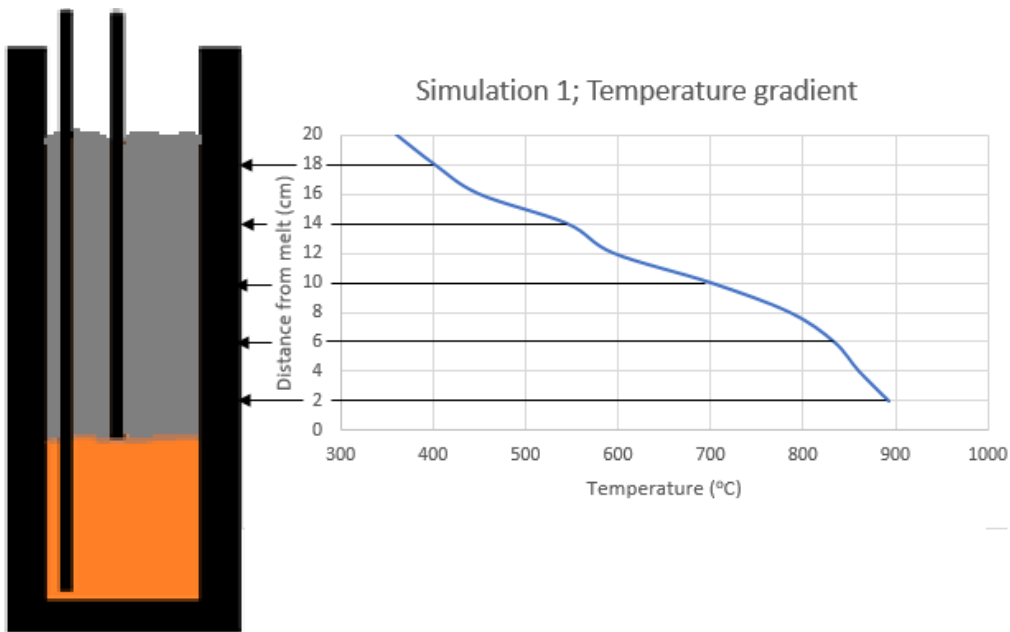


Figure 13: Temperature gradient measured above the melt

Figure 14 shows the temperature for both thermocouples as a result of time. Serie 1 represents the thermocouple placed at the bottom of the crucible, while Serie 2 represent the thermocouple that was moved upwards in the carpet layer. The gradual decline after 55 minutes is due to the thermocouple being moved. It can be seen that the temperature decrease slightly after the second layer of raw material is added

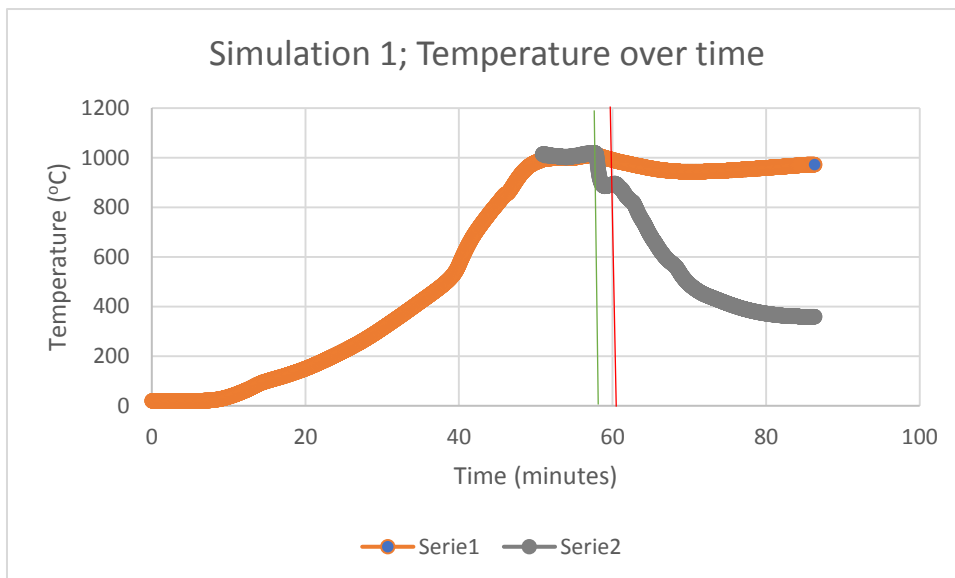


Figure 14: Temperature for both thermocouples over time

Temperature gradient from simulation 2 is shown in figure 15. Temperature ranges from 303 °C right above the melt to 201 °C at the top.

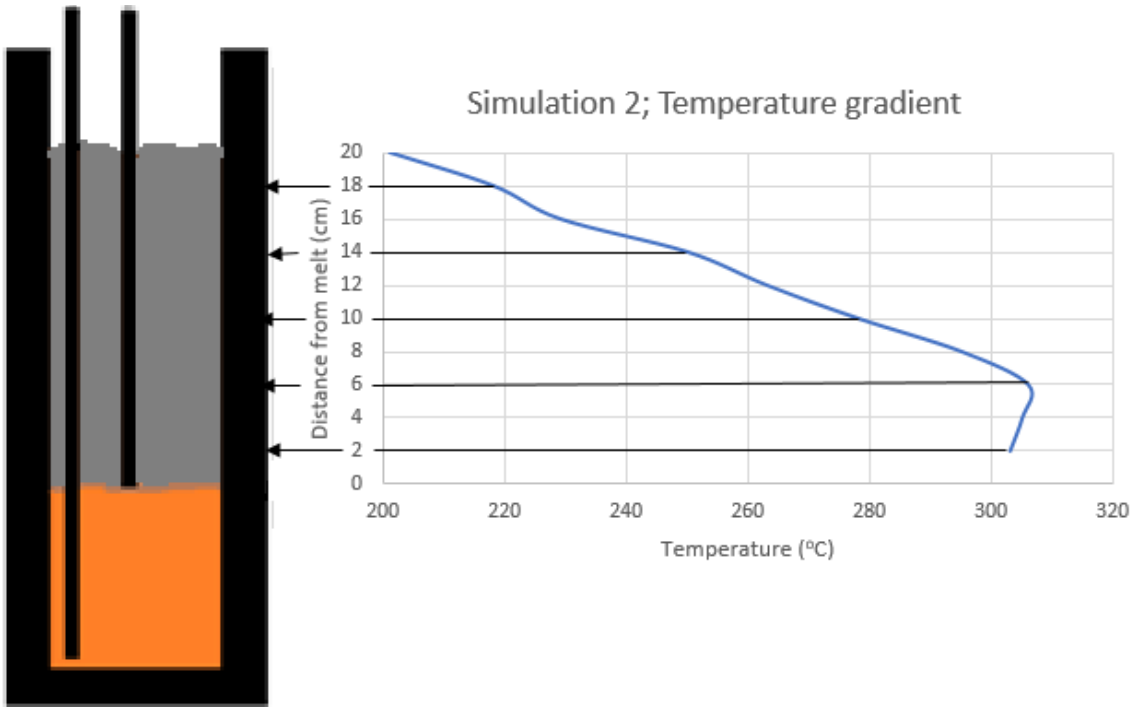


Figure 15: Temperature as a result of distance from the melt

Figure 16 shows the temperature over time for simulation 2. Serie 1 is the temperature at the bottom of the melt, while Serie 2 is the temperature in the carpet layer. As with simulation 1 the temperature in the melt decrease when the second layer is added, however after some time it increase before becoming stable at 65 minutes.



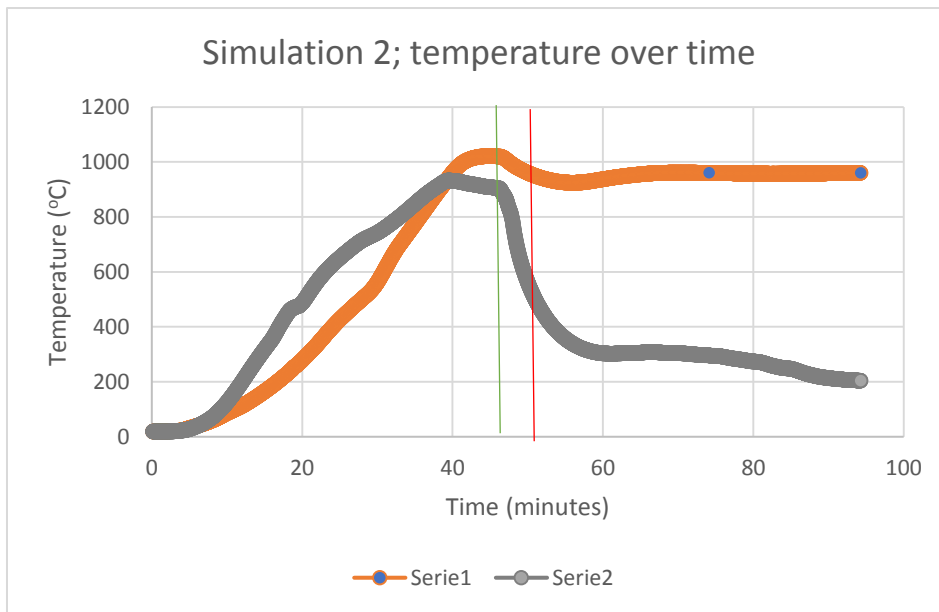


Figure 16: Temperature for both thermocouples over time

As mentioned and shown in both experiments the temperature in the melt decreased when the carpet layer was added. The temperature gradient measurement was conducted right after the addition of the layer, before the melt had the time to stabilize itself. Since no other measurements was taken, the results from these experiments does not represent those conditions that was intended.

### 3.3.2. Simulation 3

This simulation was performed in the same way as simulation 1 and 2, with the addition of glass wool waste in the carpet. The glass wool was not previously mixed with the raw materials beforehand but placed in the crucible with the raw material. The amount of glass wool added was not noted. The setup is shown in figure 17

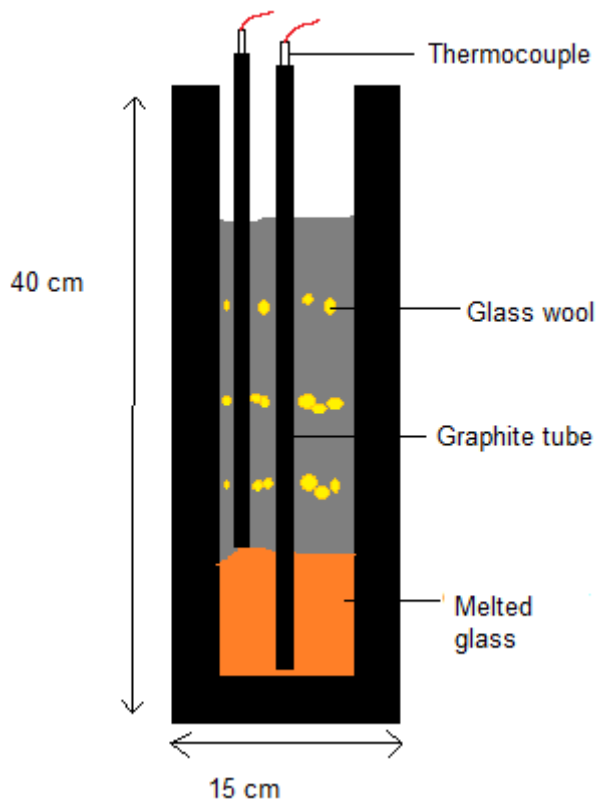


Figure 17: Simulation 3, performed with glass wool in layers within the carpet.

Figure 18 shows the temperature gradient. The temperature ranges from 937 °C at the top to 270 °C at the bottom.

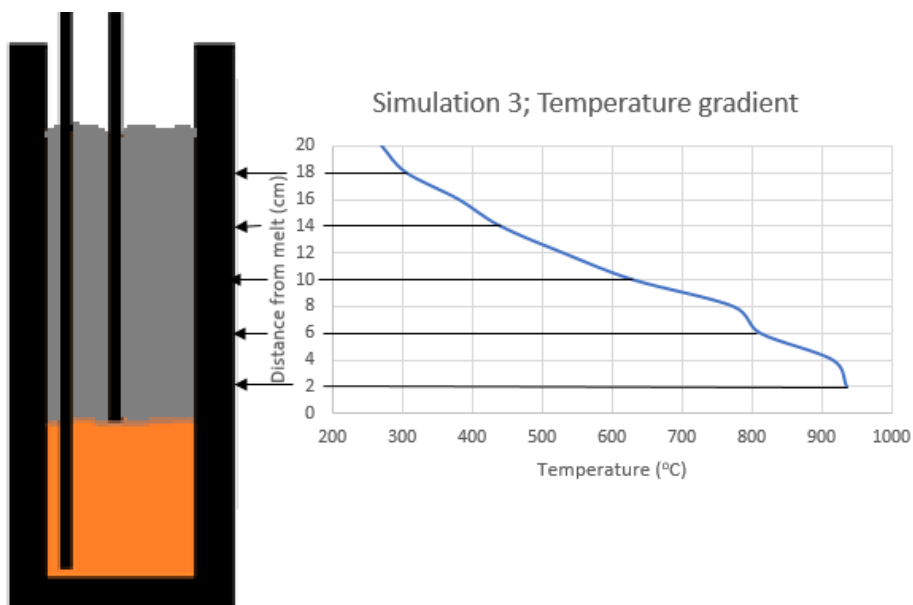


Figure 18: The temperature as a result of location

Figure 19 shows the temperature for both thermocouples as a result of time. Poor control of the temperature gave some strange result. The temperature in the bottom layer is lower than the temperature in the carpet layer before the temperature gradient measuring started. The graph also shows erratic behavior between 70 and 80 minutes due to the poor control over the thermocouple. Due to this, it became necessary to change how the thermocouple was moved, since moving the graphite tubing made precision difficult. Since the waste was added separately from the glass fines, the process of adding the carpet layer took a little bit longer time. This allowed the waste to be incinerated for a few seconds before being covered in raw material. Also, as with simulation 1 and 2, only one measurement was taken. Simulation 1,2 and 3 will not be included in further analysis since due to the unreliable results.

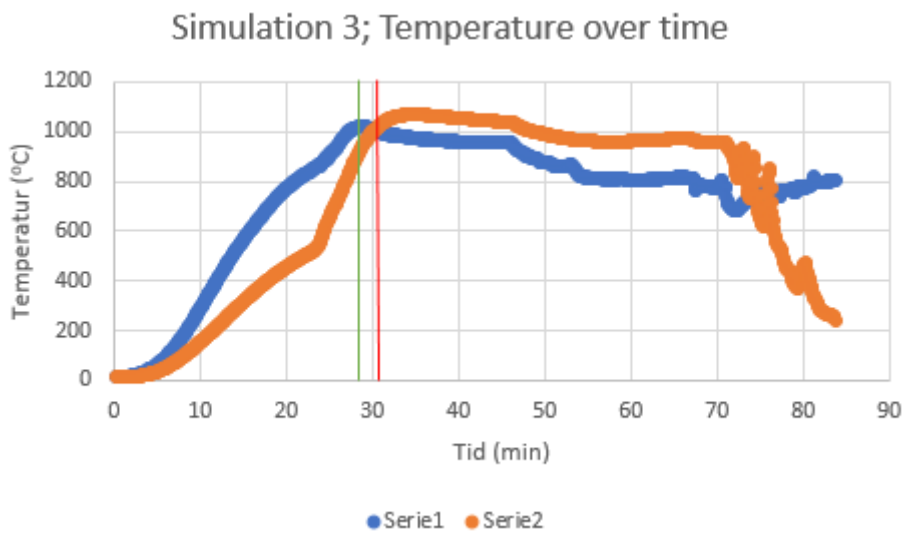


Figure 19: Shows the temperature as a result of time. Exact temperature is hard to determine erratic temperature measurement.

### 3.3.3. Simulation 4

Stable temperature was obtained at 950 °C. Instead of moving the graphite tubes, only the thermocouples incased in alumina tubes was moved, leaving the graphite tubes in its place. This resulted in much better mobility. The setup is shown in figure 20. In addition to the changes previously mentioned, two temperature measurements were conducted; one right after the second layer was added, and one after temperature in the bottom layer was stable. The second measurement is the one used in future graphs and comparisons.

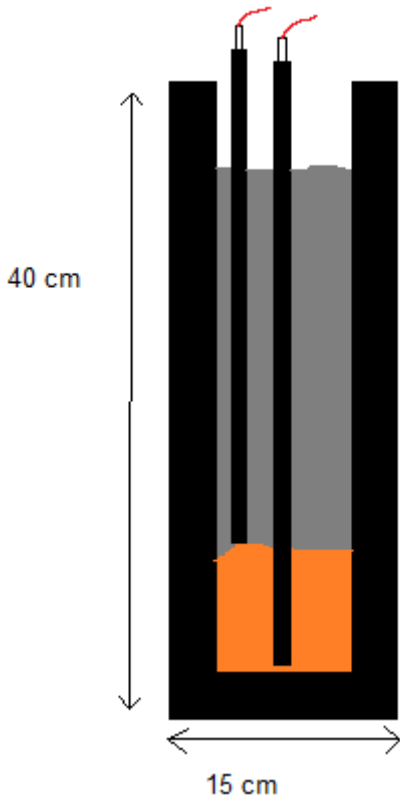


Figure 20: The set for the simulation

Figure 21 shows the temperature gradient as a result for distance from the melt. Two measurements were conducted. The temperature ranges from 903 °C to 661 °C for the second measurement.

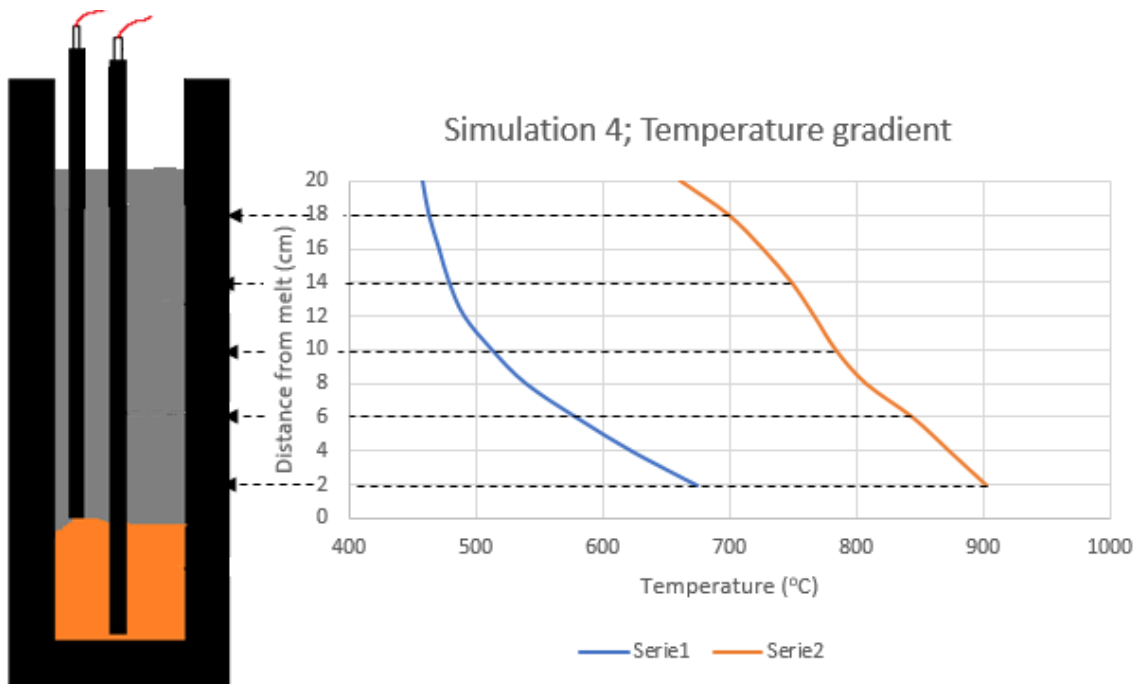


Figure 21: Temperature as a result of location in the crucible

Figure 22 shows temperature over time for both thermocouples. Two measurements were conducted. Temperature drops significantly when carpet layer is added after 60 minutes. Each downward slope for serie 2 represent a single measurement of the temperature gradient. It can be seen in figure 22 that the temperature in the carpet layer increases over time, and each temperature gradient shows a higher temperature than the previous one, thus in order to make the result comparable with the other simulations, better control of the time would be necessary.

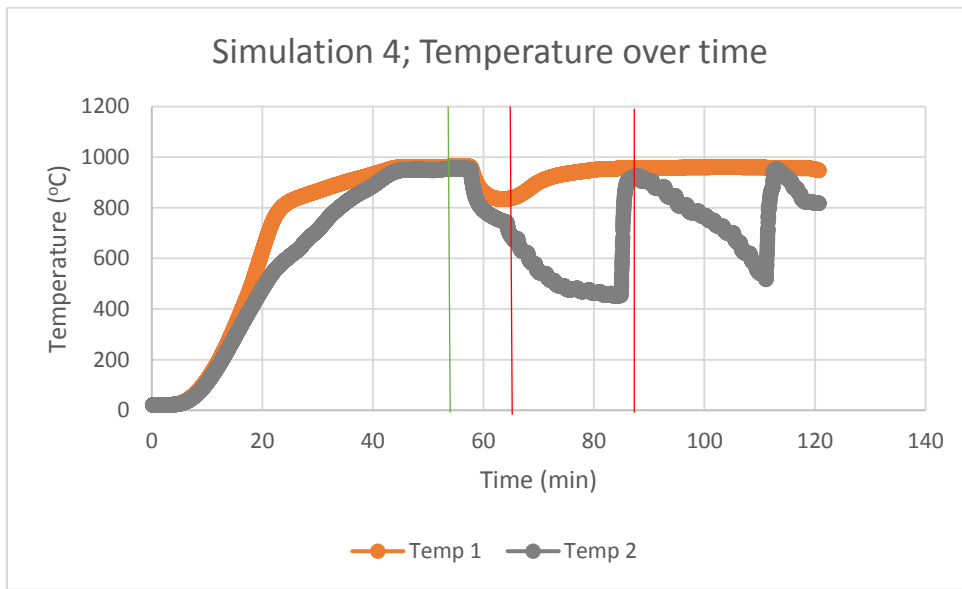


Figure 22: The temperature as a result of time for both thermocouples

### 3.3.4. Simulation 5, 7 & 9

These simulations were performed very much in the same way as simulation 4, with one crucial exception, since the temperature in the carpet layer is gradually increasing, as shown from simulation 4, better control over the timeframe was implemented in order for the results to be comparable. The first measurement of the first temperature gradient started 5 minutes after the top layer was added. The thermocouples stood at their measurement spots for two minutes before the temperature was noted and the thermocouple was moved. After the measurement of the first temperature gradient was concluded, five minutes passed before a new measurement started. In some cases, a third measurement was conducted, however this was done only to see how the temperature continued to increase. Simulation 5 were had a slightly different set-up then the rest. In this experiment, it was attempted to reuse a crucible from a previous experiment. The unmelted raw materials were removed, leaving only the melted glass in the crucible, acting as the first batch in the simulation. The level of the previously melted glass was higher than the added raw material in other experiments.

Figure 23 shows the temperature for the fifth simulation as a dependent on the distance from the melt. As with the previous simulations only the second measurement is represented here. Temperature ranges between 411 °C at the top and 711 °C right above the melt, for the second temperature gradient measurement.

