Wood ash as a resource in lightweight concrete blocks and

thermal runway as a possible production method for lightweight aggregates

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Preface

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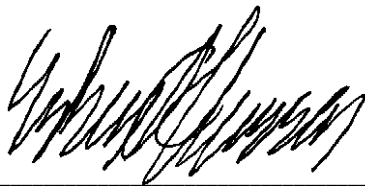
Project period:

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1. PREFACE

This thesis will, with the addition of an oral presentation, represent the result of a master project, concluding a master degree in civil engineering for Esben Østergaard Hansen. The thesis is weighted as 30 ECTS-points, and have been completed between the 25/01-16 and the 16/07-16.

The thesis is written in parallel with the thesis of Randi Juel Olsen, and a great gratitude is directed towards her. Almost all of the experiments have been made together with her, and all of the results have been discussed between us. Without her, this thesis would have never come to be.

In addition, I would like to thank supervisor Lisbeth M. Ottosen, for great and constructive guidance, lab technician Ebba Cederberg Schnell for fast and competent help in the lab and concrete technician Per Leth, for being a large help in the concrete lab. I would also like to thank Jesper and Jesper from Weber Saint-Gobain, for help with materials, recipes, and competent guidance in working with Leca.

Lastly I would like to thank my family and friends for support and encouragement, and Simon Svensson and the rest of the project family for helping to create an awesome atmosphere around the project.

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2. ABSTRACT

The report is divided into two parts. Part 1 is about replacing cement with wood ash in lightweight concrete, and part 2 is about using thermal runaway to produce lightweight aggregate from sewage sludge ash.

For part 1 two different ashes were considered, one from Køge Kraftvarmeværk and one from Amagerværket. They were both examined, and the one from Amagerværket was picked, as it had the lowest loss on ignition and solubility. It was then washed, as the solubility was still around 13 %. During washing the somewhat separated, and also the LOI increased from 2.6 % to 12 %. The reason for this increase is unknown, but it is speculated, that some of the mass lost is actually carbonate and not organic matter. The ash was used anyway. The ash was further analyzed, and was found to be very similar to cement in regards to size, but it did contain high levels of some heavy metals, including Cd and Zn.

A mix design was acquired from commercial producer, but as it did not include a precise amount of water, the absorption rates for the lightweight aggregates was found, and some test casts were made, until a satisfactory mix design was obtained. As the water requirement for ash is not the same as that for cement, an activity factor, purely relating to the consistency, for the ash was found so the W/C would be the same for the mixes containing ash. It was found to be 1.9.

The casting of the blocks was done on a small scale block machine, but not before the casting method was refined, to ensure the best quality block possible. Blocks were cast with cement replacement from 15 % to 40 % with a 5 % increment. Using commercially available blocks, the test methods for the different analysis were refined, and the blocks were tested for compressive strength, porosity and density, capillary suction and leaching.

It was found that up until 25 % cement replacement, the effect of the ash on the compressive strength was minimal, as it is the lightweight aggregates that are strength defining, and that the ash had no adverse effects on the density, porosity or leaching. The heavy metals found in the ash, stayed bound to the concrete to a degree, where the block was harmless. The ash did increase the capillary suction, but as this is not regulated, it was not deemed a problem. The high LOI did not seem to effect the blocks negatively.

Part 2 started out as failed attempt at drying out some 0-2 mm lightweight aggregate in a microwave oven, which let to it sintering together and expanding. It was found that the phenomenon of thermal runaway was to blame for the heating, and carbonate for the voids. The concentration of carbonate was checked for both raw LWA and the nut that had formed, but they were low enough that the test method did not work. This led to the conclusion that only a small amount of carbonate was needed for the process to occur.

As thermal runaway mostly occur in a small number of metal-oxides, the concentrations of these metals were found in the LWA, and compared to those of a sewage sludge ash. As there was a higher concentration of all of the different metals, especially iron, tests were conducted on the ash. It was found that it too was capable of going into thermal runaway, and creating something that resembles LWA.

3. CONTENTS

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4. STRUCTURE OF REPORT

This report contain 2 parts that are separate, but still work towards the same overall goal; to use bio ash in the production of building materials. More specifically the materials concerned in this report are lightweight aggregates, and lightweight aggregate blocks.

Part 1 is the main part of the work done for this project, and is about using a wood ash as a cement replacement in lightweight aggregate concrete. This part of the project has been done in collaboration with Randi Juel Olsen, as her thesis concerns the same subject. As many of the experiments have been made for both projects, some of the tables and figures will be near identical, and these can be seen in the list below

Tables	Figures
7-4	8-6
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8-9	8-15
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The same goes for the chapters on further research and sources of error, as they were co-written.

Part 2 is a preliminary investigation in using the phenomenon of thermal runaway in microwave ovens to produce a form of lightweight aggregate from sewage sludge ash.

Both parts are independent of one another, and will contain sections on theory, materials and methods, results and discussion, and a conclusion. The sections on sources of errors and references are shared however.

Master Thesis - Esben Østergaard Hansen

Structure of Report

Part 1

5. INTRODUCTION

Cement production is responsible for around 5 % of the CO_2 caused by humans, and with the increase in construction, this amount will only get higher. As the adverse effects of climate change have already begun to show, and will only get worse, it is crucial that steps are taken to reduce the carbon footprint, in all aspects possible. A reduction in the CO^2 emitted by the building industry could come from using less cement, and instead replacing it with other materials, like bio ash, that at the moment is a waste product.

Using ash as a hardening agent in construction, is not a new idea. Some of the oldest structures still standing today contain volcanic ash, including the Mohenjo Daro and Pantheon. Today fly ash from coal is used in a lot of construction to the point, where the price is starting to go up. If bio ashes could be used as well, both supply and possibilities would go way up.

Lightweight aggregate concrete is used in a lot of construction, as it is light and versatile. As it is not designed to have a very large compressive strength, a small reduction in strength due to the replacement of some of the cement, is acceptable. This potentially means, that a large fraction of cement could be replaced, without limiting the usability of the product.

The aggregates, the blocks and the recipe used in this project comes from Weber Saint-Gobain. They are a large international company, which deals in many different materials, of which lightweight aggregates and lightweight aggregate block are just two of them. They produce the aggregates and the blocks in two separate factories in Jutland, Denmark, and have kindly donated the materials used in this project. The blocks that they produce have a compressive strength set to 3 mPa, but they expressed interest in blocks with a strength of 2 mPa or higher.

6. THEORY

In the following, the theory behind the experiments done in part 1 will be explained.

6.1 LECA

LECA stands for Light Expanded Clay aggregate, and is both a type and a brand of lightweight aggregates (LWA). It is a brown, round, nut-like aggregate, and as hinted in the name, it is made from clay that is expanded under high heat. The process, which will be described in more detail in the theory section of part 2, yields many different sizes of Leca, and immediately after production, it is divided by size. The different sizes used in this project, can be seen on Figure 6-1.

Figure 6-1 - Leca 0-2 mm, 2-4 mm and 4-10 mm.

Usually the Leca will be referred according to the size, so the size fraction of 0-2 mm will be called Leca 0-2 or LWA 0-2.

The two larger fractions of LWA are as described earlier, round and nut-like, while the smallest size, did not fit this description. The 0-2 seems to be mostly made up of dust, and broken pieces of larger nuts, rather than small nuts in itself.

Leca usually has a bulk density between 200-600 kg/m^3 depending on the size, and is mainly used as aggregate in lightweight concrete, and as a capillary breaking layer under buildings.

From here on the Leca will be referred to, by the general term LWA.

6.1.1 Water content and absorption rates

As the LWA is made, by expanded gas inside it, it has a sort of honeycomb structure as seen on Figure 6-2.

Figure 6-2 - LWA cut in half

All of these voids means that the LWA can absorb a large amount of water, the amount and rate of which is of significance when casting lightweight concrete. The water to cement ratio (W/C) is very important for the strength and workability of a concrete. If an unknown amount of water is sucked up by the LWA, it is impossible to know the W/C, and the concrete will behave unreliably. For this, the absorption rates for the LWA must be known.