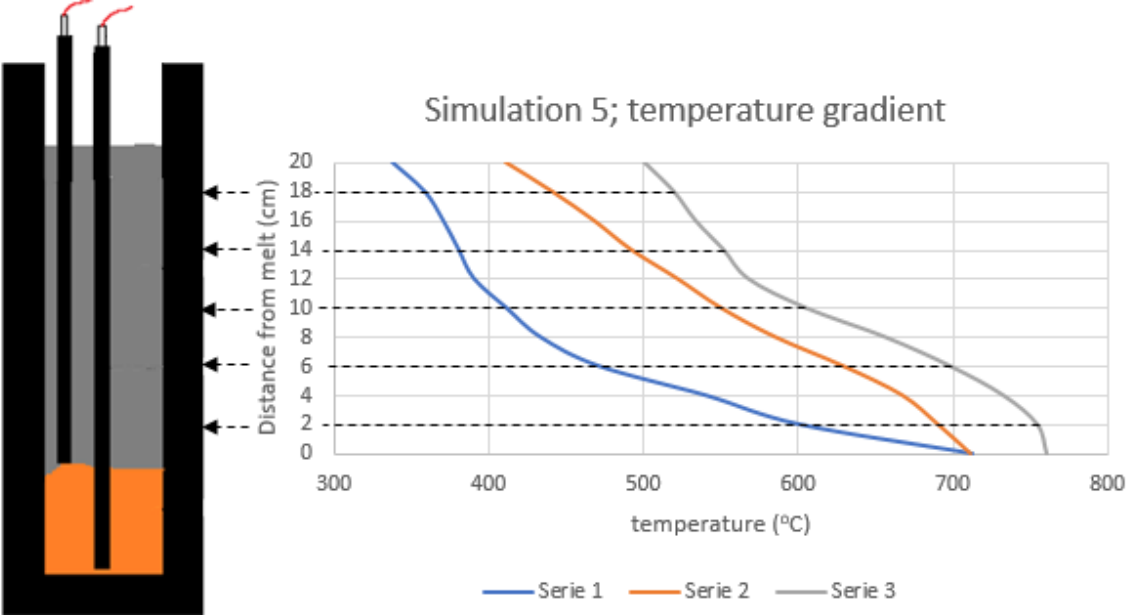


Figure 23: Temperature dependent on time.

Figure 24 shows the temperature as a dependence on time, three measurements were taken. The measured temperature shows the same behavior as with the previous experiments. However, the temperature did not significantly drop when carpet layer was added, however. The temperature gradient in the carpet layer increase steadily over time.

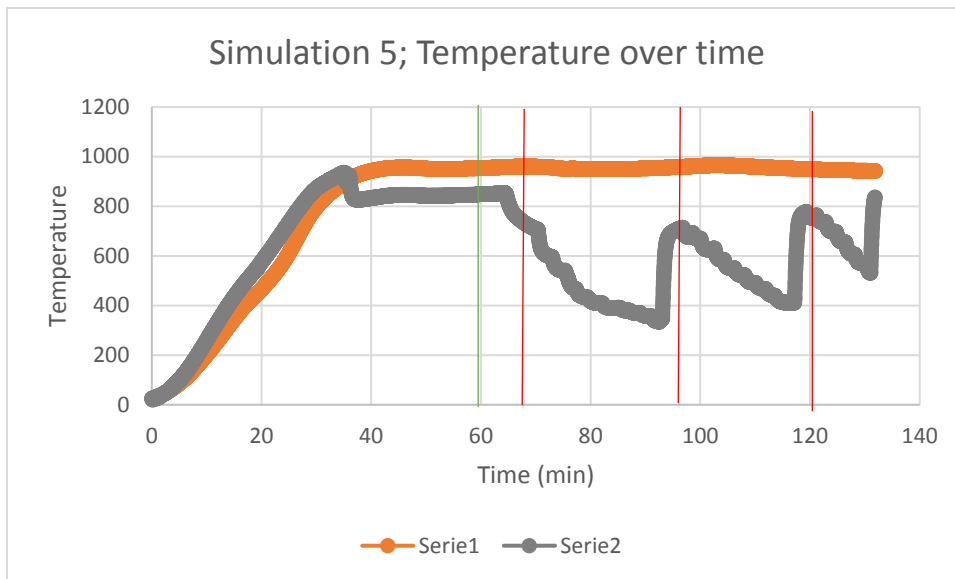


Figure 24: Temperature over time for both thermocouples

Temperature gradient for simulation 7 is shown in figure 25. The temperature ranges from 820 °C close to the melt to 520 °C at the top, for the second measurement. The third measurement showed erratic behavior, compared to the other measurements, with the temperature increasing and dropping rapidly.

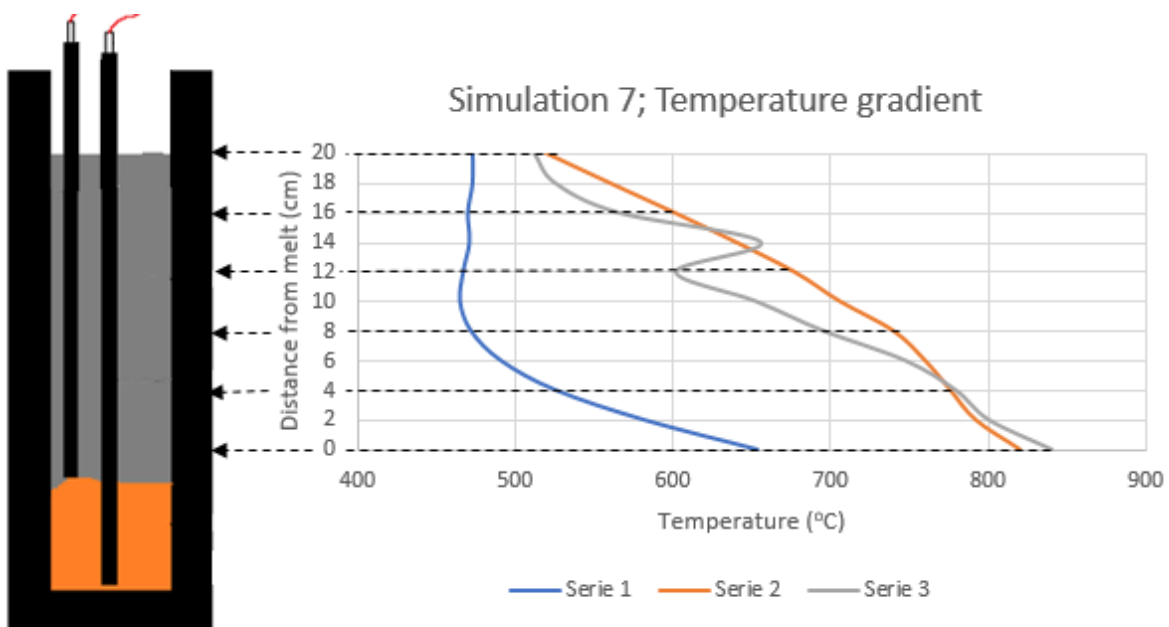


Figure 25: Temperature gradient for simulation 7.

Figure 26 shows the temperature as a result of time. Three measurements were taken. The temperature decreased almost 150 °C after adding the carpet layer.

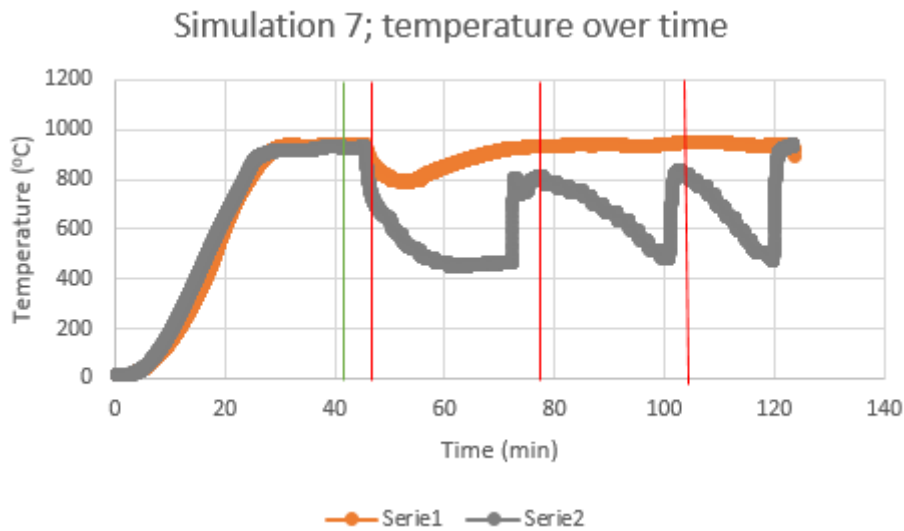


Figure 26: Temperature over time for both thermocouples

Figure 27 show the temperature as a result of distance from the melt. The temperature ranges from 506 to 790 °C for the second measurement.

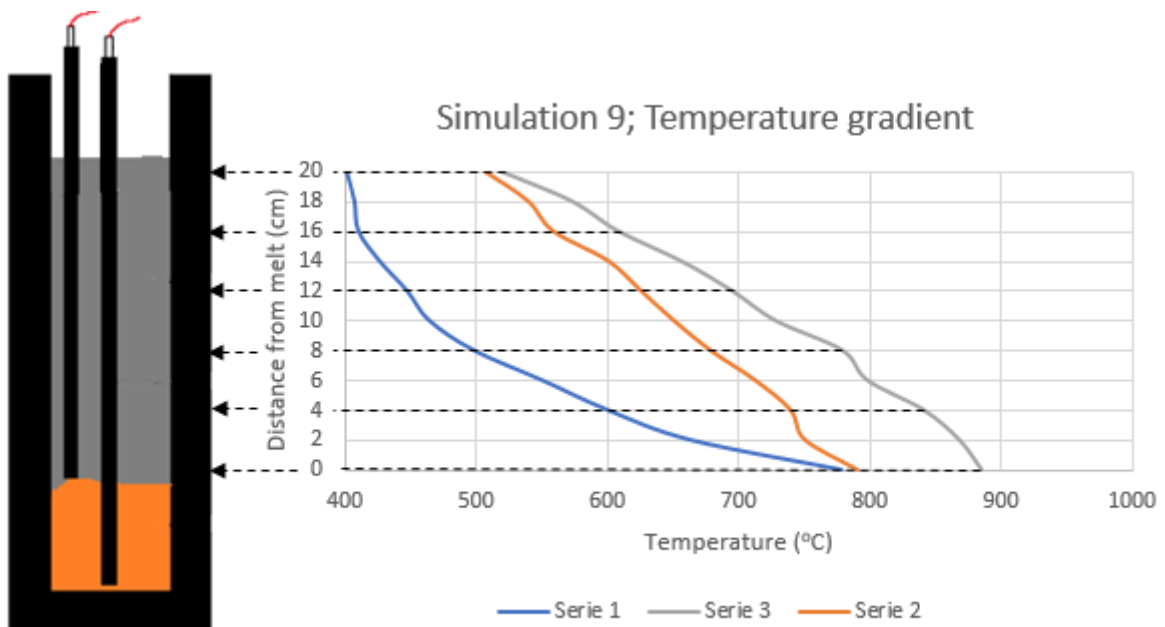


Figure 27: Temperature as a result of distance from the melt.

Temperature as a result of time is shown in figure 28. It showed similar behavior as the previous simulations. The temperature decreased by approximately 100 °C when the carpet layer was



added and rose to previous level after 15 minutes. The temperature gradient increased gradually during the whole experiment.

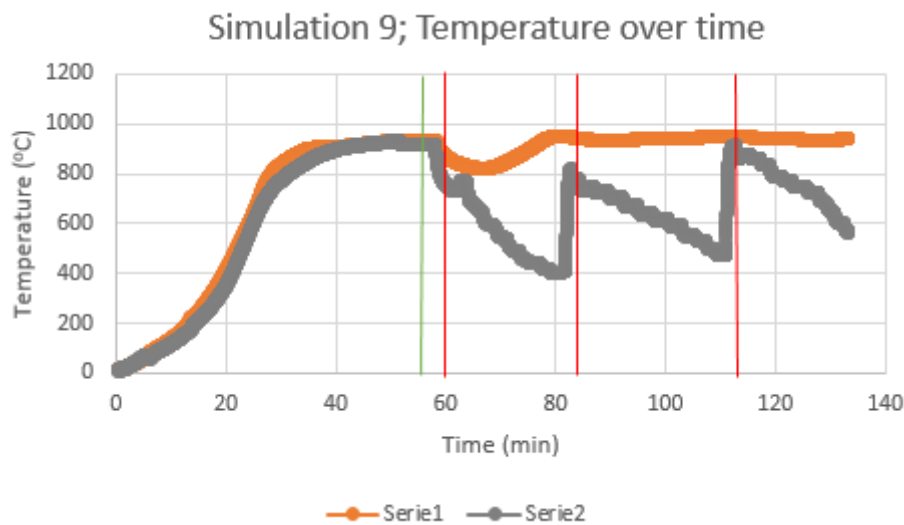


Figure 28: Temperature for both thermocouples as a result of time.

### 3.3.5. Simulation 6 & 8

In these experiments it was attempted to introduce glass wool with the carpet in a new way. The goal was to have a cylinder of wool in the middle of the crucible, surrounded by raw material. The procedure of adding the glass wool could be described in three steps. The procedure is also shown in figure 29-31

*Step 1:* After the temperature in the bottom layer is stable, a thin layer of raw material is added. At the top of the layer, a thin steel tube with a diameter of 5 cm is placed. A graphite tube is placed in the middle of the steel tube

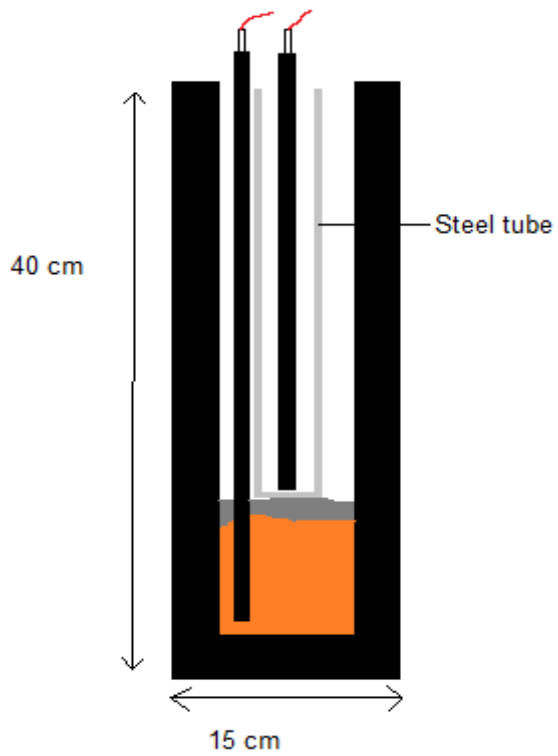


Figure 29: Step 1, place a steel tube in the crucible

Step 2: The steel tube is filled with glass wool waste, and the surroundings is filled with raw material.

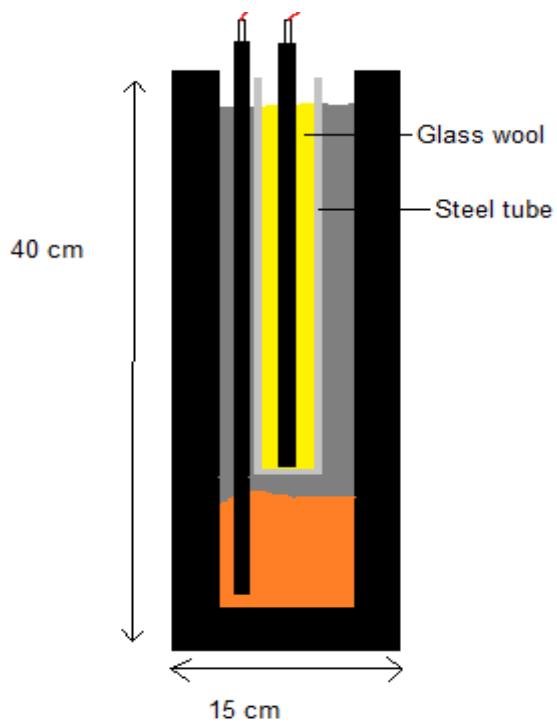
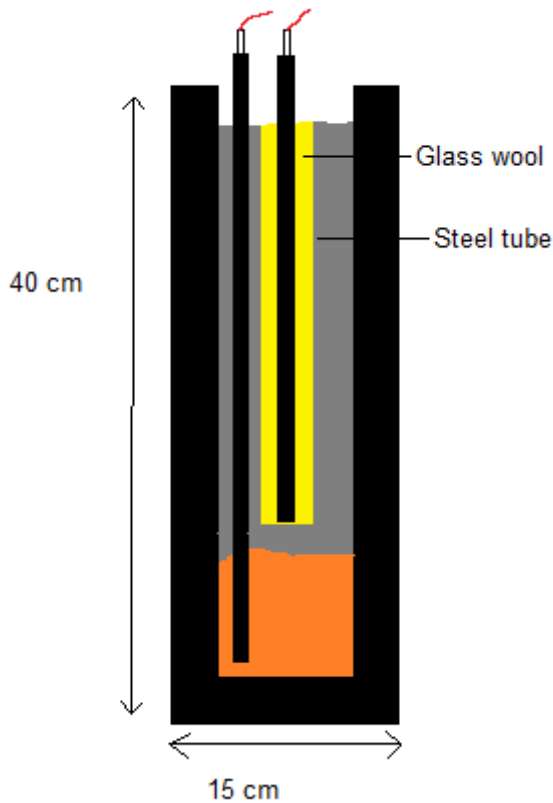


Figure 30: Step 2, add glass wool in the tube and raw material outside.

*Step 3:* The cylinder is removed leaving a cylinder of glass wool in the center of the crucible, surrounded by raw material. Place a thermocouple in the graphite tube in the center of the crucible.



*Figure 31: Step 3; remove the steel tube*

Afterwards the temperature gradient was to be measured in the glass wool by moving it the same way as done in previous experiments. However, no measurement was recorded from simulation 6 and 8. The steel tube in the center, containing glass wool waste sank into the melt thus making it impossible to remove it without removing all the waste with it. A thicker layer of powder between the tube and the melt did not solve this issue hence no temperature gradient could be measured.

### 3.3.6. Simulation 10, 11 & 12

These simulations were carried out the same way as simulation 5,7, and 9, with the exception that glass wool waste was mixed with the carpet layer before it was added to the crucible. 100g of waste was added in each simulation, which was the same type of waste used in the incineration experiments in chapter 3.2.1 The temperature was measured as done previously.

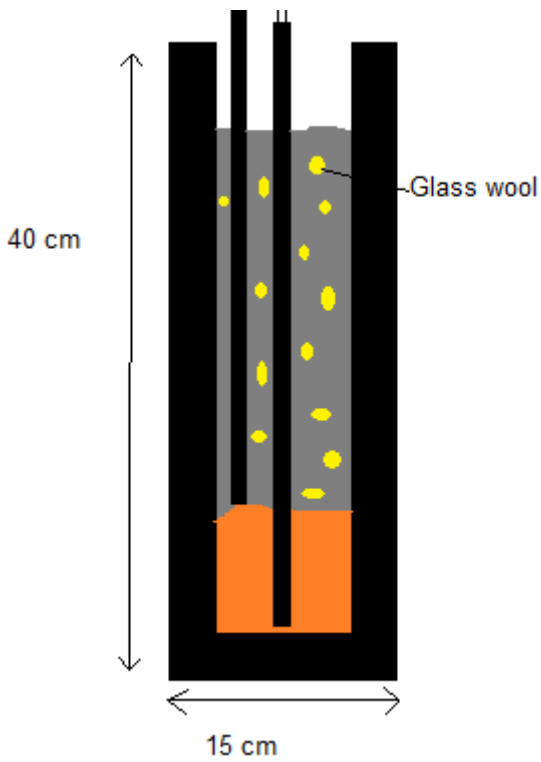


Figure 32: Glass wool randomly distributed in the carpet

Figure 33 shows the temperature as a result of distance from the melt. The temperature ranges from 550 to 850°C for the second measurement.

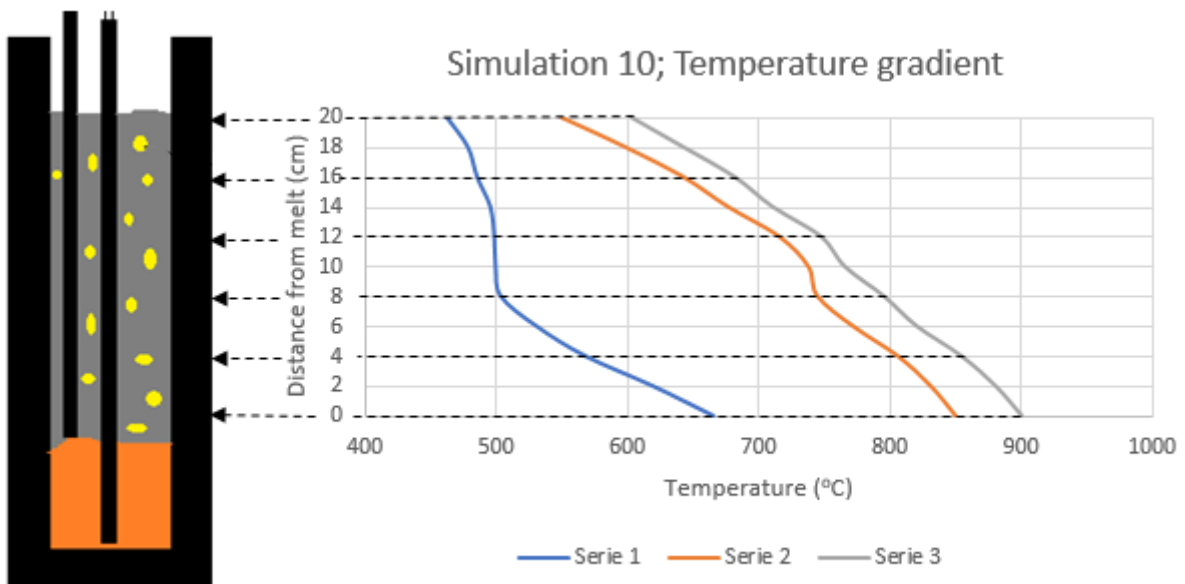


Figure 33: Temperature as a result of distance from the melt

Figure 34 shows the temperature over time for simulation 10. The temperature drops after adding the carpet layer was approximately 180 °C and was back to 960 °C after 20 minutes. The temperature increased over time in the carpet layer.