The method for finding the absorption rate, that involves soaking dried aggregate in a pycnometer, is more detailed described in section on materials and methods, but the following measurements are found:

$M_1(24h)$ mass of saturated and surface-dried aggregates after 24 h

$M_2(24h)$ mass of pycnometer, water and saturated aggregates after 24 h

M_3 mass of pycnometer and water as calibrated

M_4 mass of dry aggregate

From these the absorption for 24 hours can be found using:

$$WA_{L24} = 100 \frac{M_1(24h) - M_4}{M_4}$$

The absorption for any given time within the 24 hours can be found, given that the pycnometer has be weighed at this point. It is found using:

$$WA_{Lt} = WA_{L24} - 100 \frac{M_2(24h) - M_2(t)}{M_4}$$

where:

M_2 mass of pycnometer, water and aggregates at given time

If the pycnometers are weighed a couple of times during the 24 hours, it is possible to fit a curve to the points, and from that find the absorption at any time within the 24 hours.

In addition to the absorptions, these measurements can also be used to find the following densities:

ρ_{La} apparent particle density

ρ_{Lrd} oven-dried particle density

ρ_{Lssd} saturated and surface-dried particle density

using these formulas:

$$\rho_{La} = \rho_w \frac{M_4}{M_4 - (M_2(24h) - M_3)}$$

$$\rho_{Lrd} = \rho_w \frac{M_4}{M_1(24h) - (M_2(24h) - M_3)}$$

$$\rho_{Lssd} = \rho_w \frac{M_1(24h)}{M_1(24h) - (M_2(24h) - M_3)}$$

where:

ρ_w density of water at test temperature

6.2   CONCRETE

Concrete is one of the most widely used building materials, mainly because of a relative low cost and a high versatility. It is in the simplest form composed of three things: cement, water and aggregate, usually sand and stone. It is the cement that combined with the water create the strength, and binds it together, but cement is also the most expensive part. This is one of the reasons that the aggregates are added.

6.2.1   W/C

One of the most important parameters in concrete design, is the W/C ratio. It is a measure of how much water there is pr. unite of cement, and it is crucial for the strength and the workability of the concrete. If the ratio is high, the concrete will be wet and easy to cast, but will also develop less strength than a dryer mix. If a concrete is dry instead, it will be very strong, but hard to cast. This is the reason that plasticizers are added to high strength concrete, to make it easier to cast.

If the concrete consists of nothing but water, cement and aggregates, the W/C is easily found by dividing the water content with the cement content by weight. But if the concrete contains other things, such as fly ash or microsilica, the equation looks like this:

$$\frac{W}{C_{eq}} = \frac{W}{C + 0.5 \cdot PFA + 2 \cdot MS}$$

where:

W mass of water

C_{eq} equivalent mass of cement

FA mass of pulverized fuel ash (coal)

MS mass of microsilica

This is the formula for the equivalent water/cement ratio. The factors multiplied to the fly ash and micro silica content is called the activity factor. It is a measure, both for the impact on the consistency on the fresh concrete, but also the impact on the hardening process, and the final strength of the concrete.

6.3   LIGHTWEIGHT CONCRETE

In a lot of applications, ordinary concrete is a lot stronger than it actually needs to be, and here it can be beneficial to optimize for some other trait, such as weight. That is exactly what lightweight concrete does. Here the aggregate is not ordinary stone, but the LWA discussed earlier, and the structure is open, as can be seen on Figure 6-3.

Figure 6-3 - Standard LWA block

While standard concrete weighs about 2500kg/m^3 and has a strength of around 20 mPa, LWA concrete only needs a strength around 2-3 mPa, and it normally only weighs around 600 kg/m^3. This weight makes it possible to move and place blocks of a relevant size by hand, which is a huge advantage. The strength seems low, but for nonloadbearing walls and foundations in smaller buildings it is sufficient.

6.3.1   Compressive strength

To find the compressive strength of any material in mPa, there are two parameters that needs to be known; the force used to break the material, usually in kN, and the area over which the force works. Knowing these, the strength in mPa can be found by

$$Stength\ in\ mPa = \frac{force\ in\ kN \cdot 1000}{area\ of\ compression\ in\ mm^2}$$

For that reason, it is ideal to have smooth surfaces to test on, as this will provide an easily measurable area of compression. However, when working with LWA concrete, smooth surfaces are rare to come by. The cast surfaces of a LWA block, are often bumpy and uneven, so to do a test directly on the surface would yield in a much smaller area of compression than expected.

A partial solution is to cut off a thin slice of the surfaces, but seeing as the structure of a LWA concrete block is so open, it does not entirely solve the problem. A principle sketch of an uncut and a cut surface can be seen on Figure 6-4.
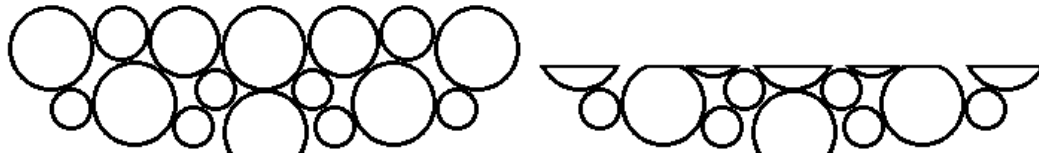
Figure 6-4 - Uncut and cut surface, principle sketch

As can be seen, the cut surface is still a bit uneven. The solution for this is to put pieces of light density fiberboard, between the specimen and the compression plates. The fiberboard will deform under the pressure, filling out the voids, and providing even pressure to all of the surface, as seen on Figure 6-5.
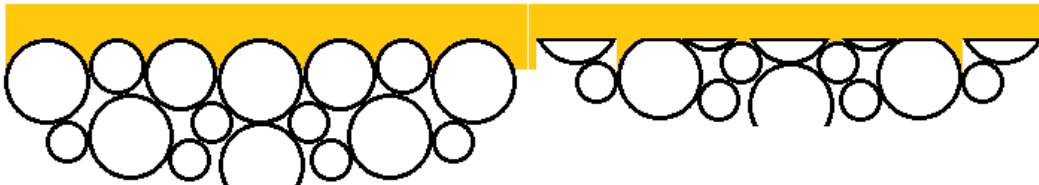
Figure 6-5 - Uncut and cut surface, compressed with light density fiberboard

When testing concretes compressive strength, there are two common sample shapes; cubes and cylinders. Cylinders are the most versatile, as they allow for samples to be easily drilled out of existing structures, they are easy to make molds for, and they are easy to cast. In this project, all the experiments will be done on cylinders.

6.3.2 Height/Diameter ratio

When testing a concrete cylinder for compressive strength, it is of course important to know the diameter of it, as this gives the area over which the force is applied, but it is almost equally important to know the height of the cylinder. Test methods for standard concrete [1] calls for the cylinder to be double as high as it is wide; a height to diameter ratio of 2. This is due to the fact that the strength determining failure mode is cracking. An optimal failure mode is where the sides of the cylinder breaks away, as illustrated on Figure 6-6.
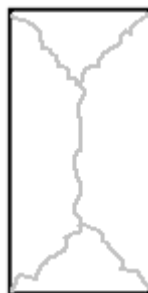
Figure 6-6 - Failure mechanism of ordinary concrete sample

As can be seen on the figure, this height gives the cracked off parts of the sample, plenty of space to move out of the compression zone. Had the sample been smaller, they might get stuck, and end up being crushed, causing an artificial high strength readout.

However, when working with LWA concrete, this is not the case. According to DS/EN 1354 [2], the height to diameter ratio only needs to be 1 for LWA concrete. Due to the relatively low strength of the LWA, it is more likely to fail by crushing, than to cracking, and so an H/D of 1 is more than sufficient.

6.3.3   Density and Porosity

As described above, one of the main goals when making LWA concrete, is to make it light, and so the density is a very important parameter. For this project, two ways of measuring a density are discussed.

Firstly the object in question can be weighed and measured, a volume can be calculated and a density can be found.

The other method involves first weighing the object dry, saturating it with water and weighing it above and below water. It has the added benefit, that it can also be used to find the porosity. Using the measured weights, the following values can be found:

Volume:

$$V = \frac{m_{ssd} - m_{sw}}{\rho_w}$$

Volume of open pores:

$$V_{po} = \frac{m_{ssd} - m_{105}}{\rho_w}$$

Dry density:

$$\rho_d = \frac{m_{105}}{V}$$

Porosity:

$$p_o = \frac{V_{po}}{V}$$

where:

ρ_w density of water

m_{105} mass, dried at 105°C

m_{ssd} mass, saturated surface dry

m_{sw} mass, saturated under water

While the first method is good for objects that cannot be weighed under water, the second is good for objects or irregular shape that are hard to measure.

When working with LWA concrete however it gets more complicated. Due to the relatively low strength, it can sometimes be hard to get a sample of regular size as can be seen on Figure 6-7, which makes the first method next to impossible.
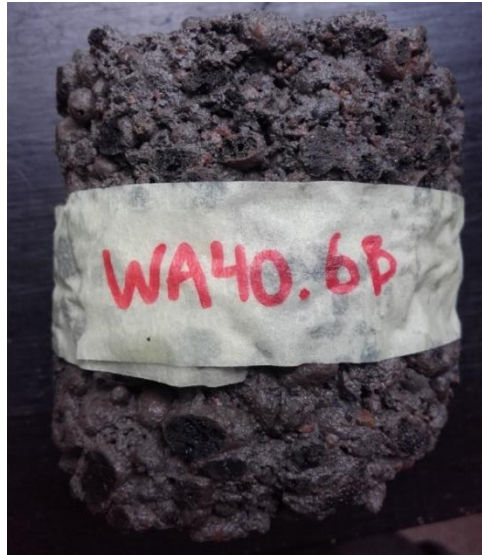
Figure 6-7 - A very irregularly sized sample of LWA concrete

However due to the very open structure of LWA concrete, it can be hard to weigh it above water in a saturated state. As soon as it is taken out of the water, it will start to drain, causing an error in the measurement.

This issue has no set solution, and so it will have to be looked into.

6.3.4   Capillary Suction
The capillary suction is a measure of how much, and how fast water is sucked up into a material. It is found by placing the samples in water, and then recording the weight gain from water. From this, the absorbed water per area can be found using:

$$Q = \frac{m_t - m_0}{A}$$

where:

Q sucked up mass per area

m_t mass, at time t

m_0 mass, dry

A area in contact with water

With enough measurement, a graph of the capillary suction can be drawn. The capillary suction is not regulated on LWA blocks, but it would still be interesting to see, what effect the addition of ash will have.

6.4 ASH AS A CEMENT REPLACEMENT

6.4.1 Cement

Cement is an artificially made powder, usually gray in color, which is very reactive with water. Once the two are mixed, it starts a hardening process in which tiny crystals are formed in what is known as a pozzolanic process. These crystals interlock, and create a strong gray mass known as cement paste.

Cement was first invented in the early 1800's, but the pozzolanic process has been known for a long time, as it is also seen in volcanic ash.

6.4.2 Replacing Cement with Ash

The idea of replacing cement with ash is not a new one. As the first cement came from ash, it was natural to test ashes for their cement-like properties. This led to the widespread use of pulverized fuel ash (PFA) in concrete, instead of some of the cement. PFA is, as it says, ash from the burning of pulverized coal. It looks very similar to cement, and has the same pozzolanic effect, although the strength in PFA develops slower than cement. As PFA can successfully be used as a cement replacement, it is not unlikely that other ashes might also be useable.

In this project, two different wood ashes are considered. The first one is from Køge Kraftvarmeværk, a power plant, and is made from the burning of leftover wood form a wooden floor production. The second one of from Amagerværket, also a power plant, but here the fuel consists of wood pellets.

Both ashes has been investigated before, but they are still to undergo an analysis prior to using, as it is not sure that they are consistent with how they were last time they were tested. This is especially true with the ash from Køge, as it varies a lot what kind of wood is burned, depending on what the wooden floor factory is making, and this can have an influence on the ash.

7. MATERIALS AND METHODS

For some of the experiments done in this project a well working method was already known, and getting reliable results was straight forward. For others, it was a long process of multiple iterations to get to a method that produced trustworthy results. The same goes for the ash used in the project; it was a process of trial and error, to figure out what ash to use where. In the following the processes of getting to the right methods will be illustrated using a sort of map, describing the different options available.

7.1   ASH FOR BLOCKS

Two ashes were considered to be used in this project. Both are wood ashes; one is from Køge Kraftvarmeværk and the other is from Amagerværket.

When using ash as cement replacement, there are a lot of things that should be tested. As the institute has long worked with ashes as cement replacement, there is a standard array of tests to be done on each ash. These are both to determine the quality of the ash, and to assess how toxic the ash is to the environment, as it often has a high concentration of heavy metals.

7.1.1   Characterization of Ash

The following is a short description of all of the experiments involved in characterizing the ash. The full guide to the experiments can be found in the appendix P1-ME-01-04.

7.1.1.1   Water content

The ash is sometimes sprayed with water at the power plant, to avoid dust, and so it can be wet. To find the water content, three samples were weighed out and dried at 105° C for 24 hours, and weighed again. From this the water content can be found.

7.1.1.2   Water solubility

When using ash as a cement replacement, it is important to know how much ash is added. This can be difficult if a part of the ash is water soluble, as this part will be dissolved in water during casting. Therefore the solubility of the ash is found.

This is done by adding a set amount of ash to a flask together with distilled water, shaking it, and letting the ash settle to the bottom. After this the water phase is decanted off through a filter, new water is added, and the flask is shaken again. This process is repeated at least 3 times. After this, all the ash is added to the filter, and left to drip dry for 24 hours, and then placed in an oven for 24 hours at 105 °C to dry completely. After this the ash and the filter is weighed and a solubility can be found.

7.1.1.3   Loss on ignition

The loss on ignition for the ash, is a measure of how much weight the ash loses when burnt at a high temperature. The loss on ignition gives a good estimate of the content of organic matter in the ash, as this is the part that will vaporize at the used temperature. The typical method for measuring the loss on ignition of ashes to be used in concrete at the institute is a modified version of DS/EN 204 [3], in which the ash is burned at 550 ° C for 1 hour, and the weight loss is recorded on a scale accurate to 0.0001 g. However, during a test done on some of the larger

particles of one of the ashes, it was noticed that they had not fully been burned, as can be seen on Figure 7-1.

Figure 7-1 - Larger pieces of ash, after 1 hour at 550° C. The black parts are unburned

Because of this, the time was changed to 2 hours.

7.1.1.4   Particle size distribution

For the ash to work well as a cement replacement, it is best, that it has roughly the same particle size distribution as cement. The distribution is measured using a machine three times, and an average is found.

7.1.1.5   Micro and Macro elements – ICP

The content of some chemical elements is to be determined, both because it can give a clue as to whether the ash is usable as a cement replacement, and because there are laws and regulations controlling the concentration of some harmful elements. To measure the concentrations, a small amount of ash is mixed HNO_3, after which it is boiled under pressure in an autoclave. This releases all of the acid soluble elements into the liquid, which is then filtered from the solids, and analyzed on an ICP.

7.1.2   Preparing ash to be used

From the analysis of the water solubility, it is found, that the ash in its raw state, is not fit for use as a cement replacement. The following section will be on the preparation of the ash.

7.1.2.1   Washing of the ash

The ash was found to have a very high solubility. This could cause problems in using it as cement replacement, as the water soluble fraction will dissolve in contact with water, and the mix will be left with less binder material than expected. Therefore it has been found to be a good idea to wash the ash.

In principle the washing method is the same as the method for measuring the solubility. However, as the solubility-setup only allows for 100-150 g of ash, and the required amount of ash is in the kilos, an up scaled version has been used.

The larger scale washing is done in a large pot, which can be seen in Figure 7-2.

Figure 7-2- Pot used for washing ash

To the pot was added ~20 l of ash, and 60-70 l of water. The slurry of ash and water was stirred using a hand drill with a mixer attachment, and then left to settle. The pot is fitted with valves at different heights, so that the water can be drained off, without having to tilt the whole pot. This allowed for a large fraction of the water to be removed without losing much of the ash. A portion of the drained off water was filtered, and the conductivity was measured. The pot was refilled with water, and the process was repeated until the conductivity of the washing water had leveled out. This was taken as a sign that all of the water soluble fraction of the ash, had been washed out.

Once the conductivity had leveled out, the pot was drained of as much water as possible, and moved to a large oven set to 105° C, to dry off the remainder of the water.

To confirm that most, if not all, of the water soluble material had been washed out, the concentrations of chloride, sulfate and nitrate in the washing water were measured.

7.1.2.2   *Mixing of the ash*
After the ash had dried out as much as possible, while still in the pot, it was scooped out. It was clear however, that 36 hours at 105° C was not sufficient to dry it out completely. This is also contributed to the relative small surface area, compared to the large volume of ash. The ash was added to a large metal tray to increase the surface area, and put back in the oven for an additional 24 hours. After this, is was completely dry.