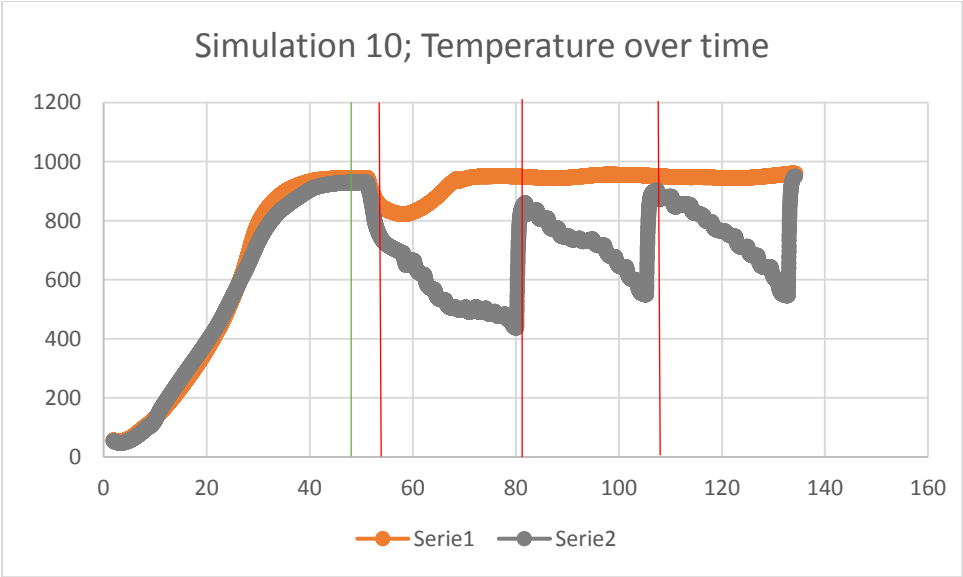


Figure 34: Temperature over time for simulation 10

Figure 35 shows the temperature as a result of distance from the melt as a result of distance from the melt. The temperature ranges from 635 to 860 °C.

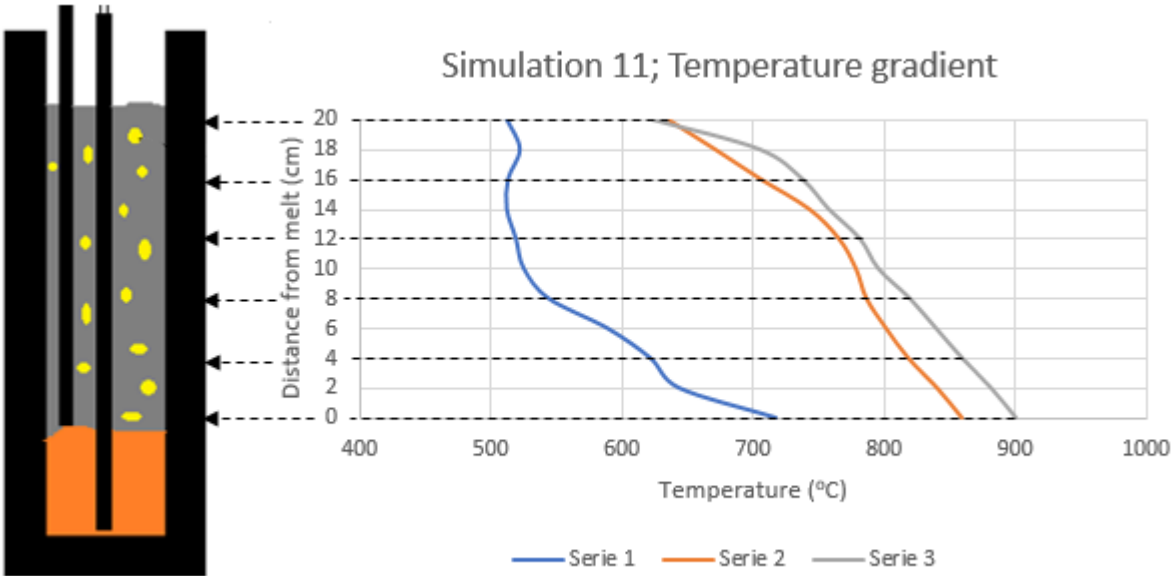


Figure 35: Temperature as a result of distance from the melt.

Figure 36 shows the temperature over time for simulation 11. Temperature drop after addition of carpet layer were only 70 °C, however it took 25 minutes before it was stable at 960 °C.

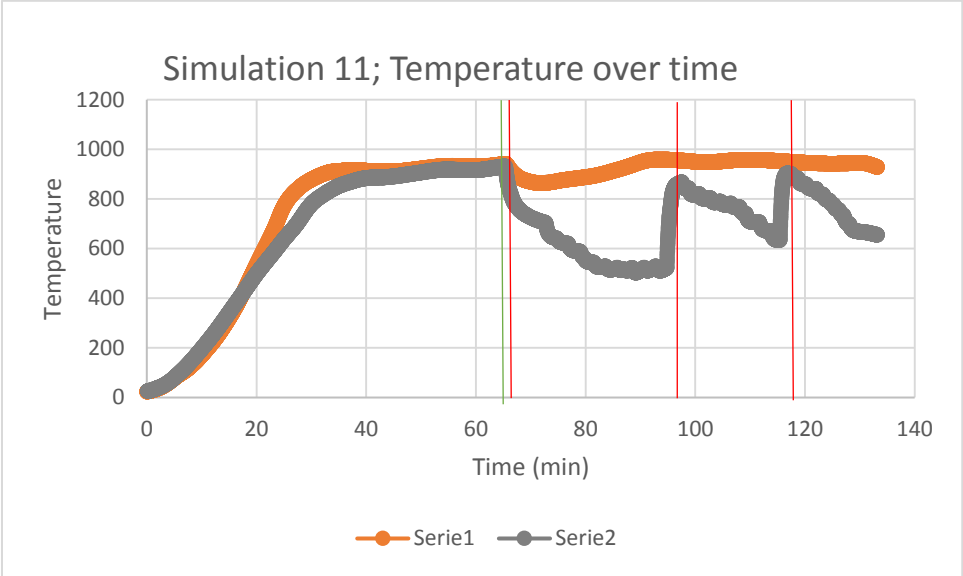


Figure 36: Temperature as a result of time.

Figure 37 shows the temperature as a result of distance from the melt for simulation 12. The temperature ranged between 849 °C right above the melt and 650 °C at the top.

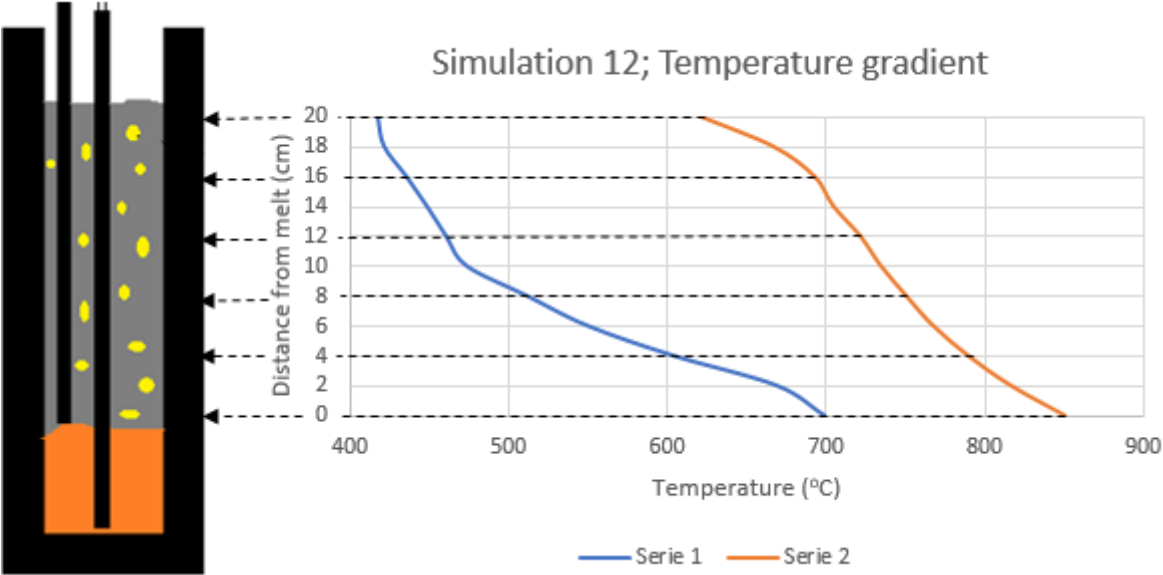


Figure 37: The temperature gradient for simulation 12

Figure 38 shows the temperature over time for both thermocouples. Temperature decreased with approximately 100 °C after carpet is added and used 20 minutes to get back to 960 °C.

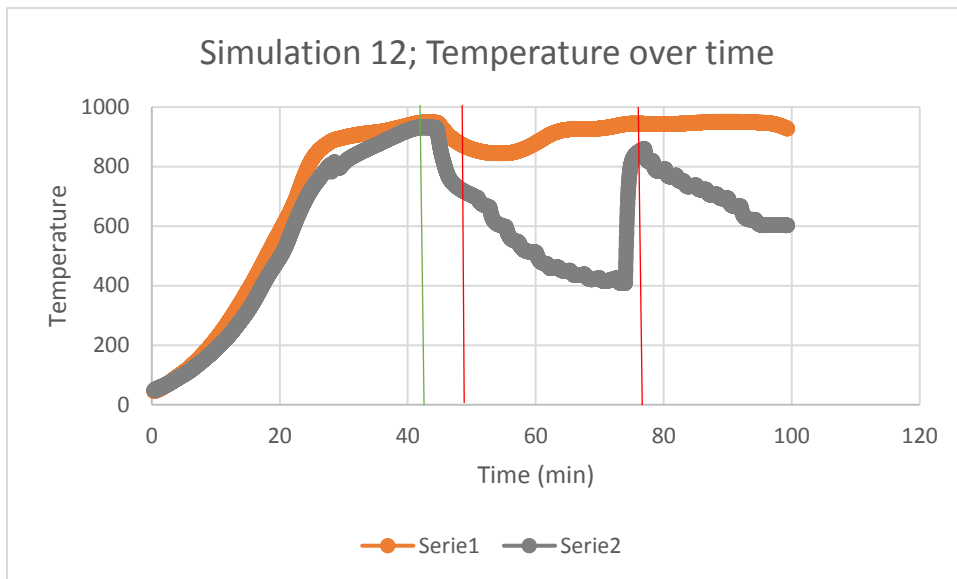


Figure 38: Temperature over time for simulation 12.

In all the simulations containing waste, smoke was observed from the crucible during heating. The smoke lasted for approximately 12 minutes each time.

### 3.3.7. Simulation 13 and 14

These simulations were conducted in the same way as simulation 10, 11 and 12. However the crucibles were not filled with epoxy afterwards. Instead, the waste was dug out, and their depth on the carpet and distance from the melt in the crucible was noted. Temperature gradient is shown in figure 39. Temperature range from 626 °C at the top to 856 °C right above the melt.

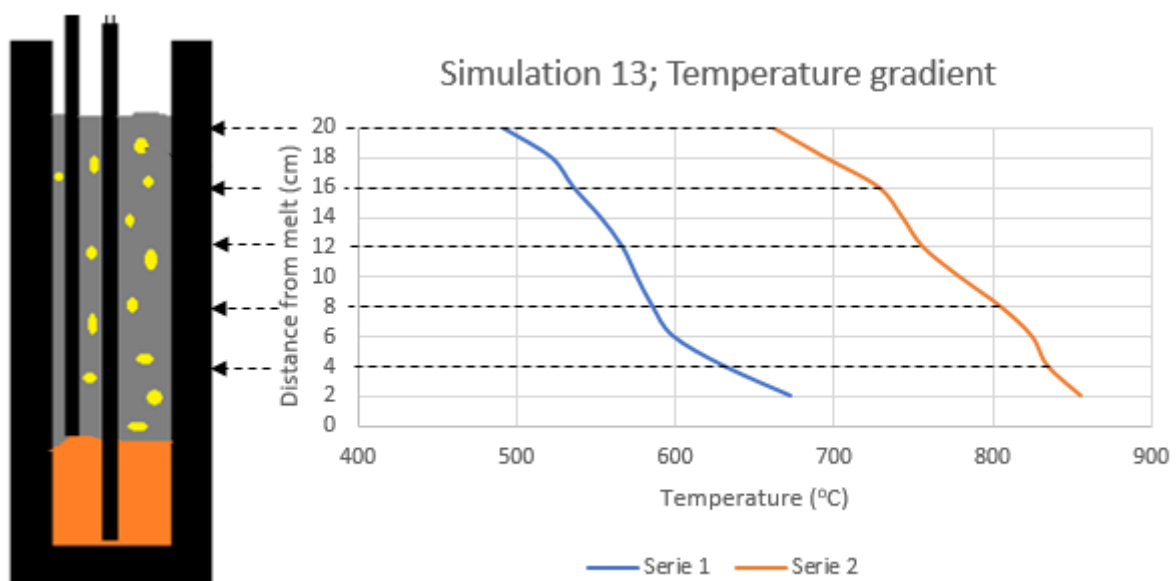


Figure 39: Temperature gradient for simulation 13

Figure 40 show the temperature over time for simulation 13. Temperature decreases with approximately 100 °C after the addition of the carpet layer and use 20 minutes to get back to 970 °C.

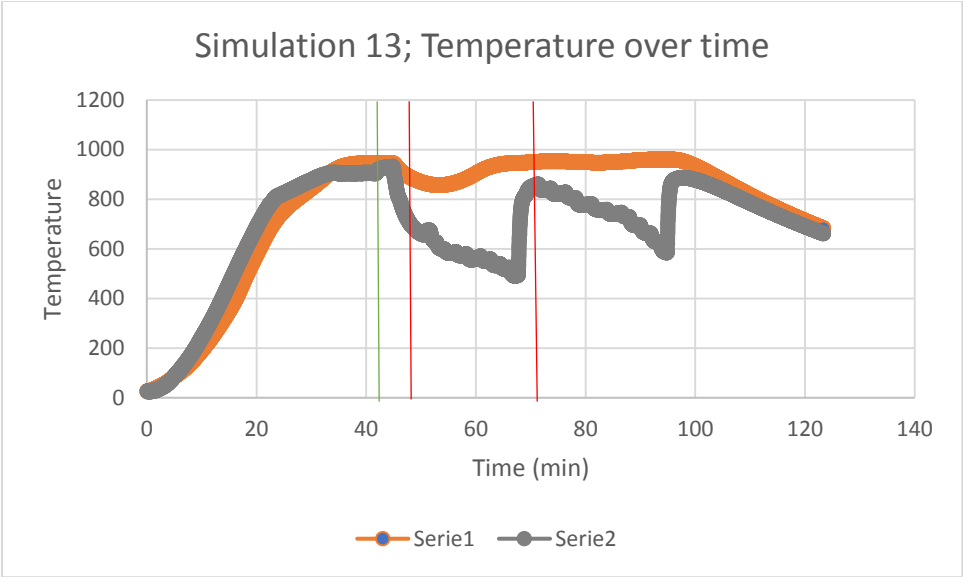


Figure 40: Temperature over time for simulation 13

Figure 41 shows the temperature gradient for simulation 14. The temperature ranges between from 505 °C at the top to 810 °C right above the melt.

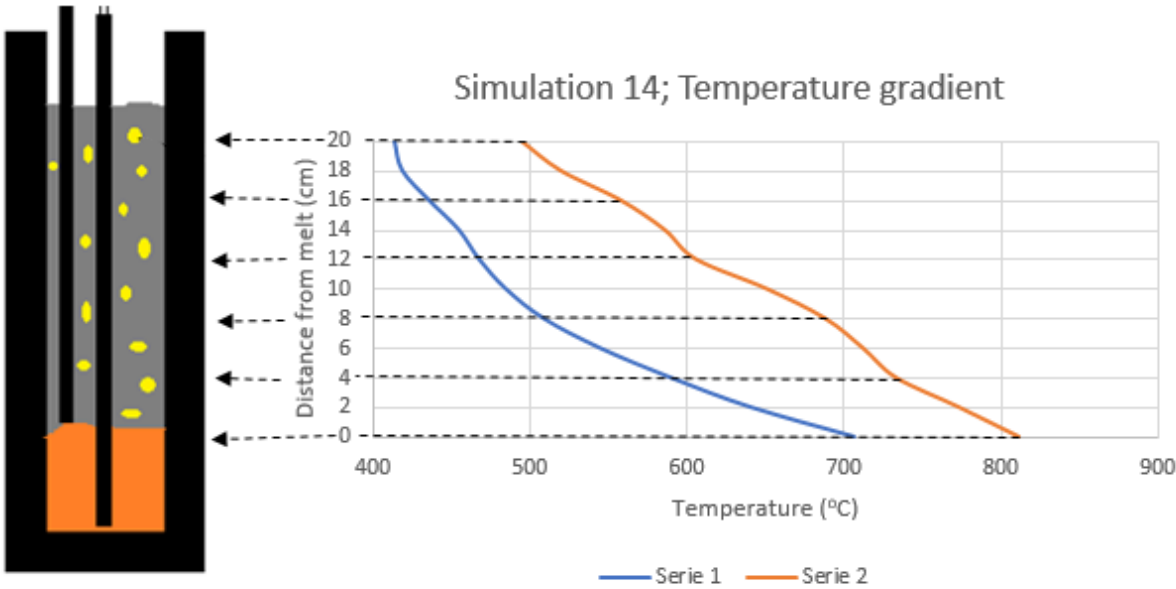


Figure 41: Temperature gradient for simulation 14



Temperature over time for both thermocouples are shown in figure 42. Temperature drops approximately 100 °C after the addition of the carpet layer, and used 20 minutes to get back to 960 °C.

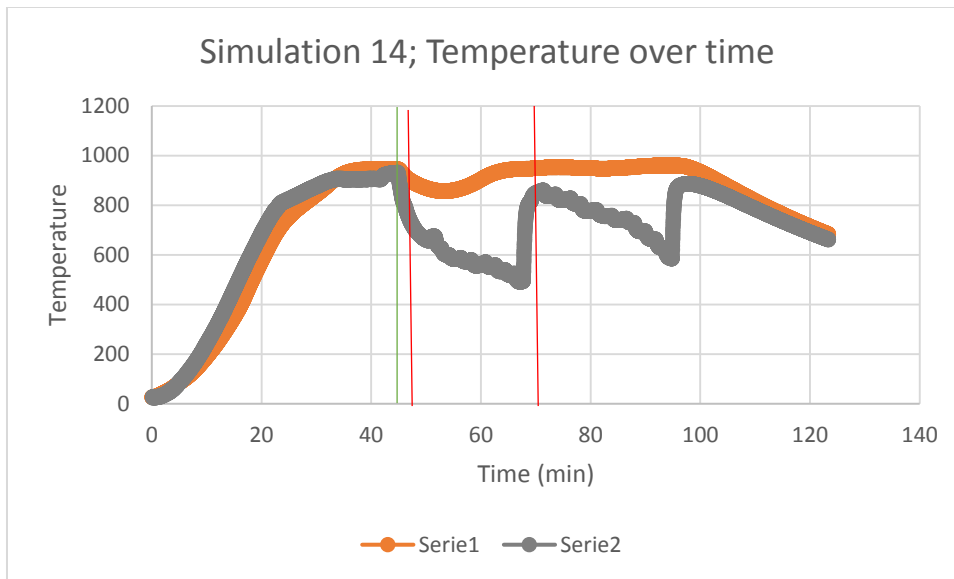


Figure 42: Temperature over time for simulation 14

### 3.3.8. Scanning electron microscope

Scanning electron microscope (SEM) is a useful instrument for characterization of materials and surfaces. The general principle of an electron microscope is that a beam of electrons is aimed at an area of the sample. When the electrons hit the sample, then the interaction between the material and the electrons will result in numerous different signals, which are captured by different detector that will interpret the signal. Figure 43 shows a setup of a SEM with its different components. Two different detectors are of particular interest for the analyses in this thesis; Secondary electron detector (SE) and energy dispersive spectrometry (EDS)[11]

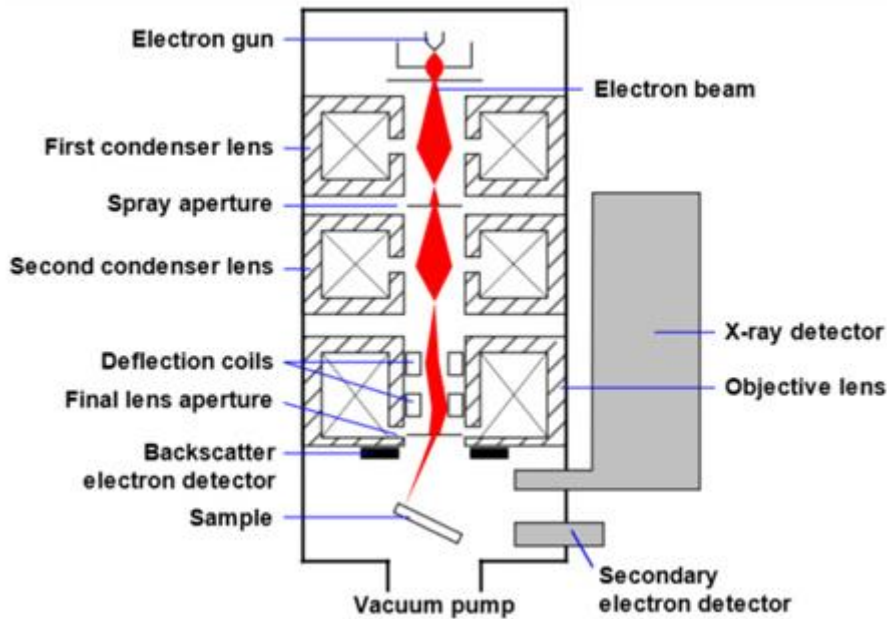


Figure 43: The set-up and the different components of a scanning electron microscope [14]

*Secondary electron imaging:* SE is primarily used for topography observation. When the electron beam hits the surface, electrons in the atoms in sample will be emitted from the surface and detected in a secondary electron detector. Secondary electrons are emitted very close from the sample surface. For this reason, the SE will give very high resolution. For this reason it is very useful for topography observations. [11]

*Energy dispersive spectroscopy:* EDS is a technique used for composition characterization of a sample. When the electron beam hits the sample, x-rays are emitted. The energy of these x-rays is specific for each element, and thus can a detector distinguish the different elements in the area detected. In order to use EDS on the sample has to be completely flat, with a smooth surface, hence the need for extensive grinding and polishing of the sample beforehand. [11]

*Acumulation of charge:* If the sample is not made of a conductive material, the accumulation of charge from the electrons from the electron beam will after a short while interfere with the picture [11]. In order to remedy for this, it is necessary to apply a conductive coating, usually of carbon or gold. In addition, the sample can be wrapped in aluminum foil with a carbon sticker to make the sample more conductive.

### 3.3.9. Carbon coater

The carbon coater is often used to provide a thin layer of less than 20nm uniform and conducting carbon coating. Two carbon rods are sharpened and placed in a chamber with the samples. After

a pump provides vacuum pressure, a high current is passed through the carbon rods at the point where they touch. This will result in a sputtering deposition of carbon on everything in the chamber, including the samples [12]. A sketch of the carbon coater is shown in figure 44.