It became clear, when emptying out the pot, was that the ash had somewhat separated. The bulk of it was a warm brown color, very soft, and the clumps that had formed were easy to break apart. But the bottom 1-2 cm of ash were very hard, and had a gray color. A piece of it can be seen on Figure 7-3.

Figure 7-3 - Piece of the bottom ash, brown on top, gray on the bottom

A piece of this were removed for ICP, but the rest of it, was broken apart and mixed in with the rest of the ash. This was done by putting all of the ash through first a 2 mm and then a 1 mm sieve. The soft pieces of the bulk of the ash went through easily, while the hard pieces of the bottom ash almost had to be grated on the sieve. Putting the clumps through the sieve, was done in a way, to try and mix the hard parts into the rest of the ash, as well as possible.

7.2   MIX DESIGN

The purpose of part 1 of the report was to see whether Weber could switch out some of the cement used to produce blocks, with ash. As the block does not have to be very strong, and the strength of them is mainly limited by the strength of the LWA, it was theorized that a rather large fraction of the cement could be replaced. In Table 7-1 an overview of the different castings can be seen.

Table 7-1 - Overview of the different blocks cast

Name	Cement replacement
[-]	[%]
REF	0
WA.15	15
WA.20	20
WA.25	25
WA.30	30
WA.35	35
WA.40	40

There have been made a single block of each of the mixes with a cement replacement, and multiple of the reference block. They are named with a WA for wood ash, to not confuse them with the blocks from the parallel running project, concerned with sewage sludge ash (SSA).

The recipe for the blocks were modelled on the recipe that Weber uses as closely as possible. However, due to differences in equipment and a lack of experience, a lot of testing needed to be

carried out to get the best results. On Figure 7-4 a process diagram, detailing the work with the mix design can be seen.
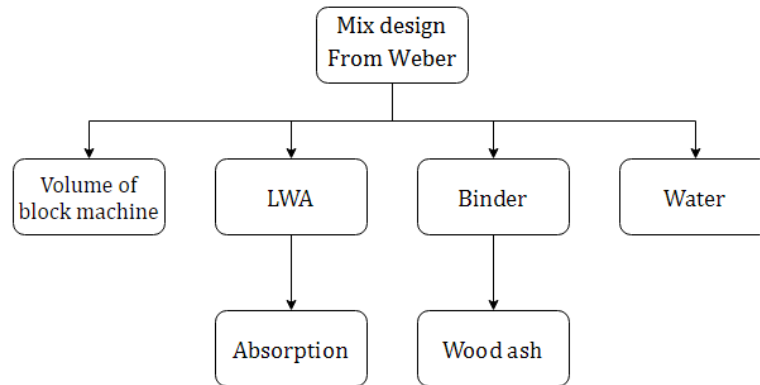
Figure 7-4 - Process diagram for the mix design for the blocks

7.2.1 Mix design from Weber

From a mail from Weber, the mix design seen in Table 7-2 is known. The mail can be seen in appendix P1-MA-01.

Table 7-2 - Webers mix design

For 1 m^3		
LWA 0-2	370	l
LWA 2-4	150	l
LWA 4-10	850	l
Rapid cement	110	kg
Coal Fly ash	45	kg
Water	125	l

The procedure for mixing that was also included in the mail, goes as follows.

1. Add 25 l of water
2. Add all of the LWA (needs time to soak up the water)
3. Add cement + fly ash
4. Add 100 l of water

7.2.2 Volume from previous project

As the recipe is for 1 m^3, it needs to be scaled down. It was known from a previous project, that the blocks produced on the block machine are 25.62 l. Knowing this, it is possible to scale down the recipe to the one seen in Table 7-3.

Table 7-3 - Downscaled recipe for blocks

For 25.62 l		
LWA 0-2	9.48	l
LWA 2-4	3.84	l
LWA 4-10	21.78	l
Rapid cement	2.82	kg
Coal Fly ash	1.15	kg
Water	3.20	l

7.2.3   Lightweight Aggregate

A casting was tried using this recipe, but it was impossible, as the mixture was way to dry. The problem was, that when Weber put in the LWA, it has a water saturation, which is a lot higher than equilibrium state with the atmosphere.

In a mail from Weber, it was described how the LWA was sprayed with about $12 \ l/m^3$ of water, before it is delivered to the block factory. The mail can be seen in appendix P1-MA-02.

Because of this it will not absorb as much water, as it does when dry, and more water will be left for the cement and ash.

As the exact water content in the LWA that Weber use is not known, it was decided that the solution was, to find the absorption rates for each kind of LWA. Knowing these rates, it would be possible to give the LWA a sufficient amount of water so as to not take any of the water designated for the binder. These absorption rates were found using the method from DS/EN 1097-6 [4], annex C.

Here a dry sample is places in a calibrated pycnometer, weighed, and then filled with air-free water, as seen on Figure 7-5.

Figure 7-5 - Pycnometers with LWA 2-4

As the LWA absorb some of the water, they are topped up with water and weighed at regular intervals. After 24 hours, the LWA is taken out, surface dried and weighed. For the LWA 2-4 and 4-10 the surface dried state (SSD) was reached by drying them in a towel, but for the LWA 0-2, this was not possible. Instead, it was dried using a hairdryer, and the correct dryness was confirmed using the cone method described in the standard.

From knowing the different weights, the absorption rates can be found, as described in the theory chapter. Based on the shape of the curve, it was decided to let the LWA soak for 1 hour, in a corresponding amount of water. For the saturation to be right, the LWA needed to be completely dry before it was soaked, so it was all dried for 24 hours at 105° C, prior to mixing.

As the absorption rate were in percent by weight, and the amounts of LWA prescribed by the mixdesign are measured in volume, the densities for the LWA was needed. These were found in the appropriate datasheets, seen in appendix P1-DA-01-03, and they were used to calculate a mass of water.

7.2.4   Binder

The binder of the blocks consist of around 70% cement and around 30% coal fly ash. Assuming that all of the second addition of water is for the binder, the W/C ratio can be found using the formula described in 6.2.1 :

$$\frac{W}{C_{eq}} = \frac{W}{C + 0.5 \cdot FA + 2 \cdot MS} \rightarrow \frac{W}{C_{eq}} = \frac{100 \, kg}{110 \, kg + 0.5 \cdot 45 \, kg} = 0.755$$

As can be seen this yields a W/C of 0.755, which is rather high, but seeing as the binder paste is to coat all the LWA, and that a high strength is not the aim, it seems reasonable. In the work with the ash, this will be the target W/C ratio.

7.2.5   Wood ash

It is the goal, when casting the blocks containing different amounts of wood ash, to hit the same workability as the reference mix. For this, a sort of activity factor for the ash is needed to be known. As opposed to the activity factors for PFA and microsilica, that also contain information about the hardening process, the factor found here will be purely based on consistency. In Figure 7-6, the process tree for finding a suitable method for determining the activity factor is shown.
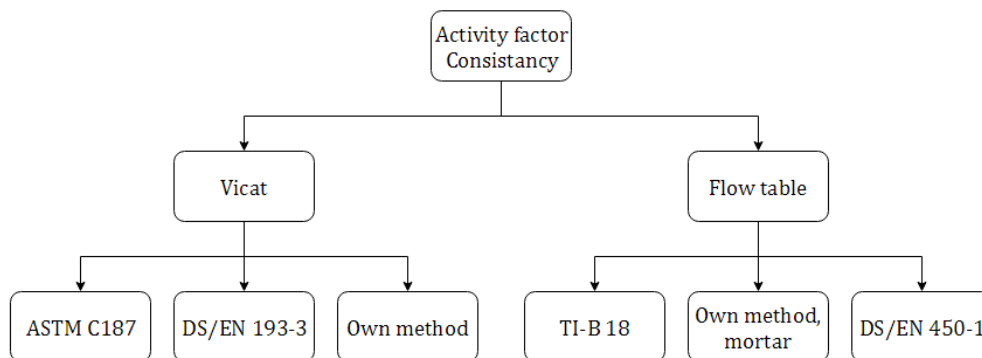
Figure 7-6 - Process tree for finding a activity factor

As there are no predefined way of finding this activity factor for a wood ash, a lot of different methods were tried, to find which one gave the most reliable result, in the easiest way.