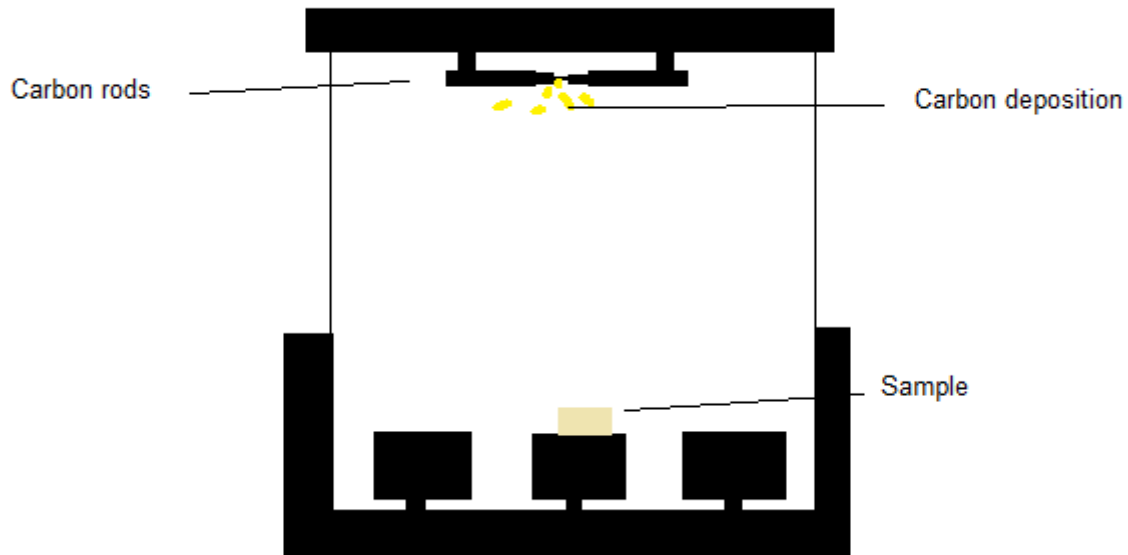


Figure 44: A sketch of the Cressington 208 Carbon coater

### 3.3.10 LECO

LECO is an analysis method used to determine the amount of carbon, sulfur or oxygen in a sample. This is done converting the sample to its oxidized form and then measuring infrared absorption of combustion gases. When determining the amount of carbon, the carbon is combusted, and then the amount of carbon dioxide produced is measured. The amount of carbon dioxide produced is proportional to the amount of carbon in the sample. Prior to the measuring, the sample has to be powdered, chemically treated and weighted [15].

### 3.4. Sample preparation and analysis

After cooling all the crucibles except those from simulation 13 and 14 were filled with epoxy and cut at the center. Four samples were cut out using a Struers LoboTom from the sample and analyzed in SEM using SE and EDS analysis. Before analyzed in microscope. The samples were grinded and polished in four steps. The parameters used in polishing is shown in table 8.

Table 8: The parameter used for grinding and polishing the sample

NUMBER	SIZE	POLISHING MATT	LUBRICANT	TIME
1	220 $\mu\text{m}$	Piano	water	2 min
2	9 $\mu\text{m}$	Allegro	Allegro9	5 min
3	3 $\mu\text{m}$	Dur/Dac/Daran	Dac3	5 min
4	1 $\mu\text{m}$	Nap	Nap1	2 min

The samples were placed in an oven for 24 hours at 66 °C in order to let the epoxy fume off before placed in a microscope. For simulation 13 and 14, the crucible was not filled with epoxy, but the samples were rather dug out, and their depth in the crucible was noted.

Before analyzed in microscope, a thin 20 nm carbon coating was added to the samples. In addition, the samples were partially coated in aluminum foil. The parameters used in the microscope is shown in table 6. The samples from simulation 13 and 14 was only analyzed using SE detector and hence were not subjected to casting in epoxy or any other pretreatments, besides carbon coating.

Table 9: Parameters used in SEM

	EHT(KW)	WD(MM)	APERTURE( $\mu\text{M}$ )	CURRENT
SE	5	10-15	30	Low
EDS	10	10-12	60	High

## 4. Results

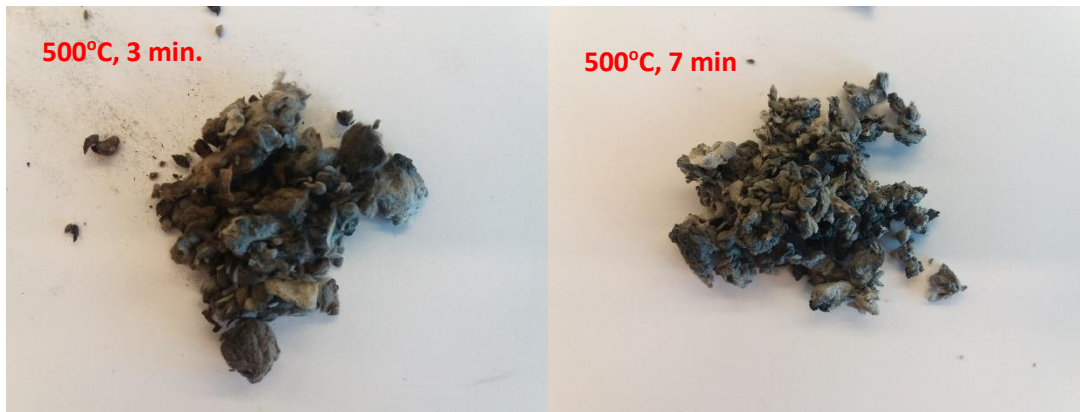
This section focusses on the results of the experiments explained in the previous chapter. The first section will emphasize on the results from the incineration experiments in atmospheric conditions, while the Second section will explain the results from the simulations. As previously mentioned all waste used was dried and had its moisture removed. The amount of water in the glass wool waste is shown in table 10:

*Table 10: The amount of water in the glass wool waste.*

Batch	Amount of water (wt%)
1	71
2	69
3	65
4	73
5	70

### 4.1. Incineration in atmospheric conditions

At 900 and 1000 °C the glass melted. The glass exposed to 500 and 600 °C however did not melt but showed a different degree of colorization. The waste subjected to lower temperature for a lower amount of time were darker and black, due to soot. The waste that had been in the furnace for a longer time was light gray/white. A visual representation is shown in figure 45-46.

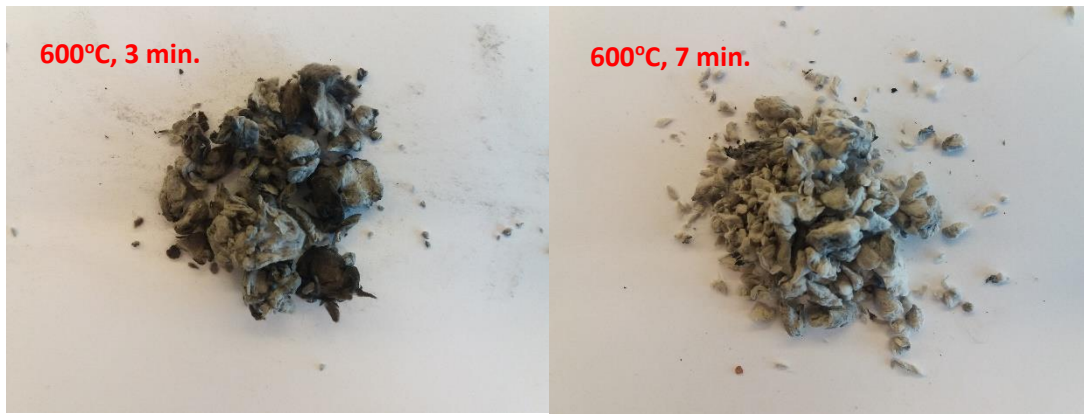


*Figure 45 a-b: Glass wool waste after being exposed to 500 °C for a) 3 minutes, and b) 7 minutes*



*Figure 45 c-d: Glass wool waste exposed to 500 °C for c) 15 min. and d) 20 minutes*

It can be seen that for the samples exposed to 500 °C there is a gradual decrease in soot the longer it was in the furnace. There are some differences in color within each sample, but the overall pattern is the same. The first sample, being in the furnace for only 3 min is almost completely black, while the sample that have been in the furnace for 20 min is almost completely white.



*Figure 46 a-b: Glass wool waste exposed to 600 °C for a) 3 minutes, and b) 7 minutes*



*Figure 46 c-d: Glass wool waste exposed to 600 °C for c) 15 minutes, and d) 20 minutes.*

For the samples subjected to 600 °C only the sample being in the furnace for 3 min shows any traces of soot, while the rest are almost completely white.

The results from the incineration experiments are shown in figure 47. The graph shows the total weight percent of organic content in the waste by weighing the sample before and after incineration. All the samples had weight percent of organic content between 15 and 17%.

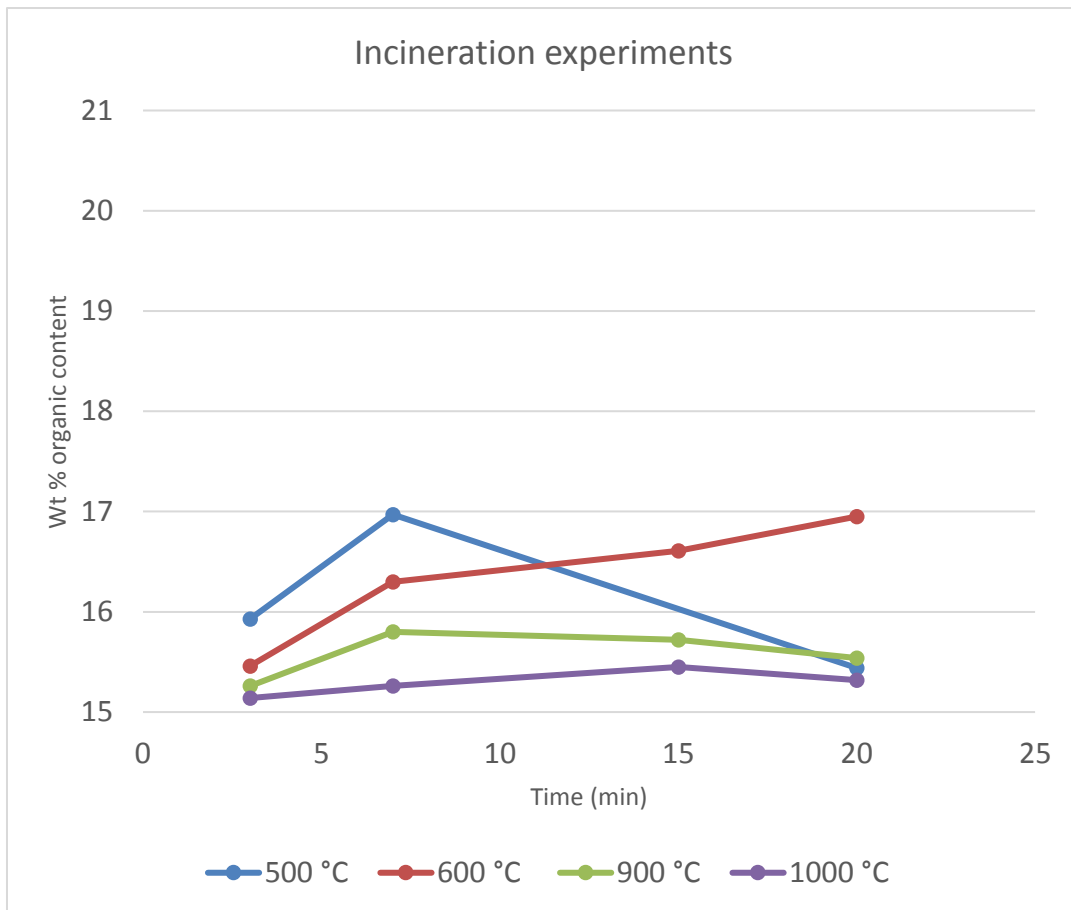


Figure 47: total mass loss after incineration for all the samples. All samples have been exposed to different temperatures for a different amount of time.

Some of the samples was sent to SintefMolab to perform a carbon analysis to determine the amount of carbon in the samples. The result is given in table 11, and it is also seen here that very little carbon was left after incineration at 500 °C for 3 minutes, and even less for the other samples.



Table 11: Amount of carbon left in samples after incineration

Temperature °C (20min)	Carbon content wt%
500	0,43
500 (3min)	1,48
600	0,07
900	0,12
1000	0,34
untreated	10,6

A SEM analysis was performed on the unmelted samples 1-8, in other words, the samples exposed to temperatures of 500 and 600 °C, as well as untreated glass wool waste. Figure 48-55 shows the pictures. Magnification 100,200,500 and 1000x was used on all the samples.

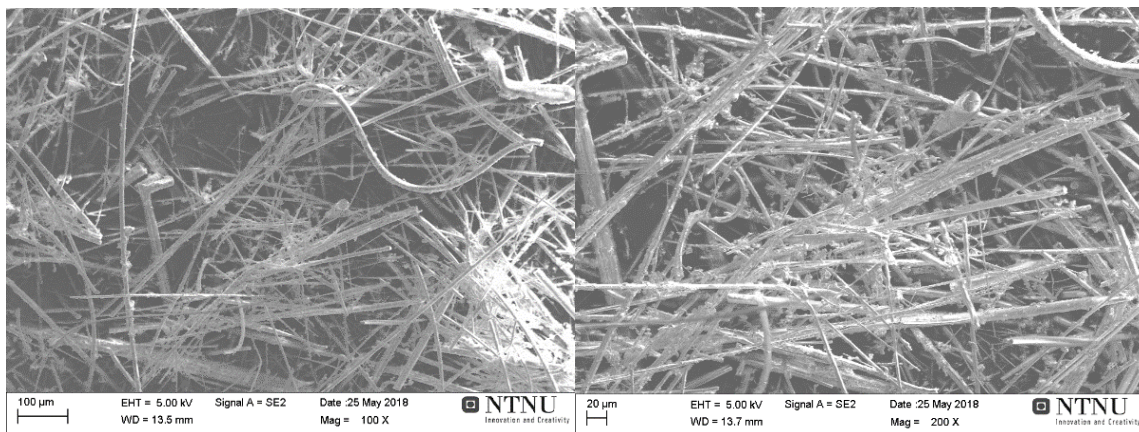


Figure 48 a-b) Untreated glass wool waste at 100x(a) and 200x(b)

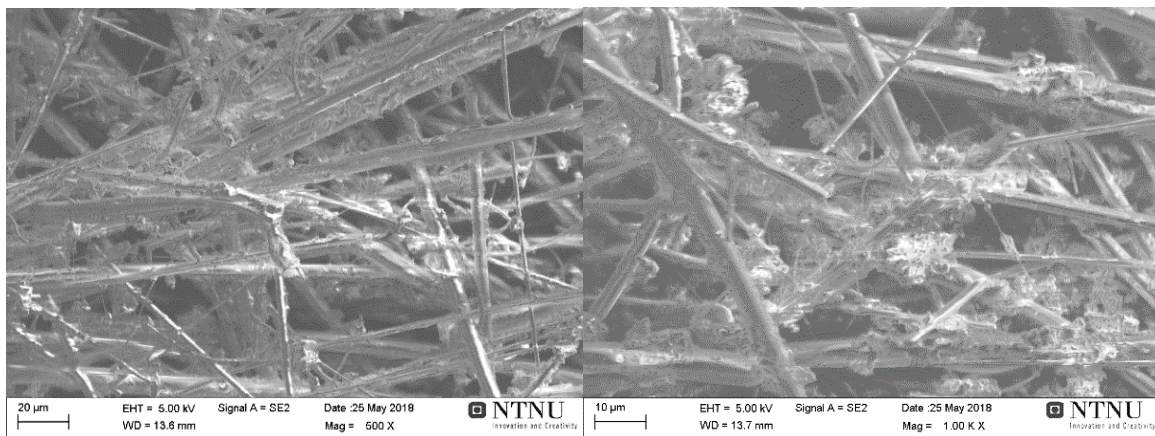


Figure 48 c-d) Untreated glass wool waste at 500x(c) and 1000x(d)

The untreated wool showed to have huge amount of organic content. As seen from the pictures the binder is attached to the fibers. A certain amount of dust is attached to the binder as well. The figures below show the samples from the incineration experiment.



Figure 49 a-b: Sample after heating at 500°C for 3 minutes, at a) 100x and b) 200x

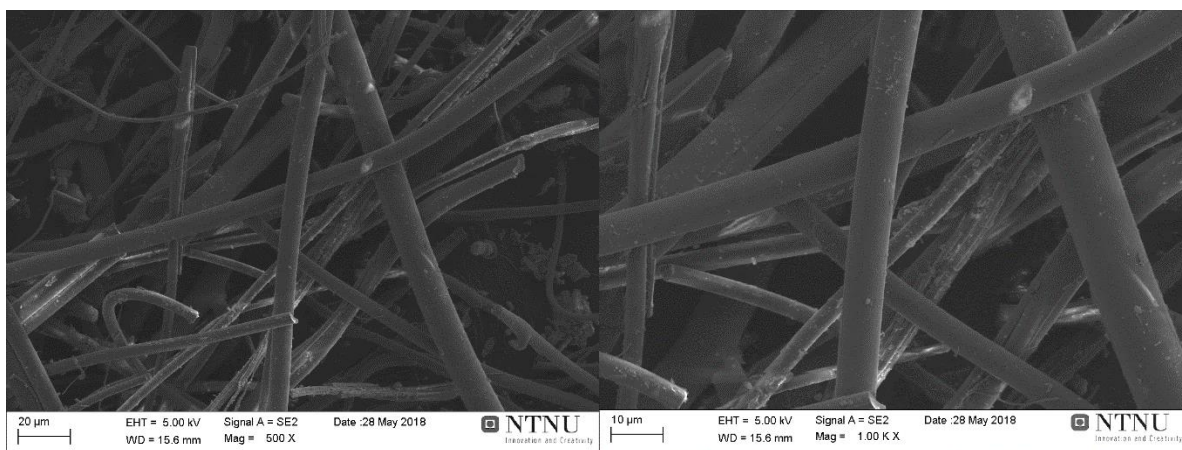


Figure 49 c-d: Sample after heating at 500°C for 3 minutes at c) 500x and d) 1000x

Figure 49 shows the sample from the incineration experiment exposed to 500 °C for 3 minutes. Almost no trace of organic content is observed. According to the LECO analysis by SintefMolab this sample have 1,48% carbon.

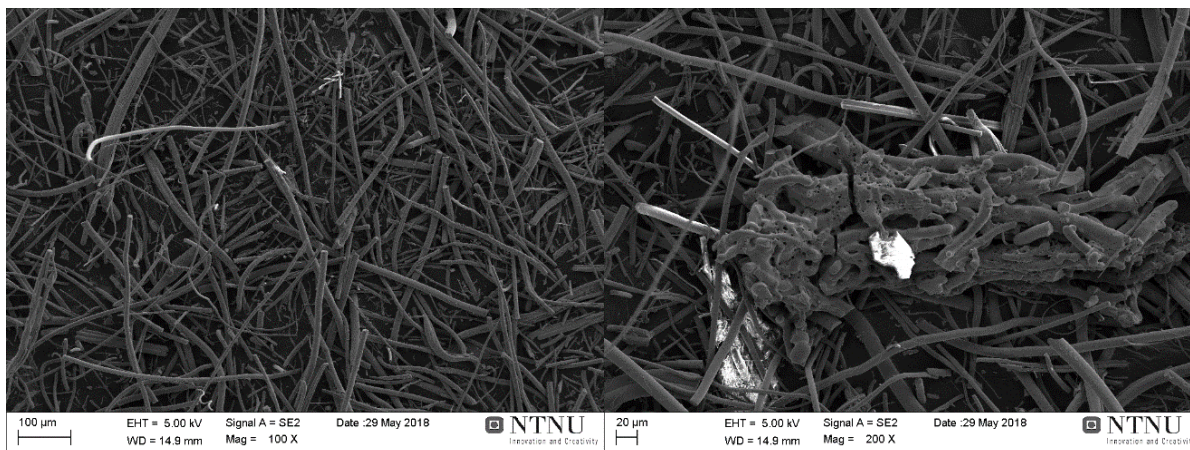


Figure 50 a-b) Sample after heating at 500°C for 7 minutes at a) 100x and b) 200x

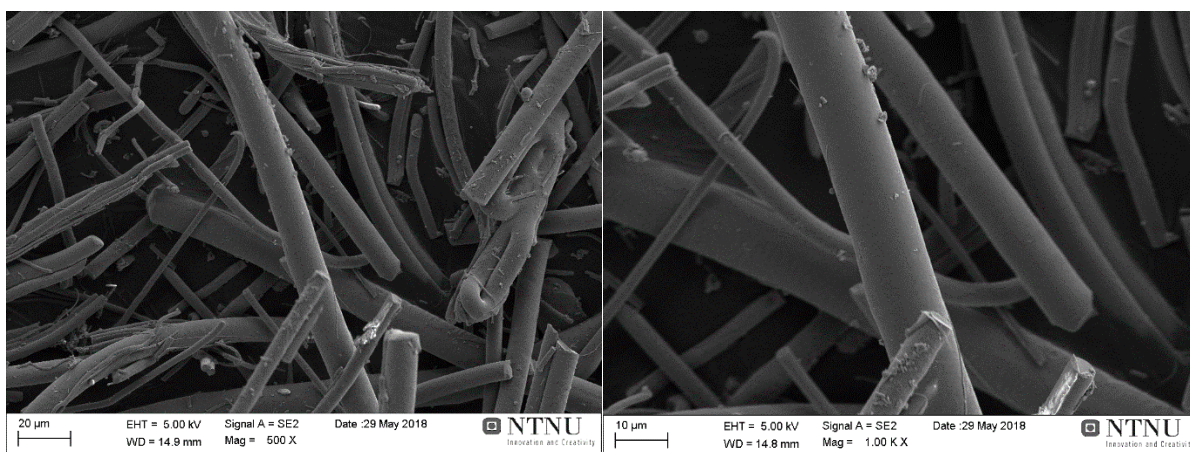


Figure 50 c-d: Sample after heating at 500 °C for 7 minutes at c) 500x and d) 1000x

Figure 50 shows the SEM pictures from the incineration experiment exposed to 500 °C for 7 minutes. There is no observable difference between this sample and the previous one regarding organic content. No LECO analysis were taken from this samples. However, based on the picture the amount of carbon left in the sample is minimal. Some of the fibers have partly melted together as seen in figure 50b.