As can be seen, the methods are divided in two categories; those done on a vicat apparatus as seen on Figure 7-7, and those done on a flow table as seen on Figure 7-8.

Figure 7-7 - Vicat apparatus used in this project

Figure 7-8 – Flow table used in this project

Below is first a short description of how each mechanism works, and then two tables describing the methods, and comparing them.

7.2.5.1 Vicat

A vicat apparatus is a commonly used tool in describing the consistency of a lot of different materials. It consists on a movable plunger, with a needle attached at the bottom. There is a ruler, so it can be determined how far the arm have traveled, and the weight of it arm is well defined. To use it, a sample is placed underneath the needle, and the arm is released. The needle will go more or less into the sample, depending on the consistency. In Table 7-4 the three different experiments using the vicat apparatus are compared.

Table 7-4 - Descriptions of the different experiments using the vicat apparatus

	ASTM C187 [5]		DS/EN 196-3 [6]		Own method	
Materials	650 g cement Water as needed		500 g cement Water as needed		500 g cement Water as needed	
Mix	(ASTM C305-14)					
	T0	Mix cement and water	T0	Mix cement and water	T0	Mix cement and water
	T30	Start mixer: slow speed	T10	Start mixer: slow speed	T10	Start mixer: low speed
	T60	Stop mixer and scrape down	T100	Stop mixer and scrape down	T100	Stop mixer and scrape down
	T75	Start mixer: high speed	T130	Start the mixer: low speed	T130	Start the mixer: low speed
	T135	Stop mixer and scrape down	T220	Stop the mixer	T220	Stop the mixer
	T225	Start mixer: high speed				
	T285	Stop mixer				
Filling and placing mould	Form ball with hands. Toss back and forth 6 times. Paste is placed in mould with hands and the excess is removed. Place mould into the vicat, and lower the needle to where it just touches the sample. **Allowed filling time = 30 s**		Transfer the paste to the mould and fill to excess. Voids are removed by gently tapping the mould against ball of hand. Remove excess. Place mould into the vicat, and lower the needle to where it just touches the sample. **Allowed filling time = 30 s**		Form ball with hands. Toss back and forth 6 times. Paste is placed in mould with hands and the excess is removed. Place mould into the vicat, and lower the needle to where it just touches the sample. **Allowed filling time = 65 s**	
Releasing the rod	5 min 15 s after zero time		4 min 10 s after zero time Read scale 5 s after penetration has ceased or 30 s after release		4 min 45s after zero time Read scale 5 s after penetration has ceased or 30 s after release	
Result acceptance	-		6±2mm between plunger and base		±2 mm	

For each of the methods a number of tests were made. The results can be seen in one of the following chapters, but common for all of them, were that it was near impossible to make consistent results.

7.2.5.2 Flow table
The other set of experiments were done on a flow table. The surface of the flow table can be raised up and slammed down by means of a hand crank. The way to use it is by adding a sample on top of the table, and then slamming it down a set number of times to see how much the sample spreads out. In Table 7-5, a description and comparison of the different experiments using the flow table can be seen.

Table 7-5 - Descriptions of the different experiments using the flow table

Method	TI-B 18 (86) *[7]*		Own method		DS/EN 450-1 *[8]*	
Materials	Cement paste		LWA 0-2 Mortar		Mortar	
	600 g cement		106.47 g cement		450 g cement	
	Water according to W/P-ratio		43.53 g coal fly ash		1350 g sand	
			357.9 ml LWA 0-2		225 g water	
			Water according to W/P-ratio			
Mix					(196-1:2005)	
	T0	Mix cement and water	T0	Mix LWA and water	T0	Mix water and cement and start mixer: low speed.
	T10	Mix at low speed	T10	Mix at low speed	T30	Add sand during 30 seconds
	T100	Stop mixer and scrape sides	T100	Add binder during 30 seconds	T60	Mix at high speed
	T130	Mix at low speed	T130	Mix at high speed	T90	Stop mixer and scrape sides
	T220	Stop mixer	T190	Stop mixer and scrape sides	T120	Let the mortar rest
			T220	Let the mix rest	T180	Mix at high speed
			T250	Mix at high speed	T240	Stop mixer
			T310	Stop mixer		
Filling cone	Fill cone half way, stamp 20 times		Fill cone half way, stamp 20 times		(DS/EN 1015-3)	
	Fill other half, stamp 20 times more. Cut off top. Remove cone		Fill other half, stamp 20 times more. Cut off top. Remove cone		Fil cone half way, Stamp 10 times Fill other half of cone and stamp 10 times. Cut of the top. Remove cone	
Dropping the table	In the span on 10 seconds, drop the table 10 times. Measure radius on 4 sides, and take an average.		In the span on 10 seconds, drop the table 10 times. Measure radius on 4 sides, and take an average.		In the span of 15 seconds, drop the table 15 times. Measure radius on 4 sides and take an average.	
Result acceptance	Test mortar should be within ± 10 mm of the reference		Test mortar should be within ± 10 mm of the reference		Test mortar should be within ± 10 mm of the reference	

The three methods were tested to see which one would work the best. The results can be found in one of the following sections, and based on these, DS/EN 450 was picked and used moving forward.

From here a number of different mixes were made. In these different amounts of cement were substituted with ash, and the appropriate amount of water for getting the same consistency as the reference were found. Knowing the amount of cement substituted, and the amount of extra water needed, it was possible to calculate an activity factor for the wood ash.

This activity factor can then be put into the formula used earlier, with the W/C set to the same as before, and a new mass of water can be found.

7.2.6   Test cast and extra water

A test cast was made with the calculated amount of water, but it was still found to be too dry. In the same mail that explained the watering of the LWA, the contact at Weber theorized that the mix would have to be wetter, to be cast on the small scale block machine, as it is not able to compresses it as thouroughly as the machine that Weber uses.

The amount of water was increaced from the calculated amount in small incriments, untill the mixture had a consistancy that allows for a "snowball" to be made. This consistancy yeilds the best results on the small scale machine according to the responsible concrete technician.

The amount of water was recorded, and a final mix design was found.

7.3   CASTING OF BLOCKS

In the following a suitable casting method will be found and described in general. A full guide in how to cast blocks on the machine, can be seen in appendix P1-ME-05.

7.3.1   Block machine

The casting of the blocks is done in a block machine, shown in Figure 7-9 that allows compaction and vibration at the same time.

Figure 7-9 - Block machine

The blocks produced by the machine have a special shape, as can be seen on Figure 7-10. This is due to the fact that the machine were made for a project regarding the lightweight part of the superlight concrete structures that the company Abeo are making.

Figure 7-10 - block made in the block machine

The form does not interfere with the present project, as it is the plan to drill cores anyway, so it is not deemed a problem.

To cast a block, the machine is first placed over top a plate. Both the machine and the plate should be level, and there should be a ~5 mm gap between them. Then the arm and pressure plate of the machine are lifted up, and the cavity can be filled with the concrete mix. The arm can then be lowered again, and used to press down on the block, compacting it. The compaction is aided by the build in vibrator.

Once a block is cast, the whole machine is lifted up gently, making sure that it does not damage the fragile block. The block is covered in plastic and left for a day or two, to harden before it is moved, to a climate controlled room.

7.3.2 Process

Casting a block on the block machine involves a number of factors, including time, total compaction, and total pressure on the block, among others. A diagram of the different factors considered, can be seen in Figure 7-11.
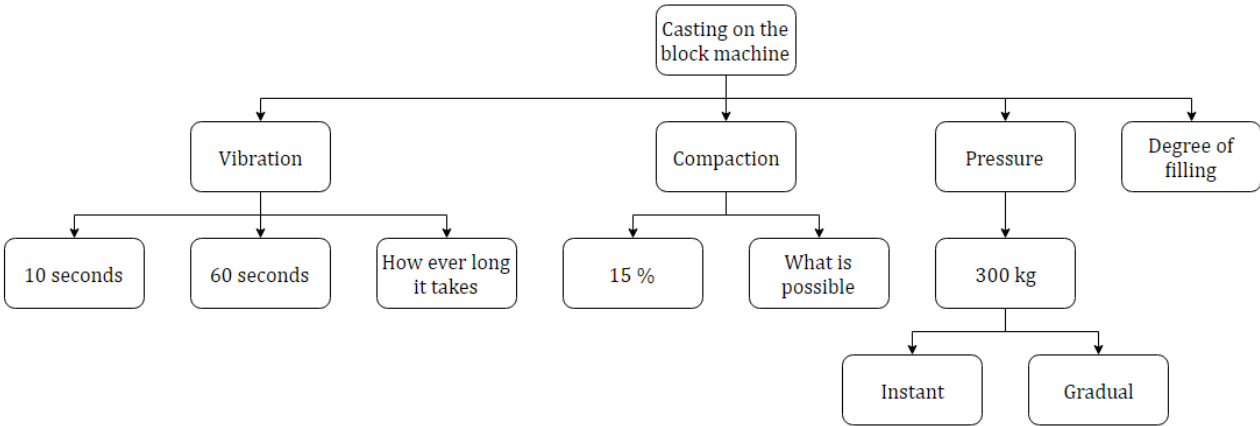

Figure 7-11 - Process of setting parameters for casting blocks

Below, the different parameters are configurations, are described in each their section.