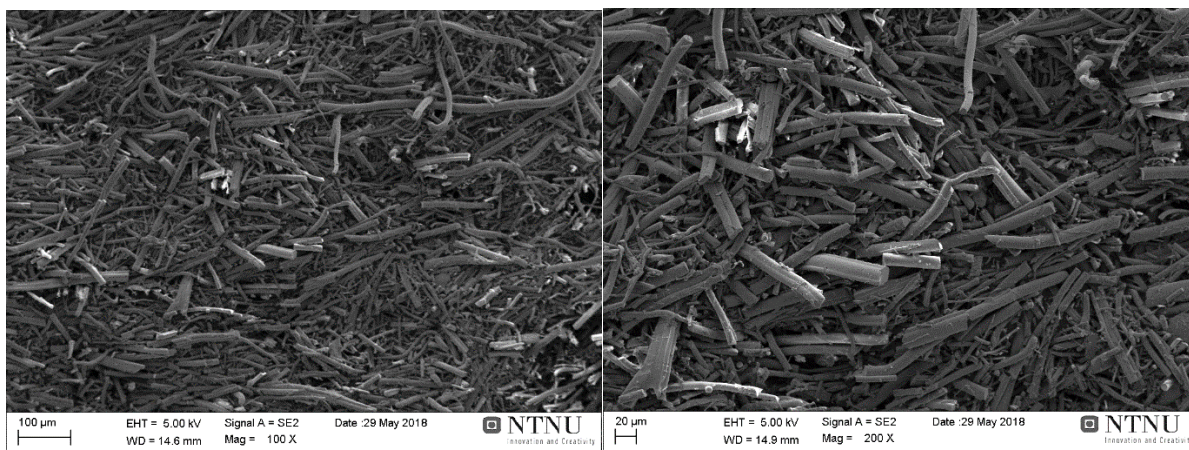


Figure 51: a-b: Sample after heating at 500°C for 15 minutes at a) 100x and b) 200x

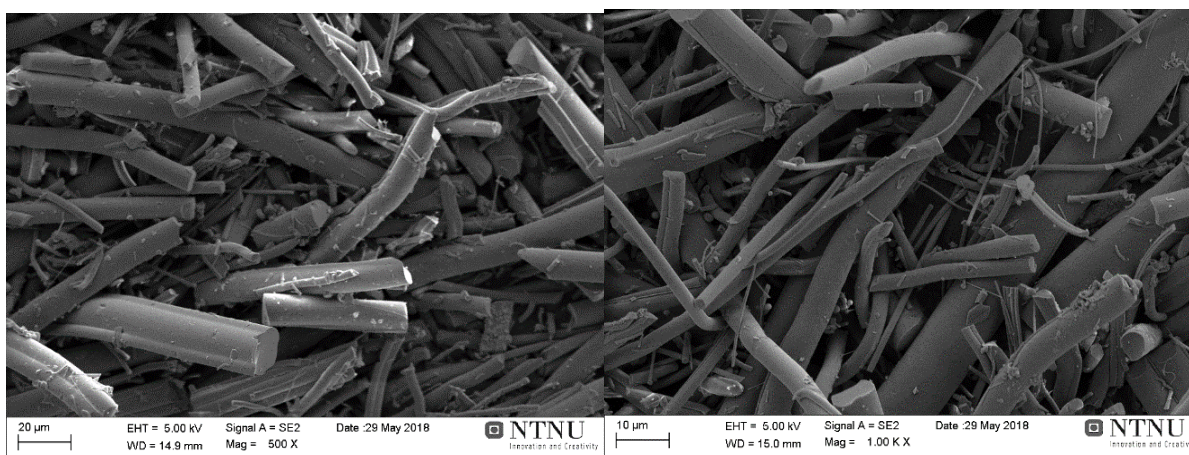


Figure 51 c-d: Sample after heating at 500°C for 15 minutes at c) 500x and d) 1000x

Figure 51 show the samples exposed to 500 °C for 15 minutes. As with the previous sample, no LECO analysis was taken of this. No significant amount of carbon was observed.

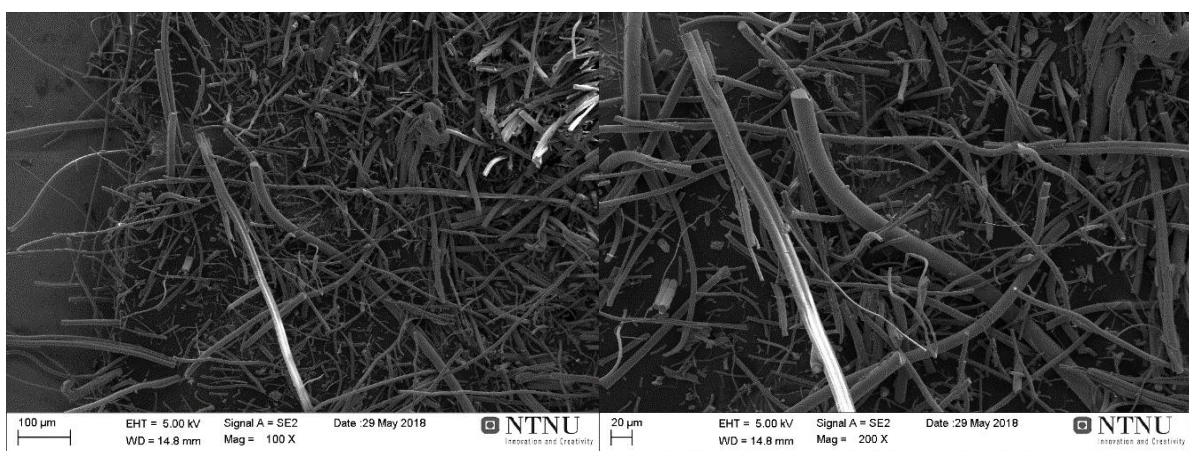


Figure 52 a-b: Sample after heating at 500 °C for 20 minutes at a) 100 x and b) 200x

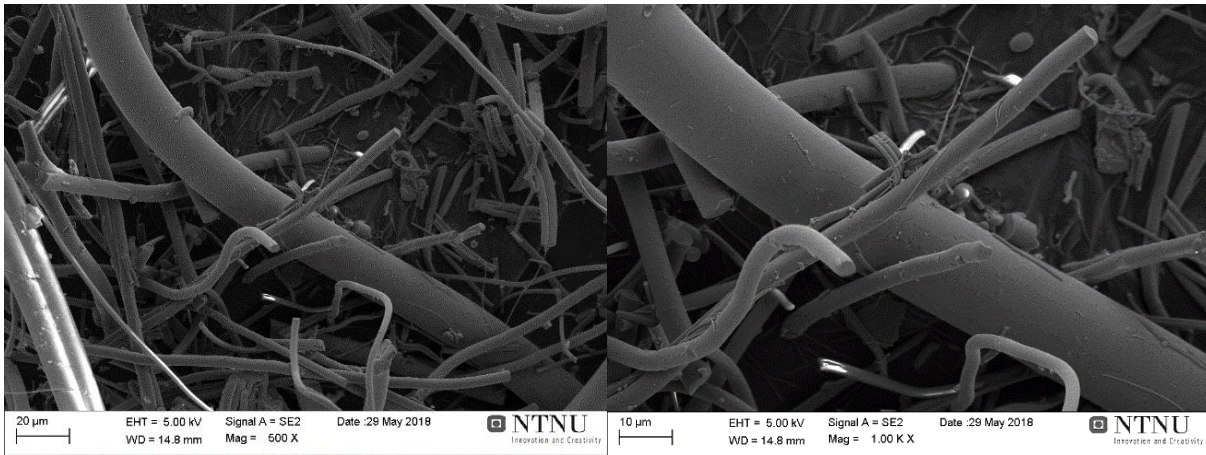


Figure 52 c-d: Sample after heating at 500 °C for 20 minutes at c) 500x and d) 1000

Figure 52 shows the sample exposed to 500 °C for 20 minutes. According to the LECO analysis this sample contains 0,43 % carbon. Since no significant amount of carbon was observed.

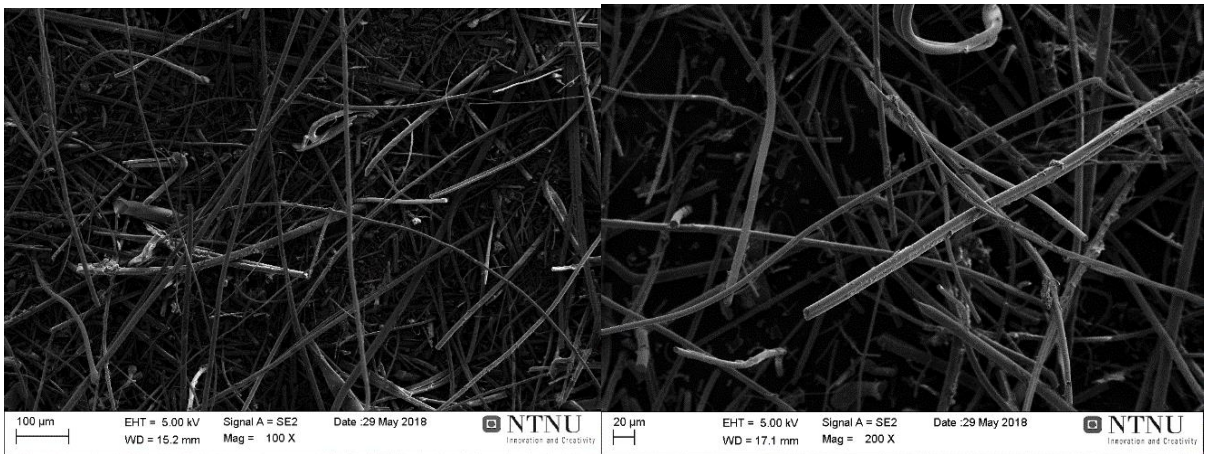


Figure 53 a-b: Sample after heating at 600 °C for 3 minutes at a) 100x and b) 200x

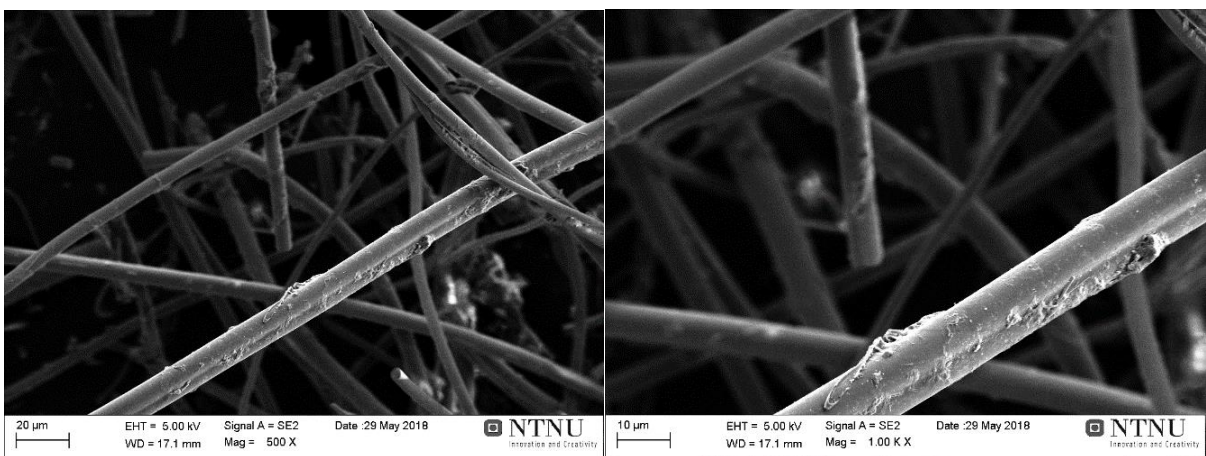


Figure 53 c-d: Sample after heating at 600 °C for 3 minutes at c) 500x and d) 1000x

Figure 53 show the sample exposed to 600 °C for 3 minutes. No significant amount of carbon was observed.

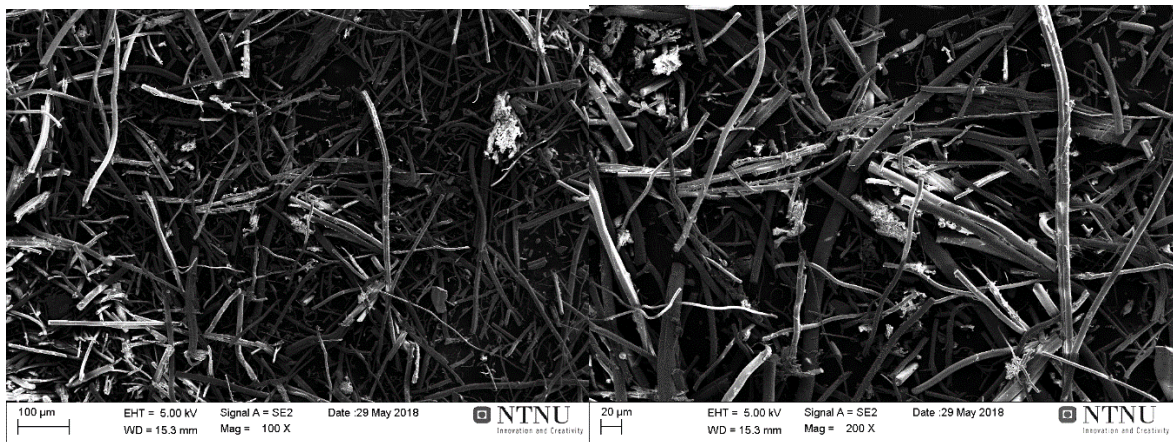


Figure 54 a-b: Sample after heating at 600 °C for 15 minutes at a) 100x and b) 200x

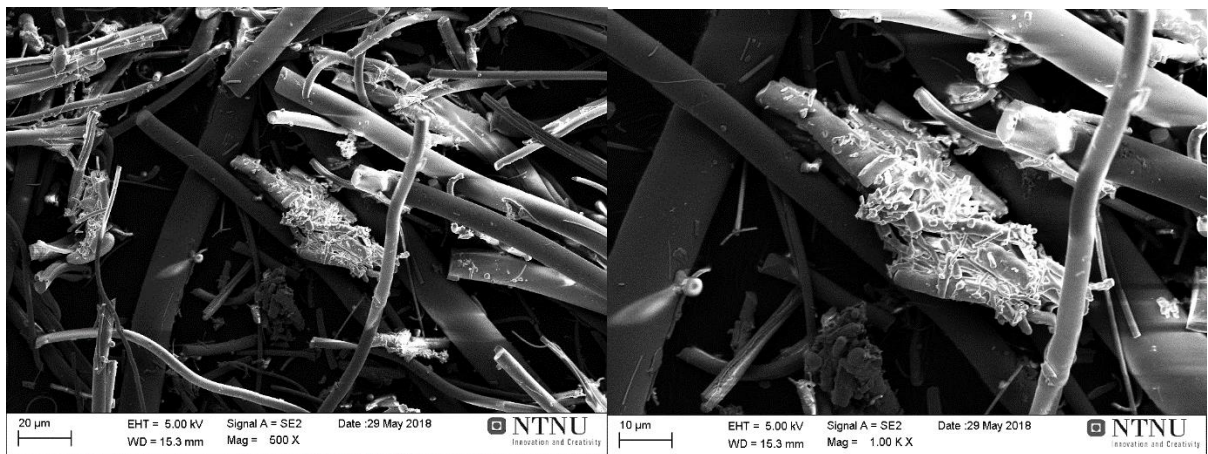


Figure 54 c-d: Sample after heating at 600 °C for 15 minutes at c) 500x and d) 1000x

The sample exposed to 600 °C for 15 minutes is shown in figure 54. Only small amount of carbon was observed. The fiber seems to have partially been melted together.

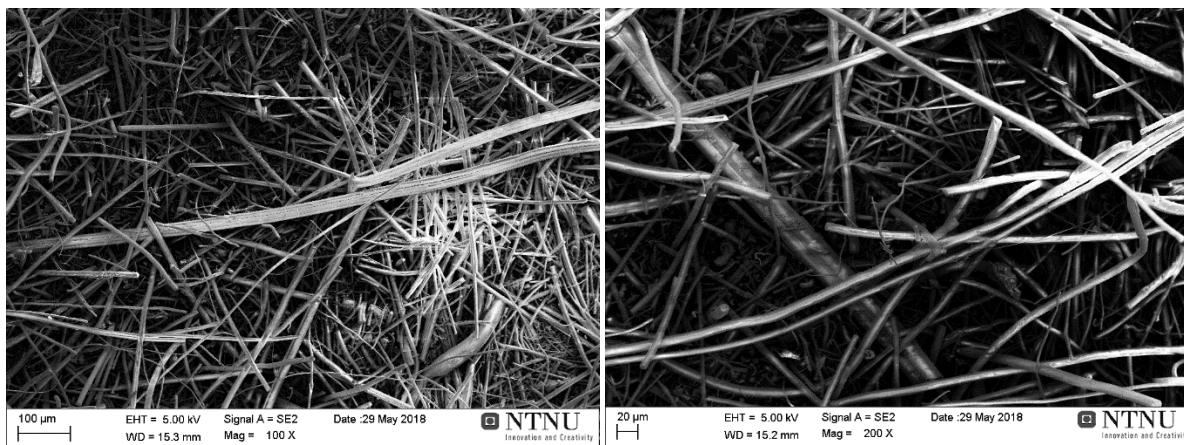


Figure 55 a-b: Sample after heating at 600 °C for 20 minutes at a) 100x and b) 200x

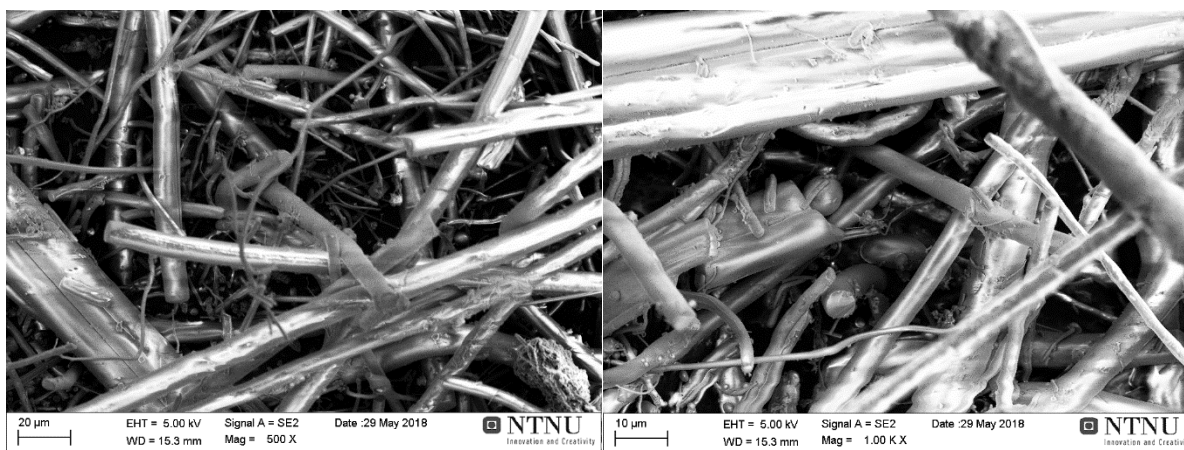


Figure 55c-d: Sample after heating at 600 °C for 20 minutes at c) 500x and d) 1000x

Figure 55 shows the sample exposed to 600 °C for 20 minutes. According to the LECO analysis the sample contains 0,07 % of carbon. As with the previous samples the exact amount is not possible to quantify from the pictures, however no significant amount of carbon was observed.

## 4.2. Simulations

A comparison of the different temperature gradients is shown in figure 56. Simulation 1,2,3 and 5 is left out of the comparison, due to not being comparable to the rest, since the method not being developed. An average temperature gradient for the simulations with waste and those without, is shown in figure 57. Only the temperature gradient from the second measurement, Serie 2 is presented in the graphs.

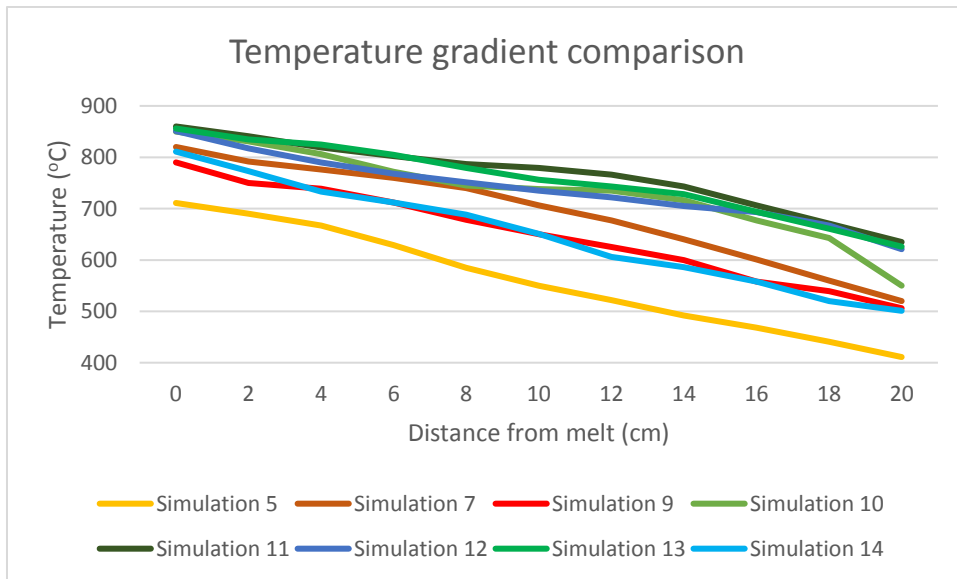


Figure 56: A comparison of the temperature gradients from the simulations.

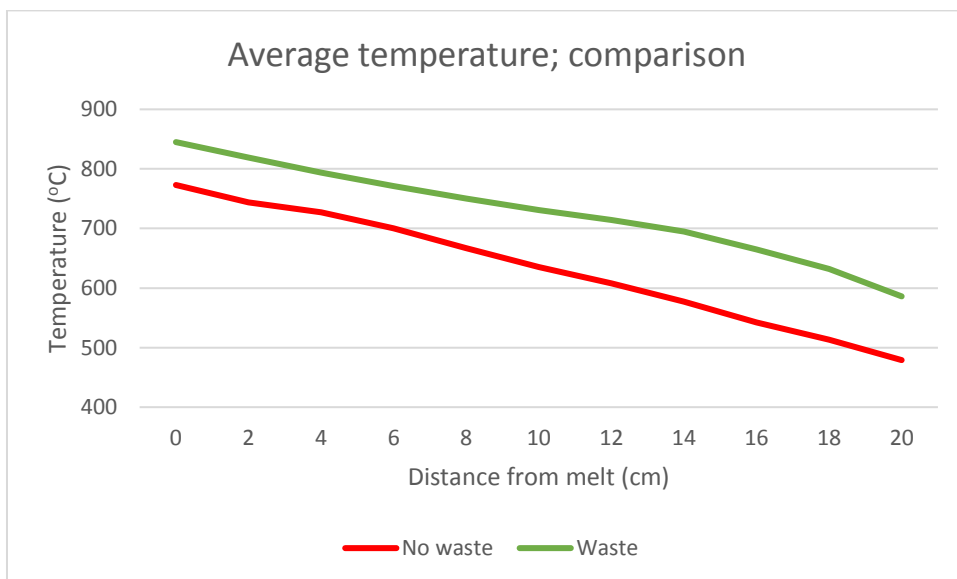


Figure 57: A comparison of the average temperature gradient for the simulations with or without waste.



It can be seen from both figure 56 and 57 that the simulations that contained waste generally had higher temperature, but the slope are still relatively parallel to those without waste.

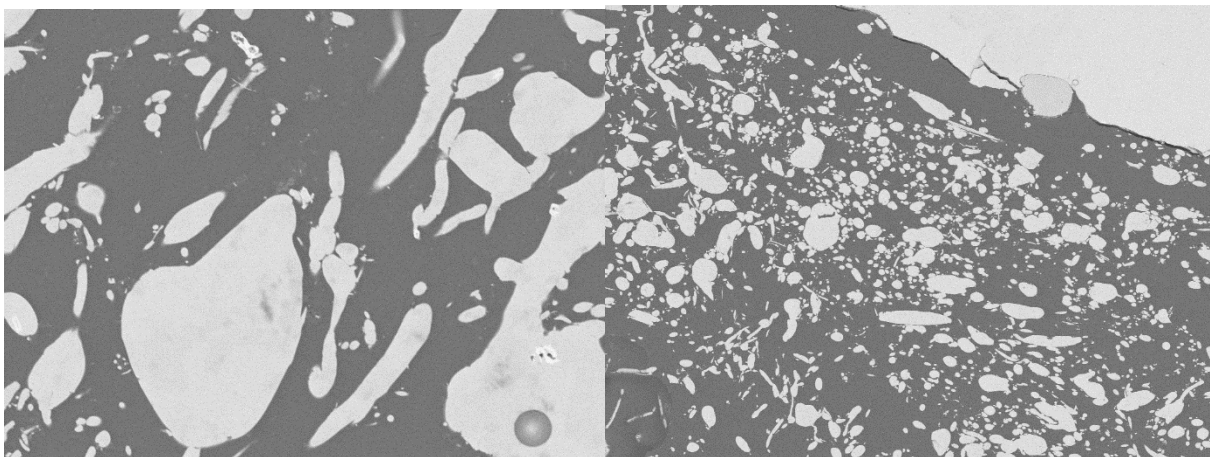
Table 12 shows the average decrease in temperature in °C when moving from the melt in the carpet.