7.3.2.1 Vibration

The machine is equipped with a vibrator that aids the compaction. The vibrator is either on or off, there are no other settings. From talking to the technician that assisted the previous users of the machine, it is known that they sometimes ran the vibrator for 10 seconds and sometimes for 60 seconds. Both of those timeframes were tried, but it was found to be better for the quality of the blocks, to just let the vibrator run for however long it took the get the blocks to the right compaction.

7.3.2.2 Compaction

From correspondence with Weber it is known, that they compact their blocks about 15 % from the initial volume. This can be seen in appendix P1-MA-02

In Figure 7-12 it can be seen what the inside of the block machine looks like.
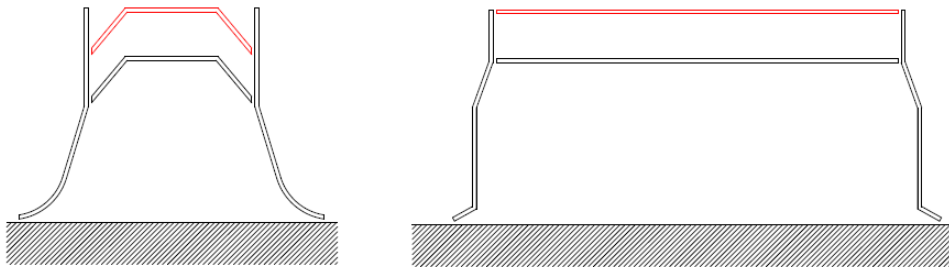

Figure 7-12 - Double view of the inside of the block machine

The drawing is made in AutoCAD based on the drawing seen in appendix P1-DR-01 and measurements taken on the machine. From this the total internal volume of the machine is found to be 33404 cm^3. A compaction of 15 % from this volume, yields a block, with a volume of 28393 cm^3. Had the top been flat, and knowing that the area of the top of the block is around 104 cm^3, this means that the plunger would have to go down 4.8 cm, however, since the plunger is not flat, a compensation is made using the "center of mass" tool in AutoCAD. This adds 3.2 cm to the required depth, and together with the wall thickness of the plunger of 0.9 cm, the total required depth, measured from the top of the plunger, to the edge of the chamber is 8.9 cm.

It was tried to reach this level of compaction, but as it was impossible, this approach was dismissed.

7.3.2.3 Pressure

The block machine is equipped with a scale, which measures how hard the plunger is pressed down. It measures in kg, and goes to 300 kg. By pressing down on the lever, the pressure can be increased, and using the principle of the lever, a pressure of 300 kg can be obtained with relative ease.

It was discussed whether it was best to increase the pressure gradually or to apply it all at once. Applying it gradually is best done by a person, pressing down on the lever, and watching the scale. Applying it all at once could be done by adding a hook on to the lever, and hanging an appropriately large weight from it. Using the measurements of the machine, and the principle of the lever, it was determined how large the weight should be:

$$\frac{l_p}{l_w} = \frac{w_p - w_a}{w_w}$$

where:

l_p Length from pivot to pressure plate

l_w length from pivot to weight

w_p weight applied at pressure plate

w_w weight of weight

w_a original weight of arm

From this, the weight of the weight can be found:

$$\frac{l_p}{l_w} = \frac{w_p - w_a}{w_w} \rightarrow w_w = \frac{(w_p - w_a) \cdot l_w}{l_p} \rightarrow w_w = \frac{(300\ kg - 28.5\ kg) \cdot 240\ cm}{46\ cm} = 52.04\ kg$$

A couple of casting were done using the weight, but it was found that it was next to impossible to make the block even using this method. It was decided that the weight should be applied gradually, and so it was done by hand.

7.3.2.4   Degree of filling

It was discussed how much to fill the machine. It is tempting to fill it as much as possible, as this will yield the largest amount of sample. However, due to the shape of the cavity of the machine, filling it too much can cause a problem. On Figure 7-13 a principle sketch of the cross section can be seen.
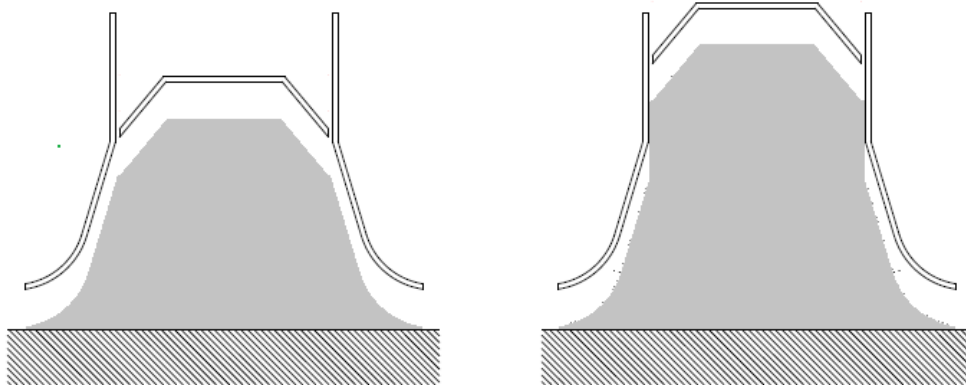
Figure 7-13 - Principle sketch of cross section of casting machine cavity

The sketch illustrates the situation where the block has been cast, and the machine is in the process of being lifted free. On the left is a smaller block, and as can be seen, the machine only have to be lifted a small bit before the block is free. On the right is a larger block that is still in contact with the machine. This means that if a larger block is casted, it is a lot more crucial to lift the machine up perfectly straight, as any movement from side to side, can cause the block to break. It was found that it was near impossible to lift the machine this straight, so it was decided to make smaller blocks.

7.4   HARDENING, DRILLING AND CUTTING CORES FROM BLOCKS

After the blocks have been cast, they have to harden for some time before they can be drilled into smaller samples. This is done in a climate controlled room, where the temperature is kept above 15° C, and the relative humidity is kept below 65 % in compliance with DS/EN 772-1 [9].

As seen earlier, the blocks are an irregular size due to the fact that the block machine is made for Abeo blocks. Both for this reason, and for the sake of getting as much data as possible, each block is drilled into multiple cores. According to the standard DS/EN 1354 [2] any core size above 70 mm is acceptable. A size of 75 mm was picked, as it was the smallest available, that was over 70 mm, and the smaller the core, the more samples.

The drilling was done on a standard concrete drilling drill, and is was found that with a bit of care, 12 cores could be drilled from each block, as can be seen on Figure 7-14.
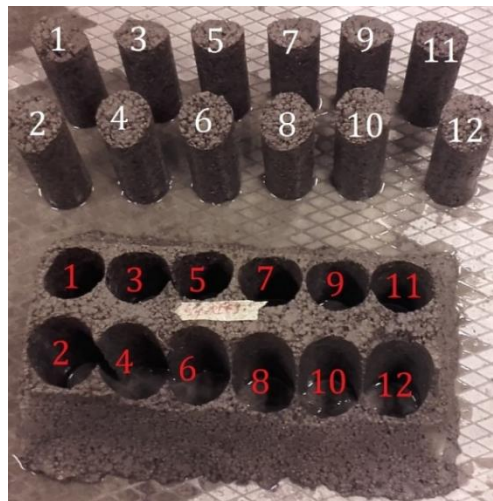
Figure 7-14 - Cores next to the block they came from

On the figure, the naming convention for the cores can also be seen.