*Table 12: The average decrease in temperature from the melt to the top*

Simulation	Temperature gradient (°C/cm)
4	-13,44
5	-15
7	-15
9	-14,2
10	-15
11	-11,5
12	-11,5
13	-9,7
14	-15,8

### 4.3. SEM analysis of simulations

This section shows the results from the SEM analysis. All the samples analyzed at four different magnifications; 100x, 200x, 500x and 1000x. No residue carbon could be found in the EDS analysis of any of the samples, and with the exception of figure 58 the pictures will not be shown here, since the information gathered from them were very limited. However, an analysis of the glass gave information of the composition of the glass. All the samples except sample 13 and 14 where filled with epoxy Backscatter pictures are given in figure 58, and the composition is given in table 13. Sample 13 and 14 was not filled with epoxy and the waste could hence be analyzed in SE.



*Figure 58: Picture of sample cast in epoxy*

Table 13: EDS analysis of the glass from simulation 10

	Mass%
SiO <sub>2</sub>	68,5
Na <sub>2</sub> O	10,4
K <sub>2</sub> O	8,1
Fe <sub>2</sub> O <sub>3</sub>	0,3
CaO	7,8
Al <sub>2</sub> O <sub>3</sub>	2,6
MgO	3,0
SO <sub>3</sub>	0,004-0,043

#### 4.3.1. Simulation 13

Figure 59 and 60 shows the glass wool waste dug out after the simulation. The waste at the top left corner was closest to the top while the bottom right was closest to the melt. The waste found further down in the crucible showed a distinctive darker color then the ones found closer to the top. Figure 59 shows the temperature gradient first presented in experimental chapter.

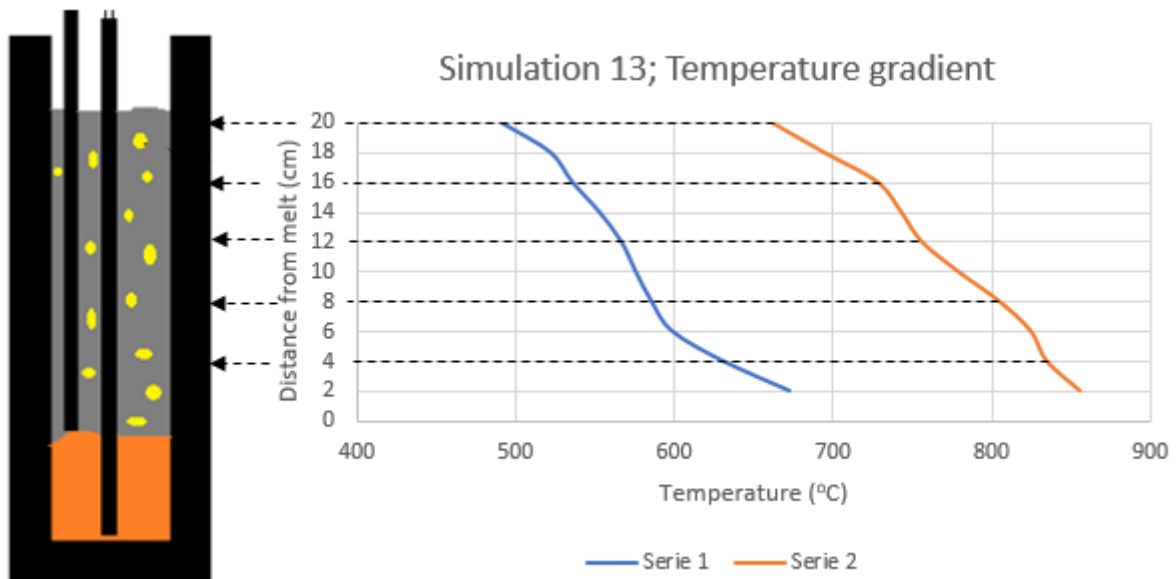


Figure 59: Temperature gradient for simulation 13.

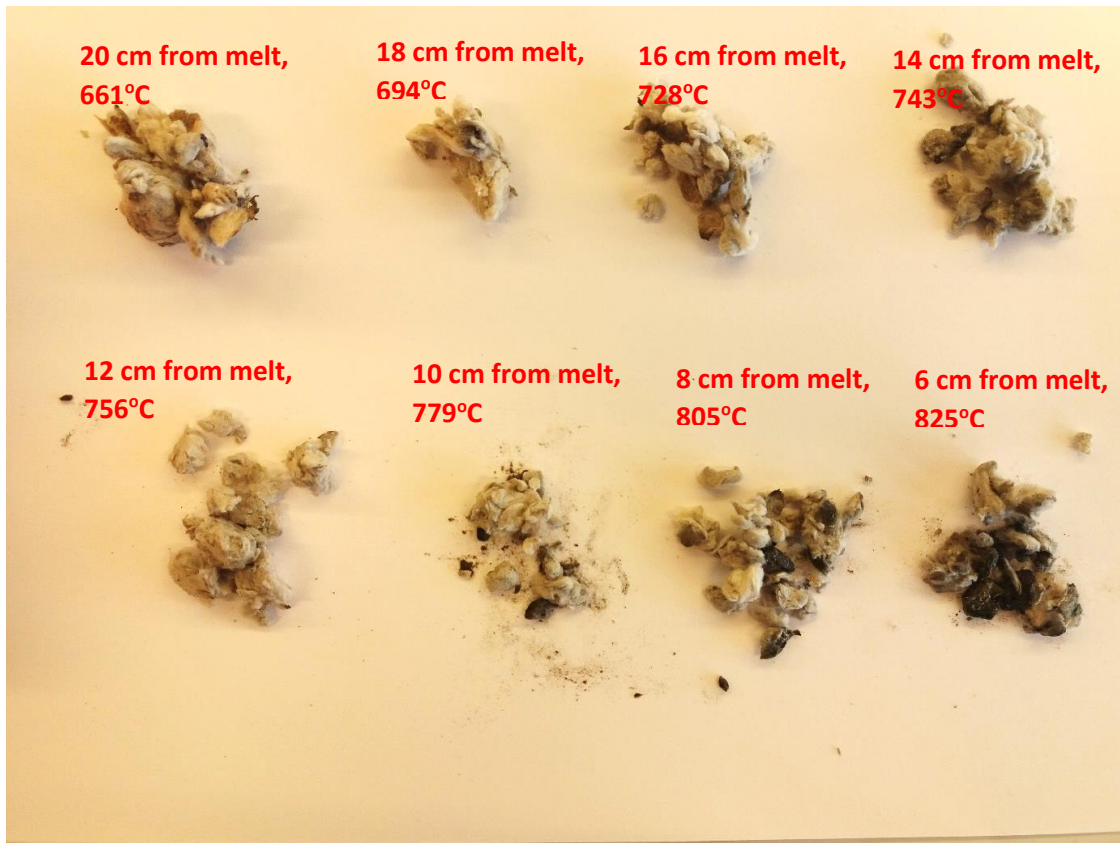


Figure 59: waste dug out from the crucible. The sample at the top left corner was closest to the top, and the bottom right closer to the melt

Picture of waste gathered from 4 cm and 4 cm above the melt is shown in figure X

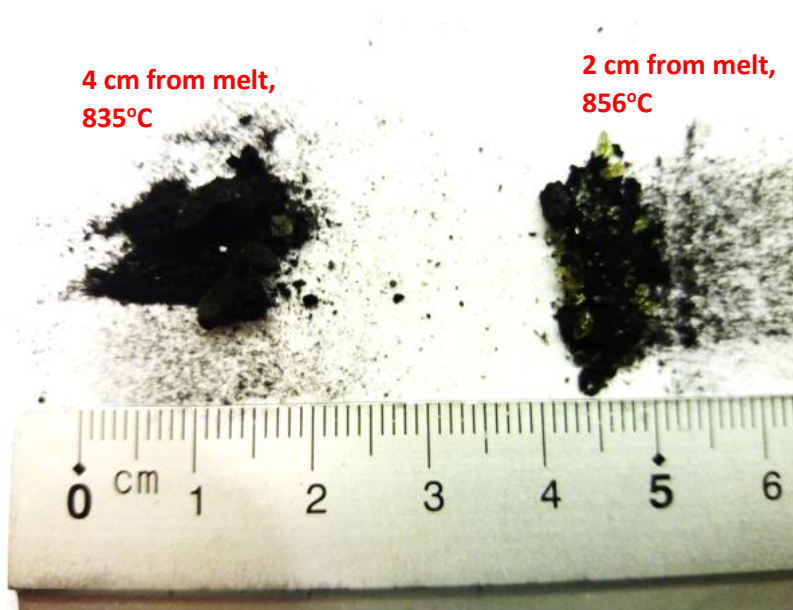


Figure 60: waste dug out from 4 and 2 cm depth.

It can be seen from figure 59 and 60 that the amount of soot increase on the samples closer to the melt. The samples from 4 and 2cm from the melt have a powder like structure compared to the rest of the fibers.

### 16 cm above melt – 728 °C

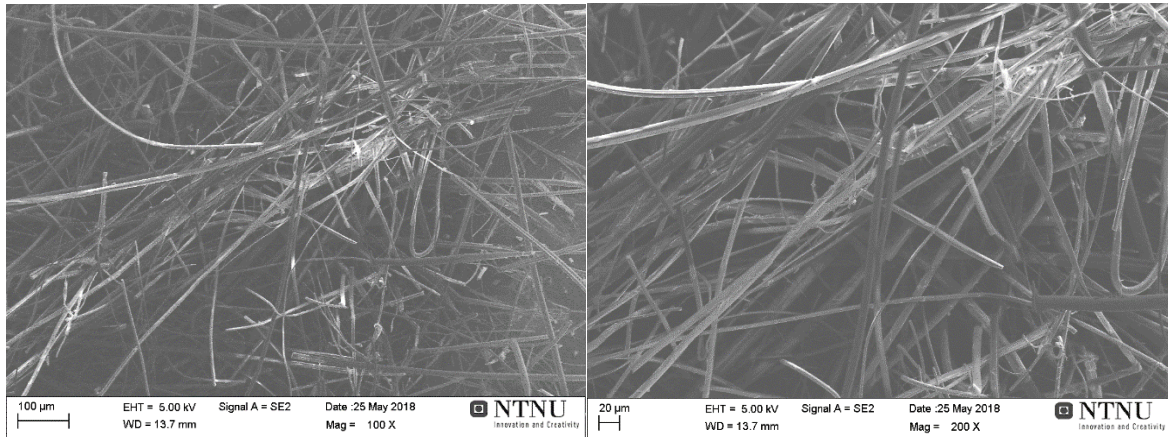


Figure 61 a-b: Waste 16 cm over the melt, beneath 4 cm of carpet at a)100x and b)200x

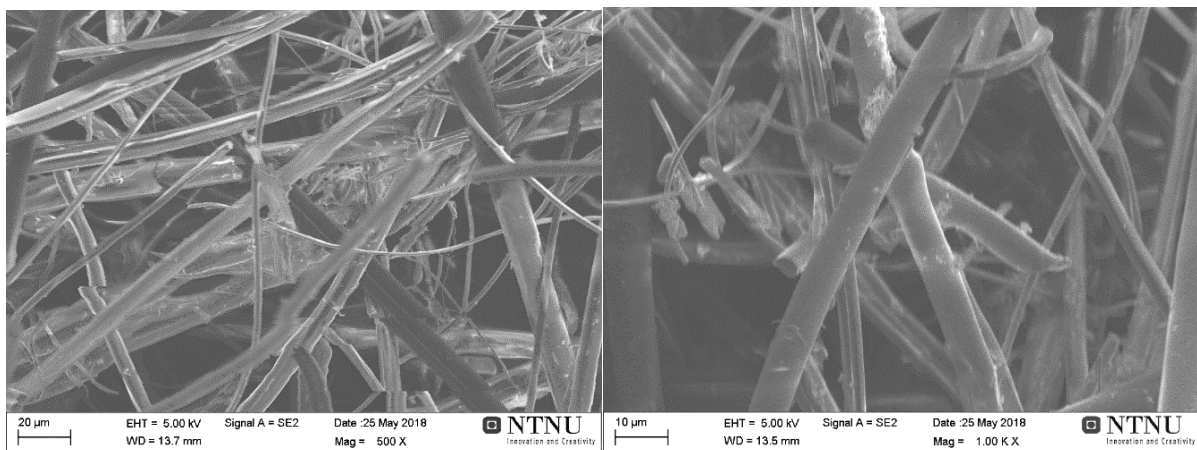
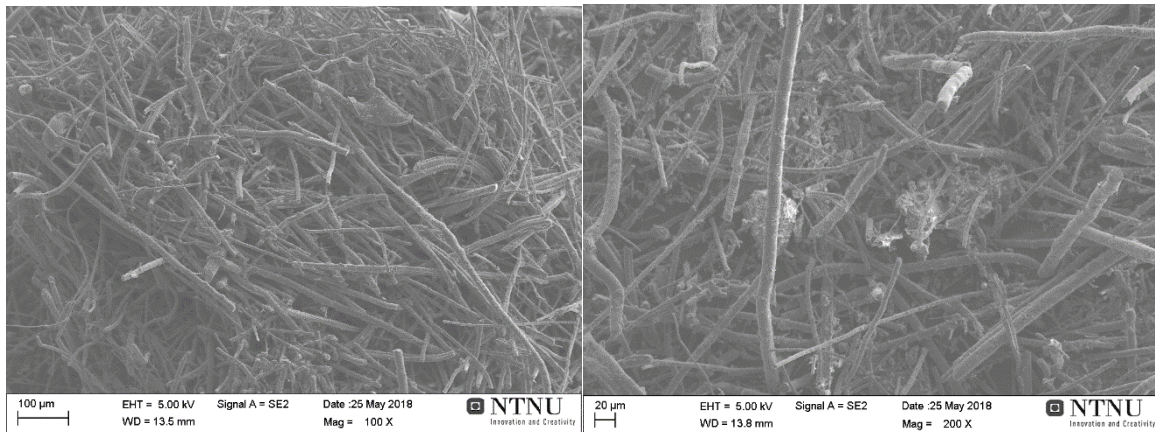


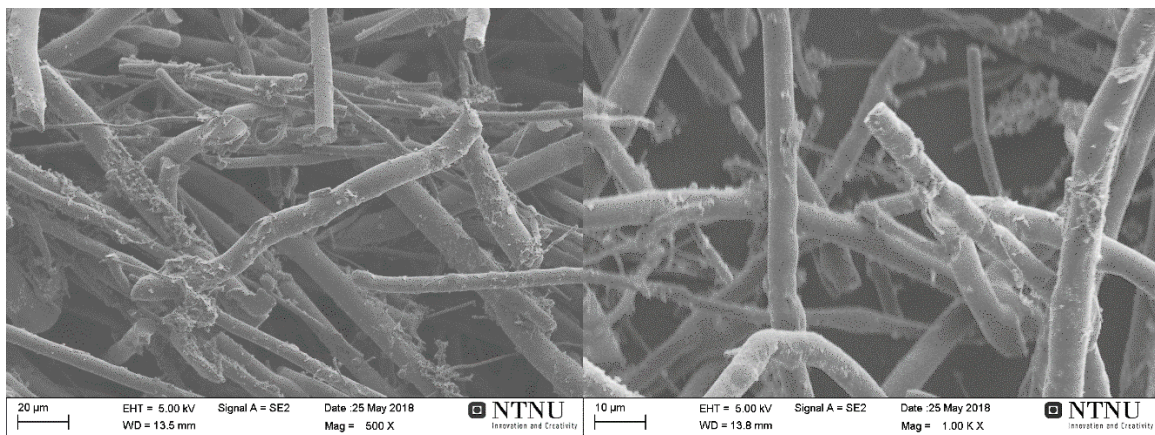
Figure 61 c-d: Waste from 16 cm over the melt, beneath 4 cm of carpet at c)500x and d)1000x

Figure 61a-d shows the glasswool waste collected from the simulation 13 simulation, beneath 4 cm of carpet. This location was 16 cm above the melt. The waste was exposed to 728 °C. The pictures show that most of the carbon is removed at this point, however small amounts are still observed.

**12 cm above melt – 756 °C**



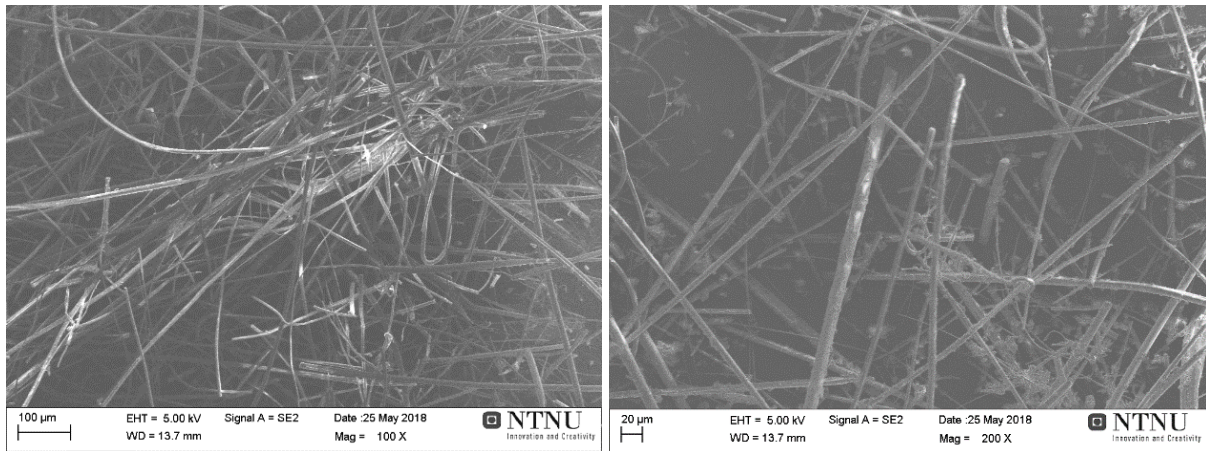
*Figure 62a-b: Waste from 12 cm over the melt, beneath 8 cm of carpet at c)100x and d)200x*



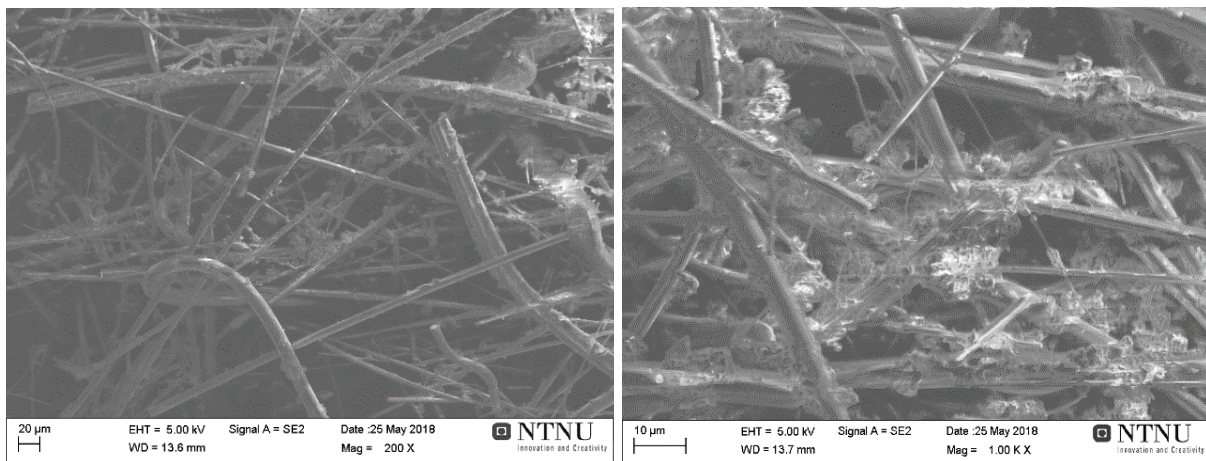
*Figure 62c-d: Waste from 12 cm over the melt, beneath 8 cm of carpet at c)500x and d)1000x*

Figure 62a-d shows the pictures of the waste from simulation 13 collected beneath 8 cm of carpet, located 12 cm above the melt. The waste has been exposed to 756 °C. Organic content can still be observed from the fiber. This is easily visible at 500x and 1000x.

**8 cm above melt – 805 °C**



*Figure 63a-b: Waste from 8 cm over the melt, beneath 12 cm of carpet at c)100x and d)200x*



*Figure 63c-d: Waste from 8 cm over the melt, beneath 12 cm of carpet at c)500x and d)1000x*

Figure 63 shows the pictures of waste collected from simulation 13 beneath 12 cm of carpet, 8 cm above the melt. This waste was subjected to 805°C. Even more organic content is observed in bigger lumps in these samples.

## 2 cm above melt – 856 °C

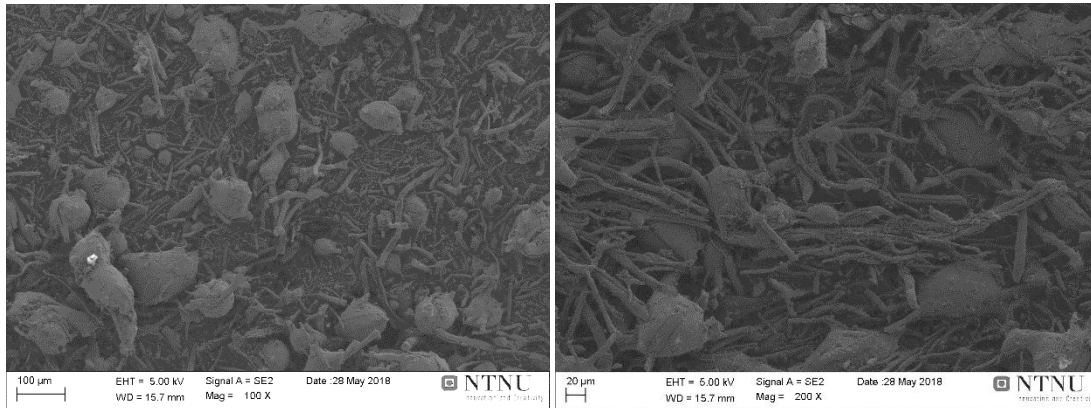


Figure 64a-b: Waste from 2 cm over the melt, beneath 18 cm of carpet at c)100x and d)200x

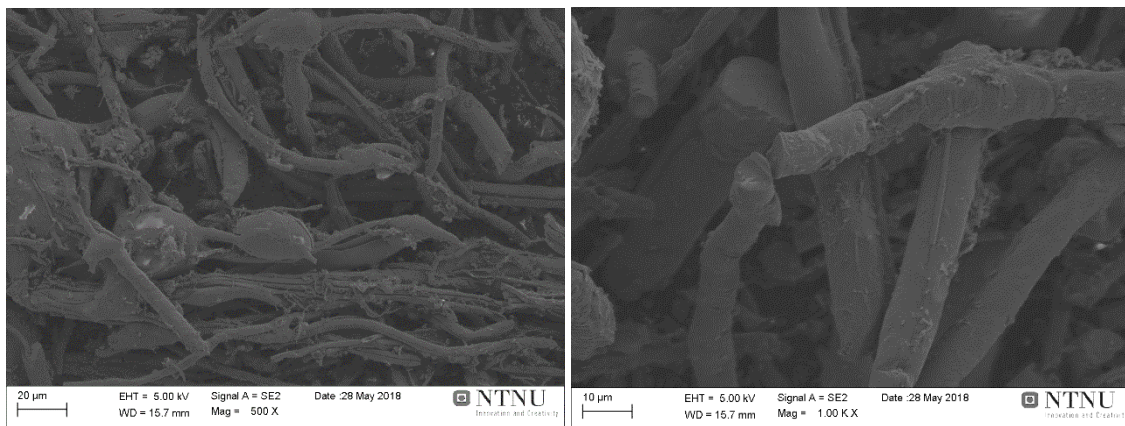


Figure 64c-d: Waste from 2 cm over the melt, beneath 18 cm of carpet at c)500x and d)1000x

Figure 64a-d show the waste collected from simulation 13 beneath 18 cm of carpet, 2 cm above the melt. The sample was exposed to a temperature of 856°C. The fibers appear to have partially melted together and broken up to shorter fibers. Not much organic content is observed, since the fibers have changed to such a degree it is difficult to compare to the other samples, and its hard to determine whether what is observed on the fibers is carbon or not.