After the cores were drilled, they had to be cut to length. As mentioned earlier, when testing LWA concrete, the core should be as high as it is wide. This means that the cores had to be cut to a length of 75 mm. This was done on a diamond saw, where first the top was cut level, and then the sample was cut. Many of the cores drilled were long enough, that they produced two samples, as seen on Figure 7-15. Care was taken however, to make sure that both edges of each sample were cut, to not compromise the data.

Figure 7-15 - Cores cut into multiple samples

In cases where a core yielded two samples, an "a" and "b" was added to the names respectively. As seen on the figure, each sample is wrapped in tape. This is due to the fact that it is impossible to write on the surface of the sample, so the name was written on the tape.

7.5   TESTING CORES

After the cores were matured to the desired age, it was time to test them. Most of the samples were used for testing compressive strength, but some were reserved for measuring the density and porosity and for finding the absorption rates.

7.5.1   Density and porosity

As mentioned above, there are two different ways of measuring the density. All of the samples tested for density and porosity are measured in both different ways. The first way is easy. The height and diameter of the sample is found, and together with the weight of the sample, after it has been dried out, a density can be calculated.

For the other method, the samples also have to be dried out completely. This is done in an oven at 105°C. For normal concrete, the temperature would only be around 50°C, due to the fact that drying it out too fast, can cause damage to the pore structure. This was not thought to be a problem with the LWA concrete, due to the very loose structure, and as drying at 50° C can take up to 3 weeks, it was decided against.

After the samples are dried out, their weight is recorded, and they are placed inside a desiccator. A vacuum pump is hooked up, and run for at least 3 hours. After this, water is let into the desiccator using the vacuum, until the samples are covered by around 3 cm of water. This can be seen on Figure 7-16.

Figure 7-16 - Desiccator full of samples

They are then left, still under a vacuum for 1 hour, before the pressure is equalized with the atmosphere, and then they are left over night. At this point, they are thought to be completely full of water. They are then weighed below and then above water. The method is described in full in appendix P1-ME-06.

It is this above water weight that poses a problem. The method is designed for ordinary concrete or mortar samples that are much tighter in structure. This means, that they hold onto the water that is inside them, and weighing them poses no problem. With LWA concrete however it is another matter. The structure is so loose, that a lot of the internal water runs straight out once they are held above water.

If the pores are not filled with water, when the mass is measured, the measurement will be wrong, and as both density and porosity are dependents, they will be wrong as well. The solution to this problem, was to have a glass jar zeroed out on the scale, that the sample were put into straight after it were taken from the water. This caught all the water, and gave the correct weight. The resulting densities were compared to the ones found using the other method for some of the blocks that had a very regular shape. As the inconsistency between the two measurements was small, the method was deemed usable.

7.5.2   Capillary suction
To see if the capillary suction of the blocks were affected by the addition of ash, two samples from each block with an ash-replacement, four samples from two different reference block, and three samples from a weber block were tested for capillary suction.

The test was done by first drying out the samples in an oven at 105°C, until weight stable. They were then placed in a tray on top of brass rods to ensure flow underneath them, and water was put in till the lower 5 mm of the samples were covered. They were then weighed a number of times, a lot in the start, and then with larger and larger intervals, until the final weighing after 36 hours.

From the weight addition, caused by water being sucked up into the sample, a capillary suction could be found. The full method can be seen in appendix P1-ME-07.

7.5.3   Compressive strength

The testing of compressive strength was done on the machine shown in Figure 7-17.

Figure 7-17 - Machine used to test the compressive strength of the samples

The samples were put in it, with a piece of light density fiberboard on each side, and a ball joint was places on top, to make sure that the load was evenly distributed over the surface, even if the sample was a bit crocked.

DS/EN 1354 calls for a loading of 0.1 ± 0.05 MPa pr. second, when the strength of the sample is unknown, however the technician responsible for the machine advised against this. As the machine works better when the loading is determined by displacement instead of force, it was decided to set it to run at 0.5 mm/min instead.

The load, displacement and time were recorded by a connected computer, every time the load changed 25 N, and also every 50 milliseconds. Once the sample broke, the machine was stopped, and the loading history was saved on the computer. From here the loading history could be plotted, and the maximum force could be found.

Before the samples were loaded into the machine, their height and diameter were measured, and they were weighed. After testing the samples were weighed again, and then placed in an oven, until they were weight stable. From this, a water content and a density could be found. This was done, due to the fact that, if the water content of the sample is under 4 %, it can affect the strength measured.

The load found was in kN, which was then divided over the area of compression, to find the compressive strength in mPa. Here a small compensation in the load was also made, based on the weight of the ball joint used.

7.5.4   Leaching

When working with incorporating ash into a material that is to be used in construction, it is important to be able to confirm that it is not, and will never be, toxic or dangerous to the environment. As some ashes contain a lot of heavy metals it is important to know if these are bound hard enough to the cement paste, to stay there for good.

To test this, one of the already broken samples of LWA concrete were crushed to a fine powder, mixed with water and placed on a shaking table for 24 hours. After this, the sample was filtered, and the water is run on the ICP. From here the concentrations of the different heavy metals that have leached out into the water could be found. The full method can be found in appendix P1-ME-08.

8. RESULTS AND DISCUSSION

8.1 ASH

In the following, the analysis done on the ash will be described.

8.1.1 Picking the ash

The first three analysis were conducted on both ashes, and the results can be seen in Table 8-1.

Table 8-1 - First three results for the ashes

		Køge	Hofor
Water content	[%]	6.49±0.54	0.32±0.02
Water solubility	[%]	24.7	13.11
Loss on ignition	[%]	6.84	2.59

As can be seen, both in solubility and in loss on ignition (LOI), the ash from Køge performs a lot worse than the one from Hofor. Because of this, it is decided to keep working with the Hofor ash. From here all the ash mentioned will be Hofor ash. The data for these three analysis can be seen in appendix P1-DA-04.

8.1.2 Washing the ash

As the solubility of the ash was rather high, the ash was washed as described in chapter 7.1.2.1. It took 8 washes to get the conductivity to level out, as can be seen on Figure 8-1.
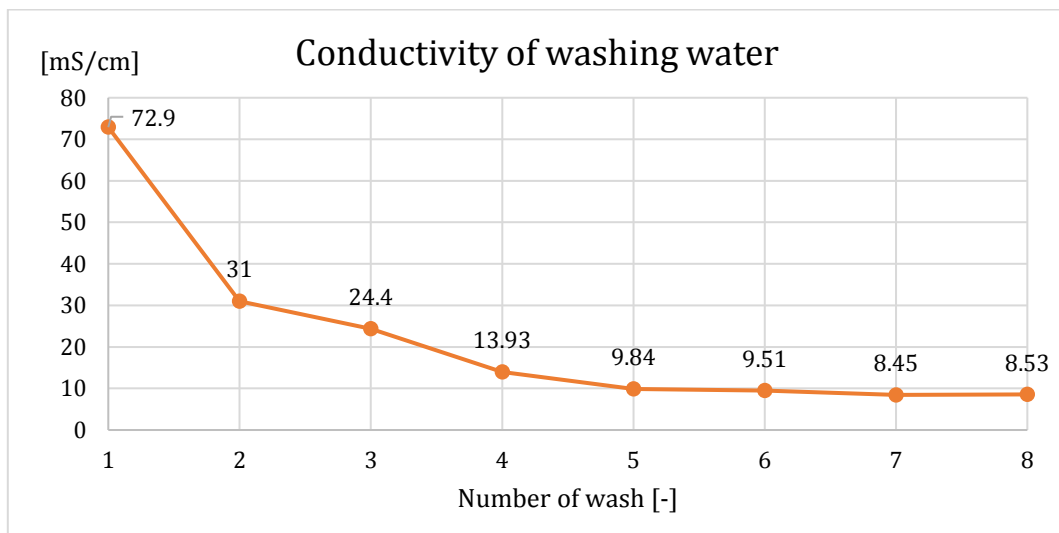