### 4.3.2. Simulation 14

Figure 45 shows the glass wool waste after simulation 14. The temperature and distance from the melt is written above every sample. The waste at the top in the crucible is found at the top left corner, while the waste from the bottom part is at the bottom right corner. The picture shows the waste getting gradually darker as it gets deeper in the crucible. The temperature gradient from simulation 14, first presented in the experimental chapter is shown below:

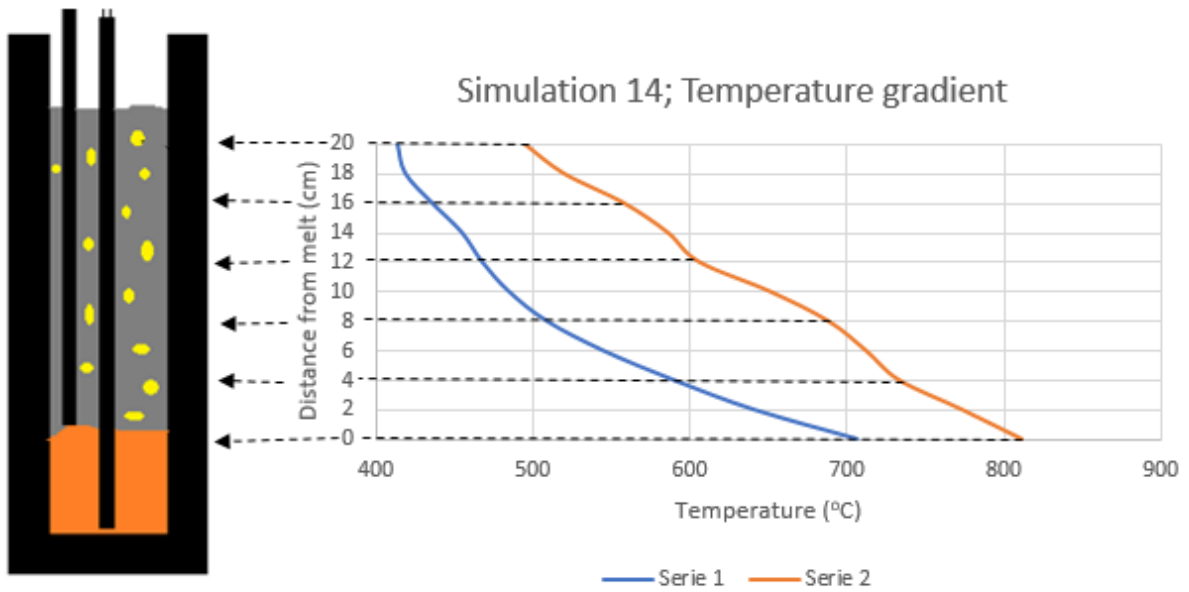


Figure X: Temperature gradient for simulation 14



Figure 65: waste dug out from the crucible. The sample at the top left corner was closest to the top, and the bottom right closer to the melt

A picture of waste gathered from 4 cm and 2 cm above the melt is shown in figure 65



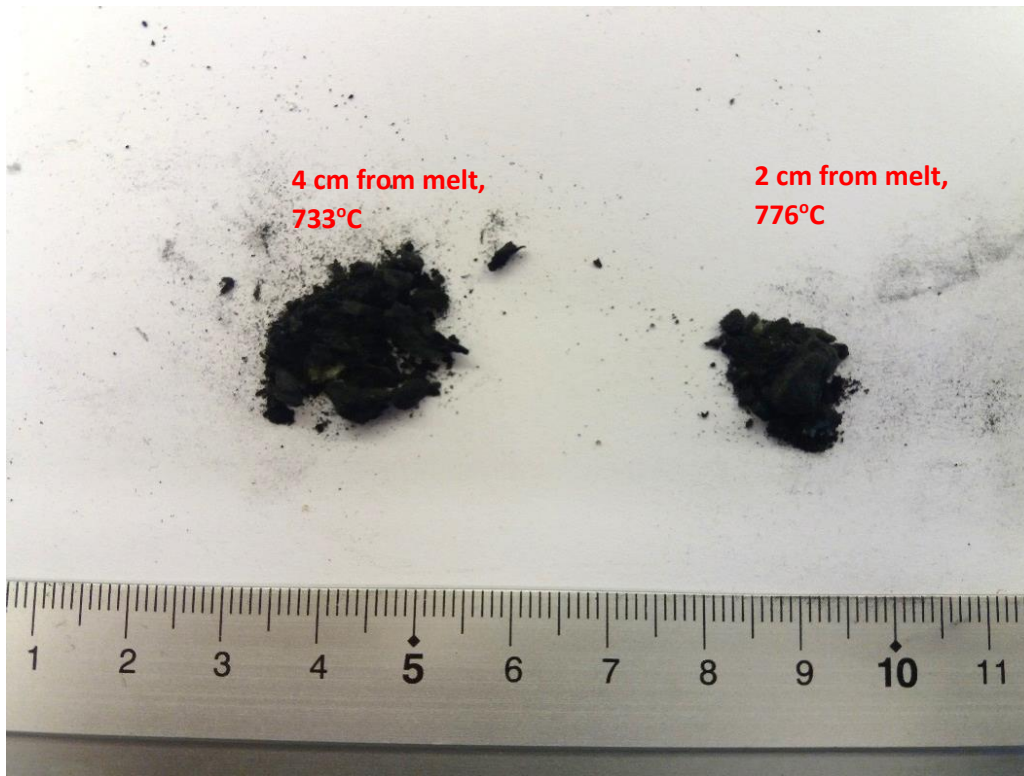


Figure 65: Waste gathered from 4 and 2cm above the melt from simulation 14.

As with simulation 13 the samples from simulation 14 shows an increasing amount of soot, closer to the melt. The samples closest to the melt have a powder like structure compared to the rest of the samples.

**16 cm above melt; 558 °C:**

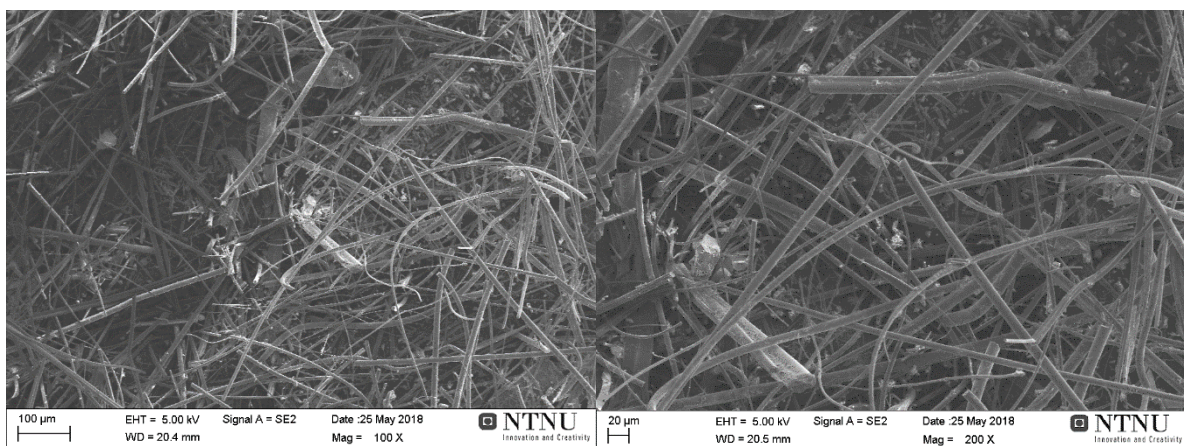


Figure 66a-b: Waste from 16 cm over the melt, beneath 4 cm of carpet at c) 100x and d) 200x

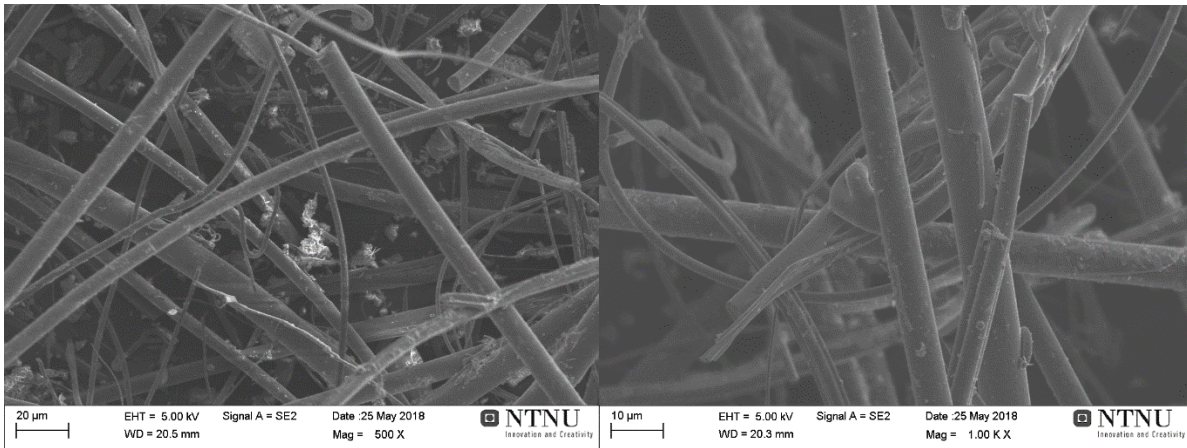


Figure 66c-d: Waste from 16 cm over the melt, beneath 4 cm of carpet at c) 500x and d) 1000x

Figure 66 shows pictures of the waste from simulation 14, beneath 4 cm of carpet, 16 cm from the melt. This sample was exposed to 558 °C and looks very much like the sample from the same depth in simulation 13. Almost no carbon is observed on the fibers.

**12 cm above melt; 606 °C**

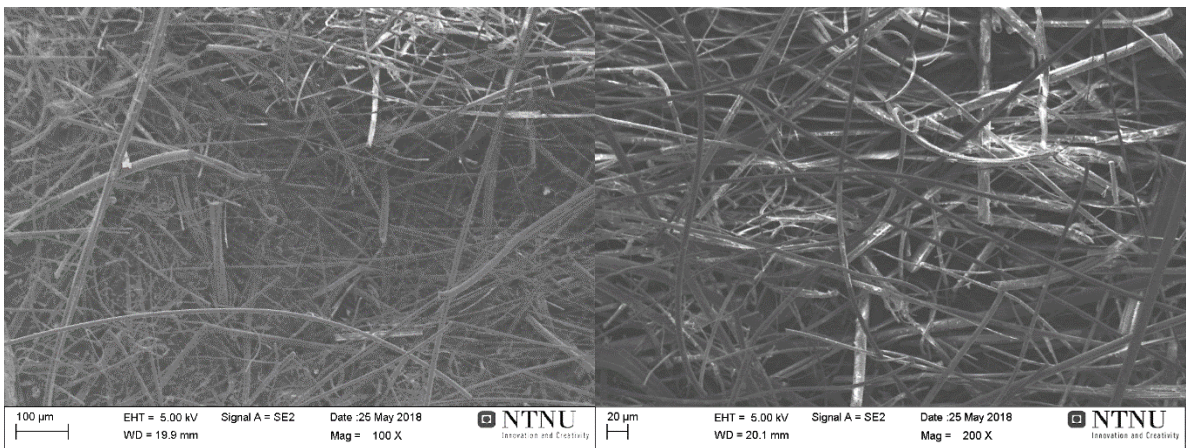


Figure 67a-b: Waste from 12 cm over the melt, beneath 8 cm of carpet at a) 100x and b) 200x

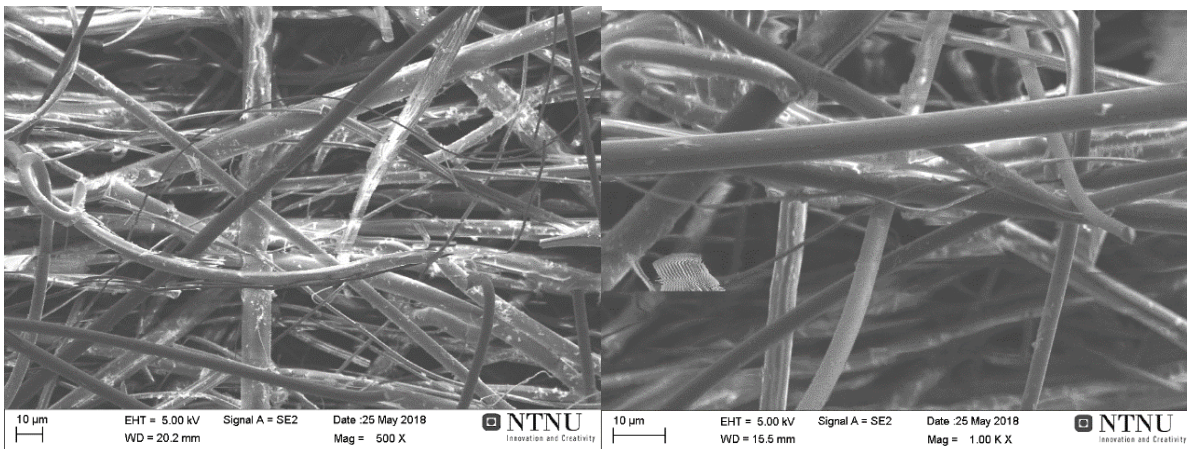
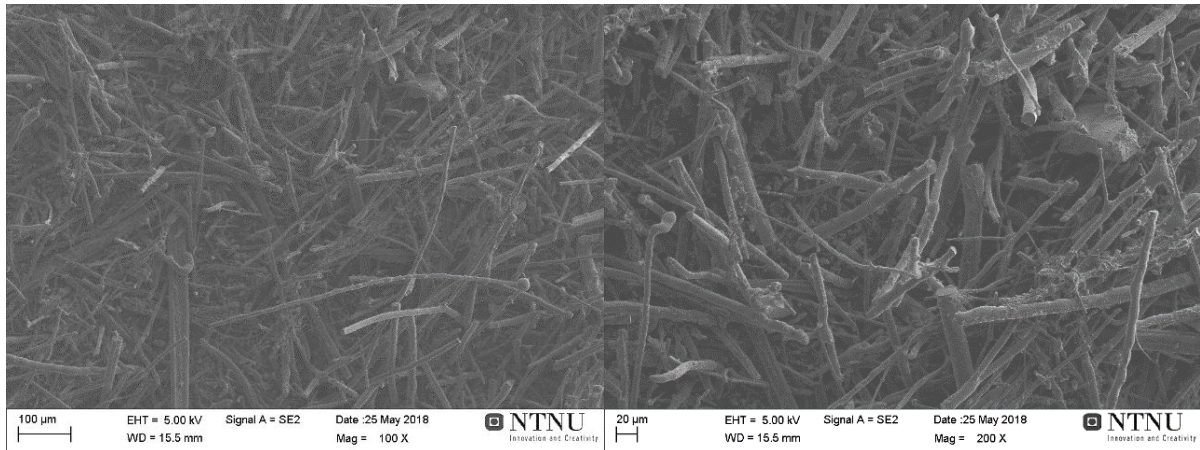


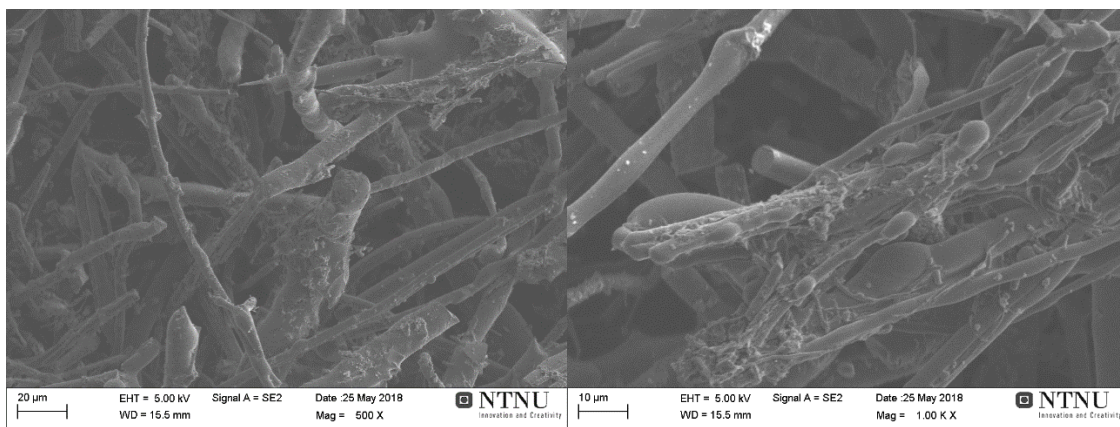
Figure 67c-d: Waste from 12 cm over the melt, beneath 8 cm of carpet at c) 500x and d) 1000x

Figure 67 shows pictures of the waste from simulation 14, beneath 8 cm of carpet, 12 cm from the melt. The sample was exposed to 606 °C. As with its counterpart in simulation 13, carbon was observed. Most easily seen in figure 67c.

**8 cm above melt – 668 °C:**



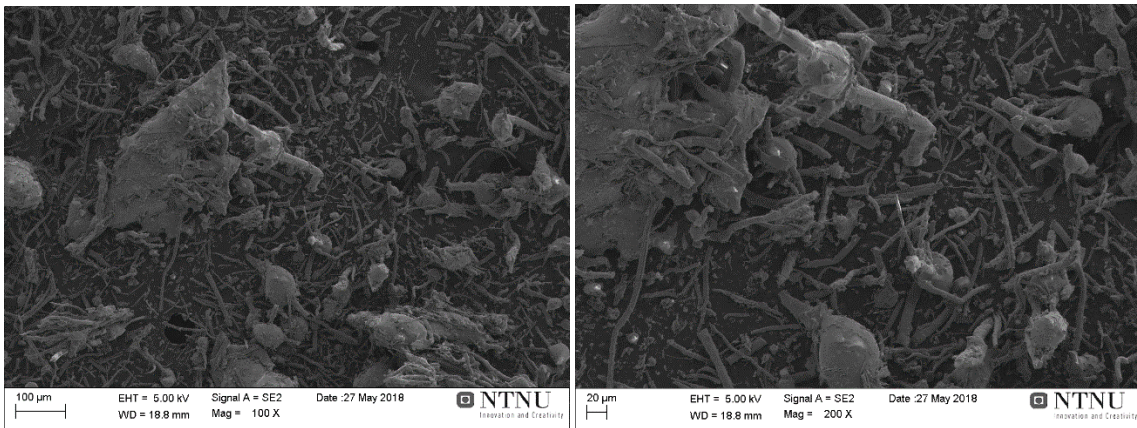
*Figure 68a-b: Waste from 8 cm over the melt, beneath 12 cm of carpet at a) 100x and b) 200x*



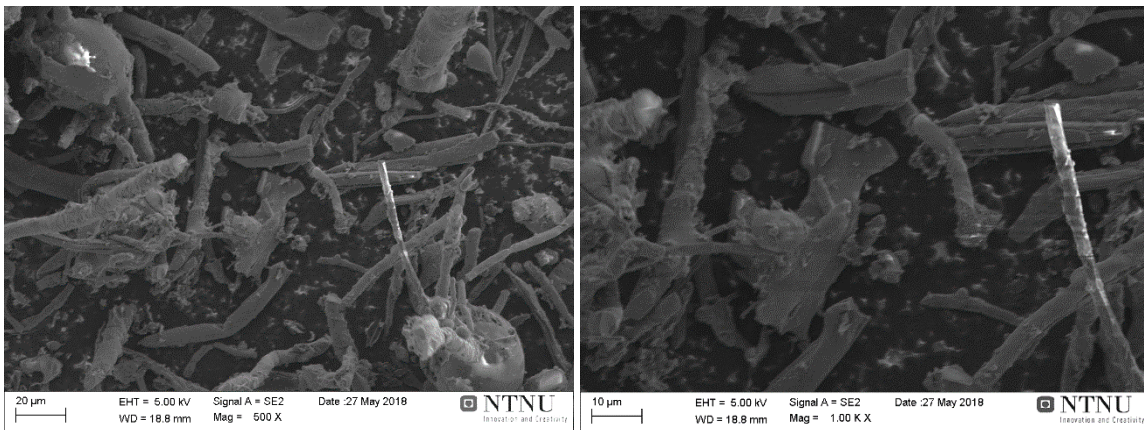
*Figure 68c-d: Waste from 8 cm over the melt, beneath 12 cm of carpet at c) 500x and d) 1000x*

Figure 68 shows pictures of the waste from simulation 14, beneath 12 cm of carpet, 8 cm from the melt. The sample was exposed to 668 °C. Larger amount of carbon was observed on the fibers in this sample.

**2 cm above melt – 776 °C**



*Figure 69a-b: Waste from 2 cm over the melt, beneath 18 cm of carpet at a) 100x and b) 200x*



*Figure 69c-d: Waste from 2 cm over the melt, beneath 18 cm of carpet at a) 500x and b) 1000x*

Figure 69 shows pictures of the waste from simulation 14, beneath 18 cm of carpet, 2 cm from the melt. The sample was exposed to 776 °C. Fiber are partially melted together. Due to the change in structure it is hard to determine the amount of carbon in the sample.

## 5. Discussion

In this chapter the result from the previous chapter are discussed. First the part of chapter will focus the burning of organic content in the waste, in both atmospheric conditions, and in the low oxygen atmosphere in the simulations. The second part will focus on the method development. The third and final part will focus on part will focus on how the results are applicable to the industry.

There are several things of interest after this experiment. The first observation is the appearance of the different samples. In the incineration experiment, the sample being exposed to the lowest temperature, the shortest amount of time at 500 °C for 3 min, shows a distinct black color assumed to be soot. Being exposed to heat for a longer time will result in a gradually lighter color, until it is light gray or white. This is not particularly surprising, considering that glass wool without any organic content is white. The conclusion that can be drawn from this is that the longer the waste is exposed to elevated temperature, and the higher the temperature, the closer to fully incinerated the waste will be. However, the carbon analysis performed by SintefMolab, shows that despite the different appearance of the samples, the amount of residue carbon in the sample is rather low in all the measured samples. The highest amount of carbon is found in sample 1, being exposed to 500°C for 3 min. However, with a weight percent of only 1,48 wt% carbon, almost all the carbon was removed. These results are supported by the weighing of the samples before and after they entered the furnace as shown in figure 47.

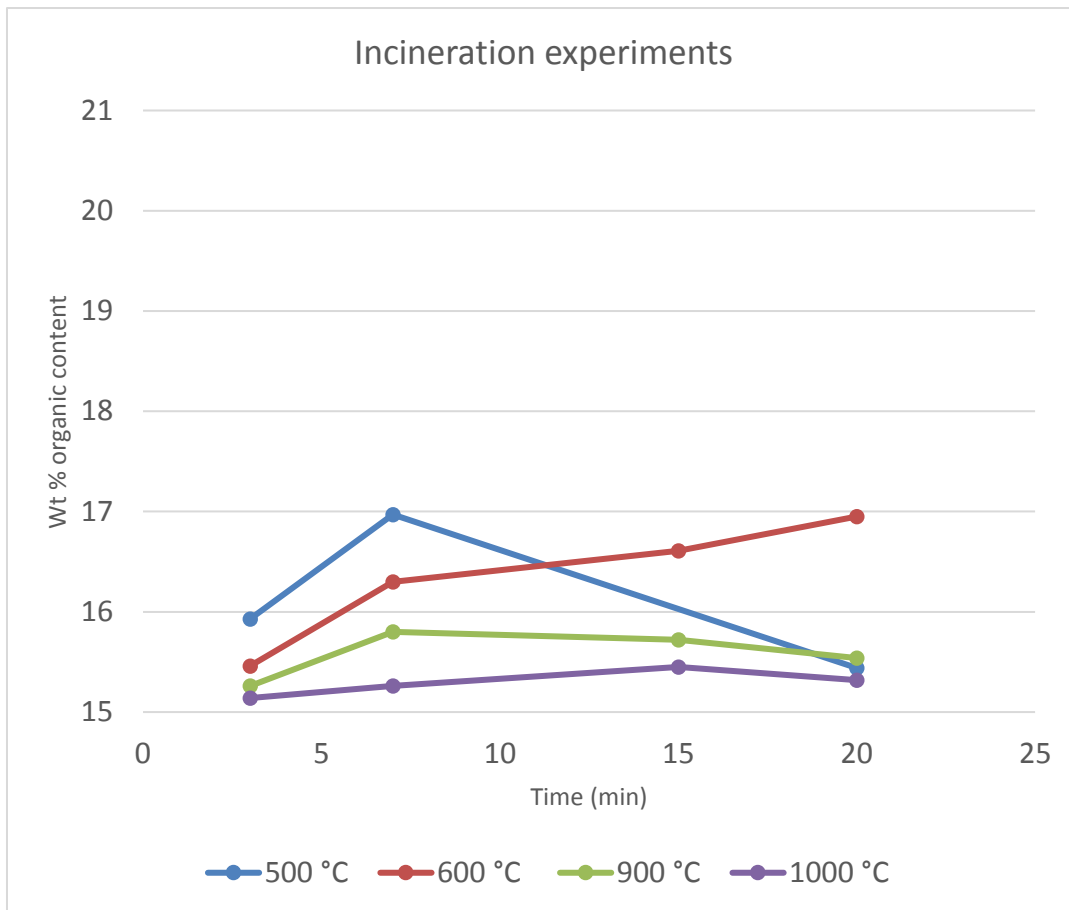
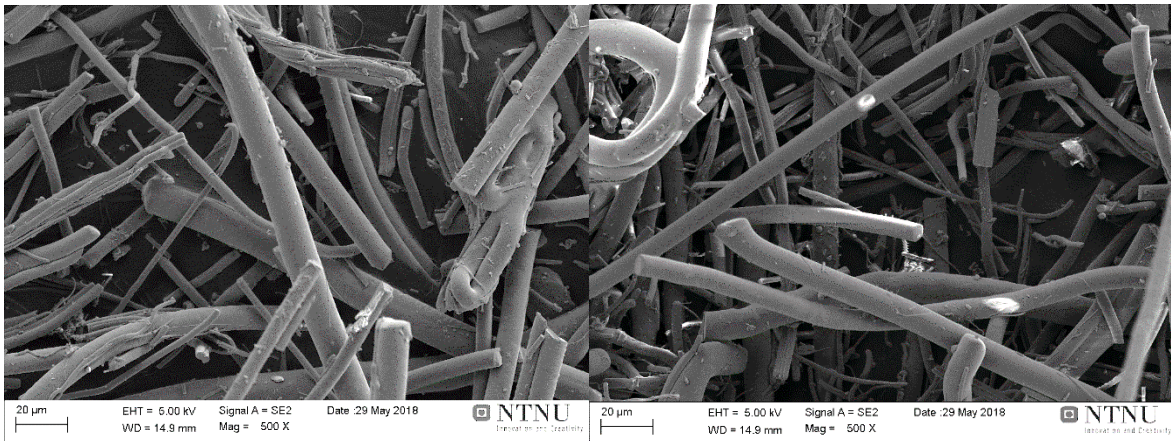
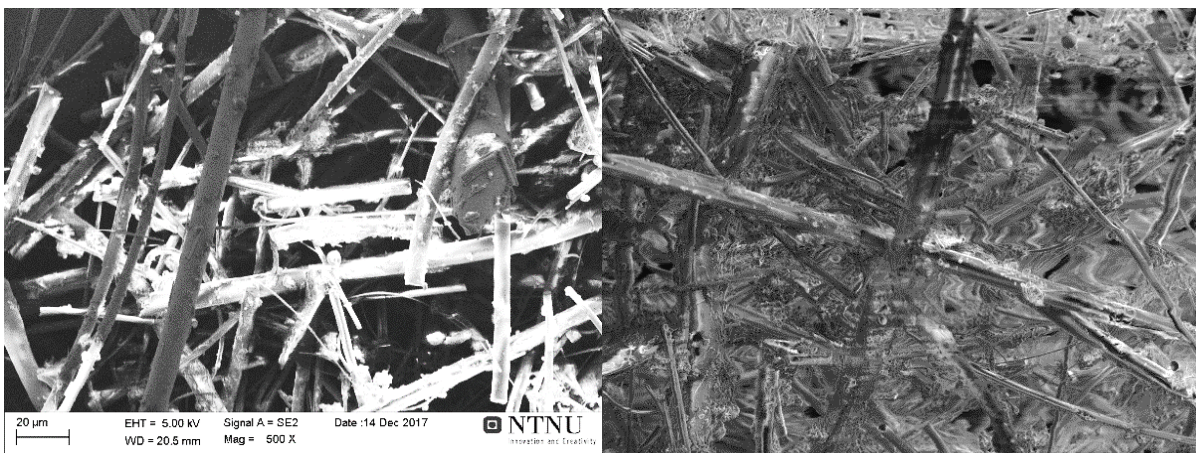


Figure 47: The wt% organic content removed from the incineration experiment. Almost no correlation between carbon removed and heat treatment in atmospheric conditions.

These results show no significant difference between any of the heat treatments of the samples, some variation between samples are seen, but this is well within what could be expected differences in the original carbon content of the samples. The conclusion that can be drawn that even some small differences are shown in weight percent, and even if the less heated samples shows soot residue, heating to 500 °C for 3 minutes is sufficient to remove almost all of the carbon in the waste, as long as sufficient oxygen is available. Furthermore, the pictures taken in SEM, show that almost no remaining carbon in the material. Figure 70a-b shows two pictures of the sample with the highest amount of residue carbon from the incineration, the one subjected to 500 °C for 3 minutes. Figure 70c-d shows for comparison two pictures at untreated glass wool waste.



*Figure 70a-b: Waste incinerated at 500 °C for 3 min in an oxygen rich atmosphere*



*Figure 70c-d: Untreated glass wool waste*

The conclusions from these pictures is collaborating the conclusions from the LECO analyses, and the weight measurements, that if the temperature is sufficiently high, 500 °C or above, the incineration process will be rapidly remove organic content, and after 3 minutes almost everything is gone, as long as sufficient oxygen is present.

The pictures from the simulation experiments show a decrease in carbon content compared to the untreated waste. However, in comparison to the waste in the incineration experiments there are still organic content present in the waste. Figure 71 shows four pictures at different depths in the crucible.

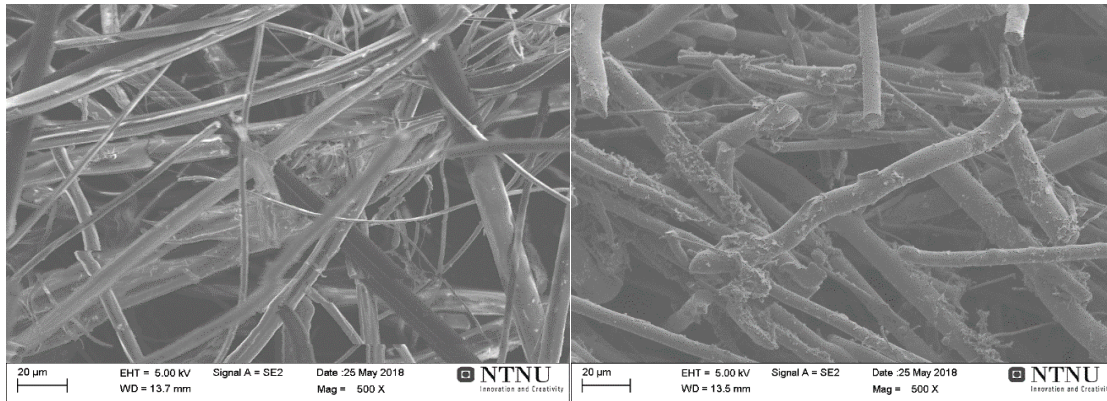


Figure 71: Waste from simulation 13 at depth a) 4 cm and b) 8 cm

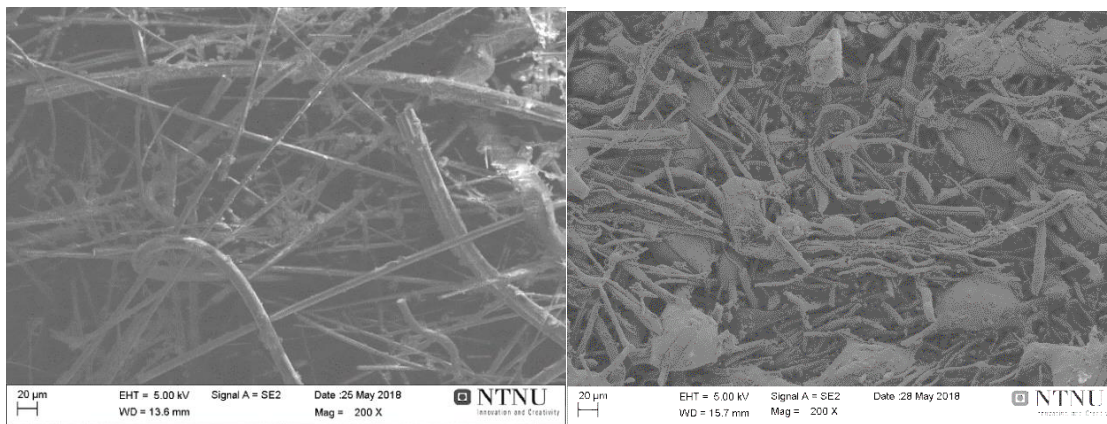


Figure 71: Waste from simulation 13 at depth b) 12 cm and b) 18 cm

Since the waste have been subjected to much higher temperature roughly between 600 °C and 800°C, for over an hour, it would be expected that all the carbon would be removed from the samples, if all the other parameters where the same as for the incineration experiments. However, since the pictures shows clear signs of residue carbon, it can be concluded that the lack of oxygen prevents a complete removal of the organic content. In particular, the samples 12 cm from the melt or closer, or in other words, samples at a depth of 8 cm or deeper appear to have its incineration process severely crippled by the lack of oxygen. The fact that the waste found deeper in the crucible were black and the waste further up was white further leads to this conclusion that the lack of oxygen hinders the combustion.



## 5.1. Method development

The method developed as a glass furnace simulation were successful in giving much useful information. The method was successful in determining the temperature in the carpet layer of the crucible, and thus made it possible to determine both the temperature the waste was subjected to as well as how deep the samples was submerged in the carpet. However, its biggest flaws, is regarding accuracy. Since all the measuring, moving of the thermocouple is done manually, and since it is impossible to look within the crucible during experiments some inaccuracy albeit small, is unavoidable. The simulations can thus not reproduce the exact same temperatures every time. Also, the degree of which the simulation can represent the industrial parameters. Since the glass melting furnace in the industry cover a much larger area, and since the glass carpet might be thinner, the industrial process would have more oxygen available than the simulations. However, the data gathered from the simulations would still be useful, to which degree adding glass wool waste is a melting furnace regardless of the exact parameters.

Since the objective were to develop a method both for this thesis and for future use, not all the simulations gave the same amount of useful data. The development process was a gradual one, where an increasing amount of success were achieved for each experiment. Discussing what worked and what didn't will provide useful information for future experiments. The first 3 simulations suffered from not having a standardized way to handle the amount of energy provided to the sample. Further, since the temperature kept increasing in the carpet layer, and since the no standard time was used for and between measurements, makes it hard to compare with other temperatures. In addition, since the whole graphite crucible was moved during measurements, made the process much more inaccurate and clumsy. This is evident in figure 19, showing the thermocouple going rapidly up and down during measurements.

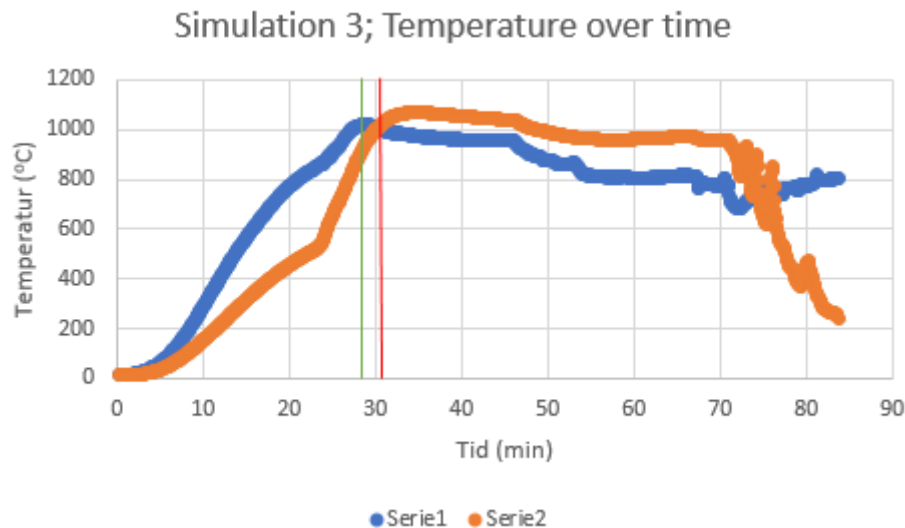


Figure 19: Temperature over time for simulation 3

Simulation 4,5,7 and 9 was conducted in very much the same way. The exception being simulation 5, where a crucible from a previous experiment was used. The result being the melted bottom layer was higher, and thus the temperature at the top layer being lower since the temperature in the furnace is generally higher at the bottom of the crucible.

Regarding the simulations containing waste, simulation 6 and 8 will not be discussed in detail. The method used did not produce any measurements to discuss, and even if it had worked as planned it did not take into consideration how the waste shrinks after being burned. This would have left the upper part without any waste.

Measurements from simulation 10, 11, 12, 13 and 14, all showed a higher temperature in top layer than the simulations without waste. This is probably partly due to the heat generated by the incineration. This is showed in figure 56:

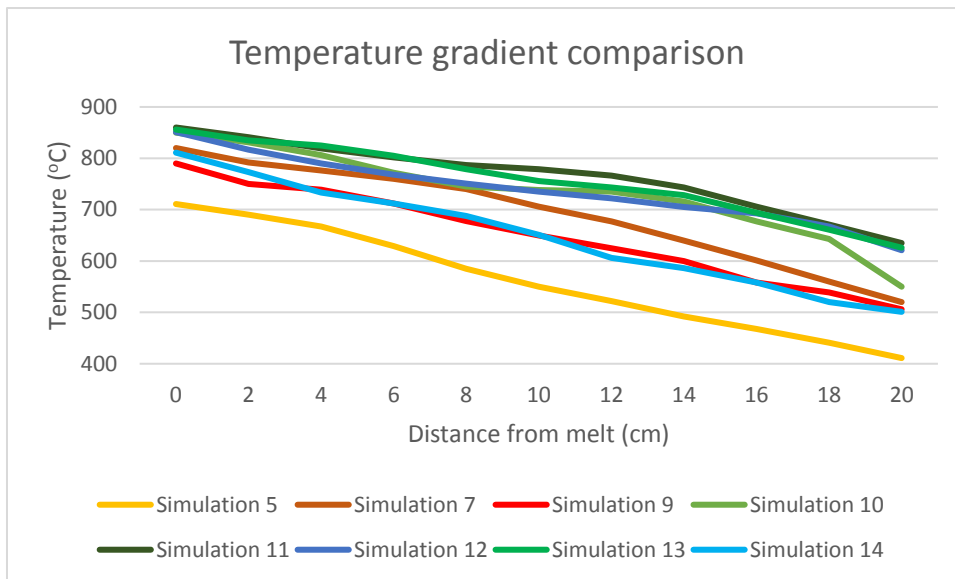


Figure 56: Temperature gradient for simulation experiments. The simulations without waste are red and the ones with are blue.

Since the flames that appeared whenever the waste was added to the crucible, where extinguished every time new raw material was added, it is clear that the carpet layer has an effect on the incineration process of the waste. However, the smoke did not disappear, and even though the intensity was greatly diminished, it lasted much longer than what it would in atmospheric conditions. When incinerated in atmospheric conditions, the sample showed no smoke after 3 minutes. In the simulations however, smoke was observed for approximately 12 minutes. This indicates that being buried in a carpet layer does not completely prevent the incineration of the organic content, but it does cripple it.

Regarding the analyzing of the samples after the simulation, filling the crucible with epoxy proved to make it difficult to observe any carbon residue in the sample when analyzed in SEM. Since the BSD most clearly distinguishes between elements of different atom weight, then the carbon-based epoxy would appear to be very similar to the residue carbon. If a method to differentiate between the epoxy and the carbon could be developed, then that might be quite useful for future experiments. However, as the simulation is at the present, the best method is to avoid using epoxy and rather analyze the samples in SE as has been done in simulation 13 and 14.

## 5.2. Industrial considerations

If the glass wool waste is to be used as raw material in the melting furnace it is several options to take into consideration. The first option would be to remove the organic content in beforehand. This can be done rather quickly in an oxygen rich atmosphere at temperatures no higher than 500 °C. However, this would include adding a new separate step to the production process which would take up extra space, resources and potentially manpower.

Another option would be to add the waste to the furnace without prior removal of the organic content. However, with such a solution care has to be taken to ensure that the waste incinerated before it reach too deep into the glass carpet. If the waste gets too deep in the carpet before all the organic content is incinerated, the lack of oxygen will hinder the removal of the remaining organics, and the carbon will reach the melt, causing gas bubbles that will disturb the carpet. Parameters to take into consideration would be thickness of the carpet, the temperature at the top of the carpet and the rate of which new powder is added, or in other words how long time it would take for the waste to be submerged and to reach a level where there is a lack of oxygen. If the carpet has a thickness above 8 cm, particular consideration has to take place, since these were the depths where most residue carbon started to appear.

Another thing to take into consideration would be whatever the water content is removed from the waste. Since it would the water would require extra energy to be removed, it reduce the temperature of the waste, and thus the incineration of the organic content might not begin until the waste have reached the depths where oxygen is lacking. Figure 72 shows the theoretical reduction in temperature in the carpet, however in these calculations it is assumed that the temperature reduction is distributed through the carpet, while in reality it might be more localized to the waste, which would increase the temperature drop.

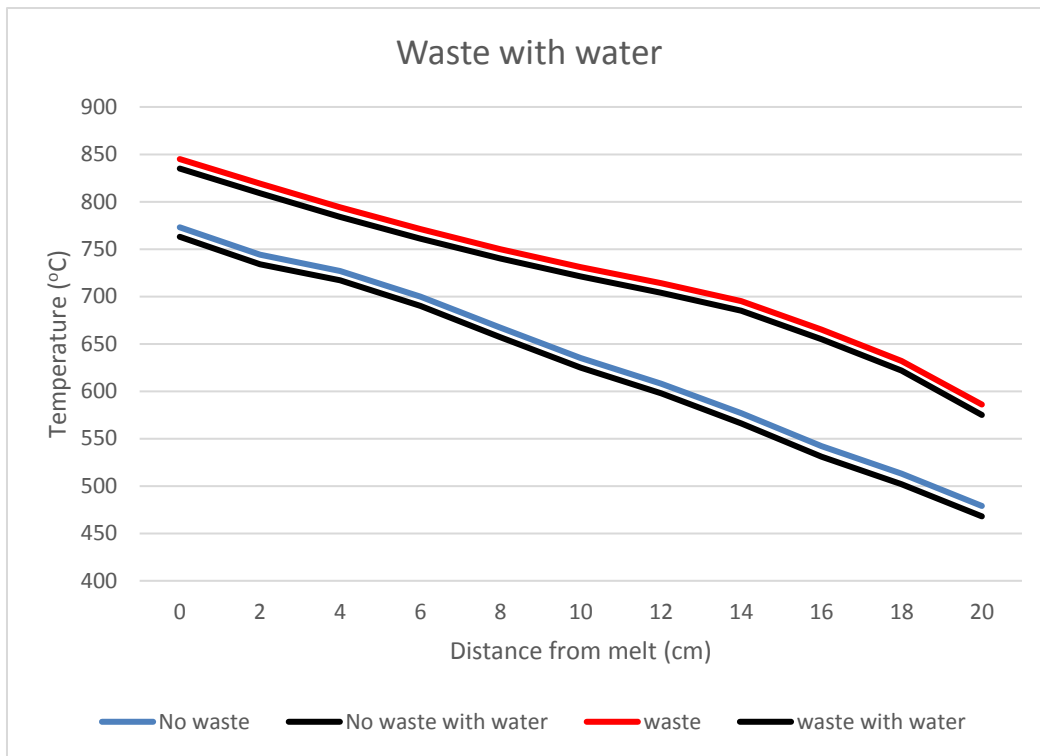


Figure 72: Average temperature gradient with theoretical addition of water



## 6. Conclusion

The low oxygen atmosphere beneath the carpet layer showed to have a limiting effect of the incineration process. In an atmospheric environment saturated with oxygen, almost all the carbon is removed within 3 minutes. However, glass wool waste submerged in raw material showed to have a significantly larger amount of residue carbon left, despite being exposed to significantly higher temperature for a much longer time than the glass wool from the oxygen rich incineration experiments. This is due to the lack of oxygen. The structure of the glass closest to the melt, and subsequently the part subjected to melting showed to have been partially melted. The fibers were significantly shorter, and randomly oriented, in comparison of the other waste in which the fibers were to a larger degree bundled together.

The simulations proved to give useful results. The measuring of the temperature gradient made sure it was possible to know both the temperature and as well as the depth of the waste. However, since the whole measuring process and time management was done manually, some inaccuracies are to be expected. If further improvements are to be made it should be in these areas.





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