Figure 8-1 - Conductivity of washing water

From the first seven of the washes a sample was taken, and the content of chloride, nitrate and sulfate was analyzed. Results of this can be seen in Figure 8-2.
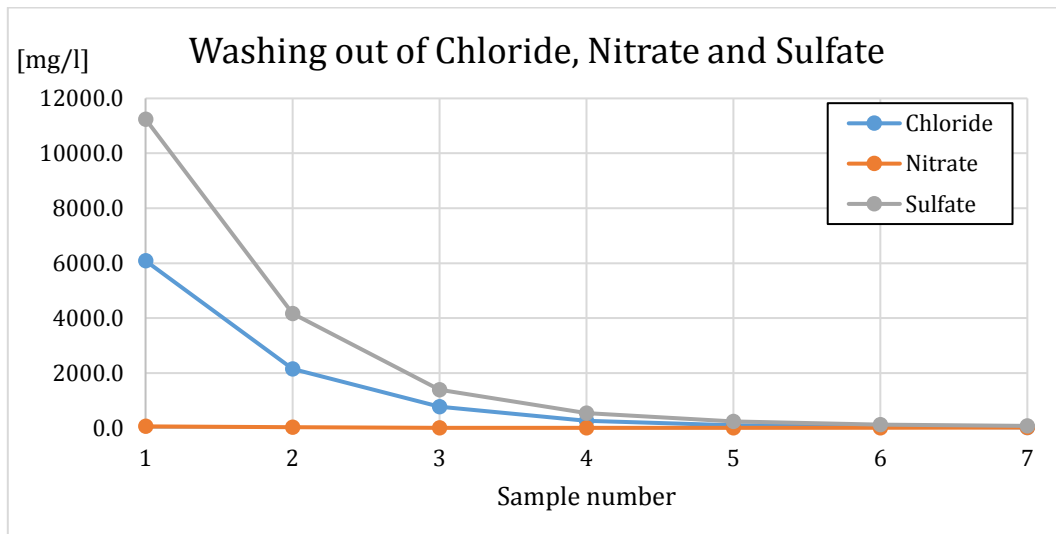
Figure 8-2 - Concentration of Chloride, Nitrate and Sulfate

From both plots it seems clear that the ash has been sufficiently washed. The concentrations that this plot is made from can be seen in appendix P1-DA-05

8.1.3   Loss on ignition of the washed ash

The analysis done on the ash before it was washed should be repeated after it has been washed. However the solubility is thought to be 0 %, as all of the water soluble material have already been washed out, and the water content is 0 %, as the ash was dried thoroughly after it was washed. That leaves the loss on ignition.

A theoretical maximum for this is calculated, using the LOI from before and the solubility:

$$LOI_{theoretical\ maximum} = \frac{LOI_{before}}{100\% - solubility} \rightarrow \frac{2.59}{100\% - 13.11\%} = 2.98\ \%$$

However, when the test was actually made, it turned out that the LOI for the washed ash was actually 12.07%.

This is way higher than expected, and also higher than the standard allows. According to DS/EN 450-1 [8], there are three categories for ash, called A, B and C, where it is defined that the LOI can be no higher than 5 %, 7 % and 9 % respectively.

As mentioned earlier, the ash had somewhat separated during washing, where some of the heavy particles had collected to the bottom. It therefore seemed safe to assume, that the lightest particles would have been on the top. As the ash sample, used for the LOI was taken from the top, before the ash was mixed this might have something to do with the high LOI. It is theorized that there is a correlation between the density of the particles and their LOI, as the lightest particles will be them, most resembling charcoal, and they will have a very high LOI.

Because of this the LOI test was redone with the mixed ash, and it came back as 11.05 %. It is a bit lower than that of the first test, but still way higher than the theoretical max, and also still higher than the standard allows for. The data for the LOI can be seen in appendix P1-DA-06.

An article written by Mingyan Zhao et al. [10] was found on what parts of a bio ash that decompose at what temperature. In it, they found that the LOI will mainly happen between the temperatures 350-530 °C, and that degradation of carbonate can start below 550°C. This means that some of the measured weight loss might be contributed to the decomposition of carbonate rather than to LOI.

As this was discovered rather late in the project period, it has not been investigated further. It was also too late to find another ash to use for casting.

8.1.4 Particle size distribution
The particle size distribution can be seen on Figure 8-3. It can be seen that the raw ash is more evenly distributed between the different sizes, whereas the washed ash has fewer very small and very large particles, and more in the middle.
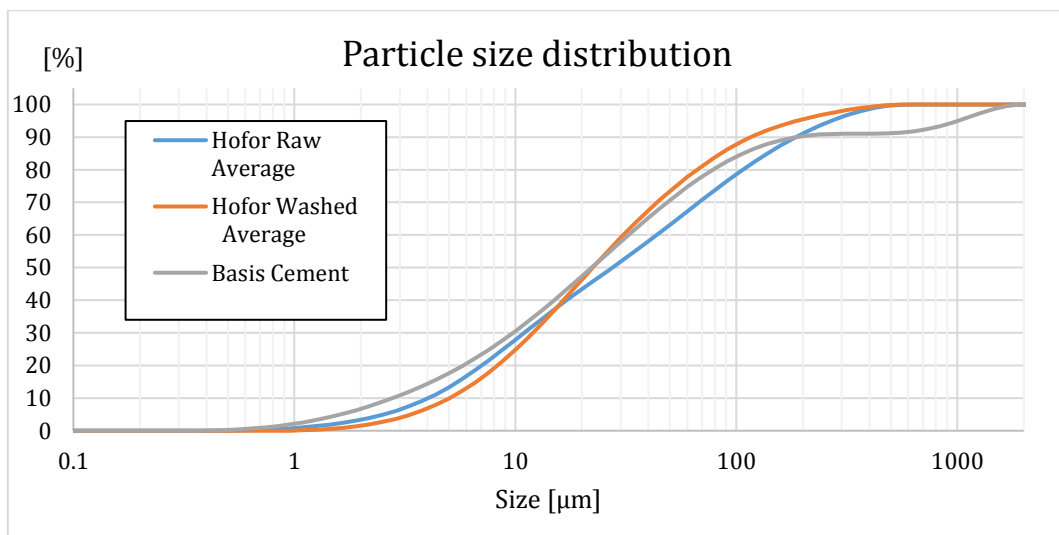
Figure 8-3 - Particle size distribution for both raw and washed HOFOR ash, compared with a basis cement

On the figure, the size distribution for a basis cement can also be seen, and it seems that the ash is very similar to the cement in size, apart for the fact that the cement have a larger quantity of large particles.

In this regard at least, the ash seems very suitable as a cement replacement. The raw data can be seen in appendix P1-DA-07-09.

8.1.5 Micro and macro elements – ICP
The concentrations of a lot of different acid-soluble elements have been measured, both for the untreated ash, the washed ash, and the gray fraction of ash that collected in the bottom of the washing pot. All of the concentrations can be seen on Figure 8-4.
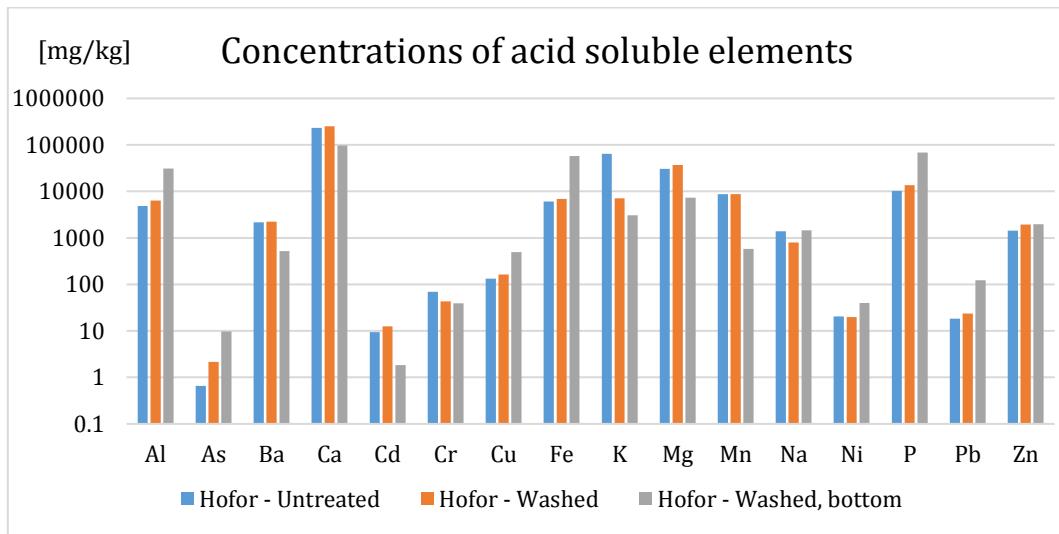
Figure 8-4 - Concentration of acid soluble elements

As not all of these are deemed harmful, in Figure 8-5 the ones for which there is an upper limit, are plotted next to these limits.
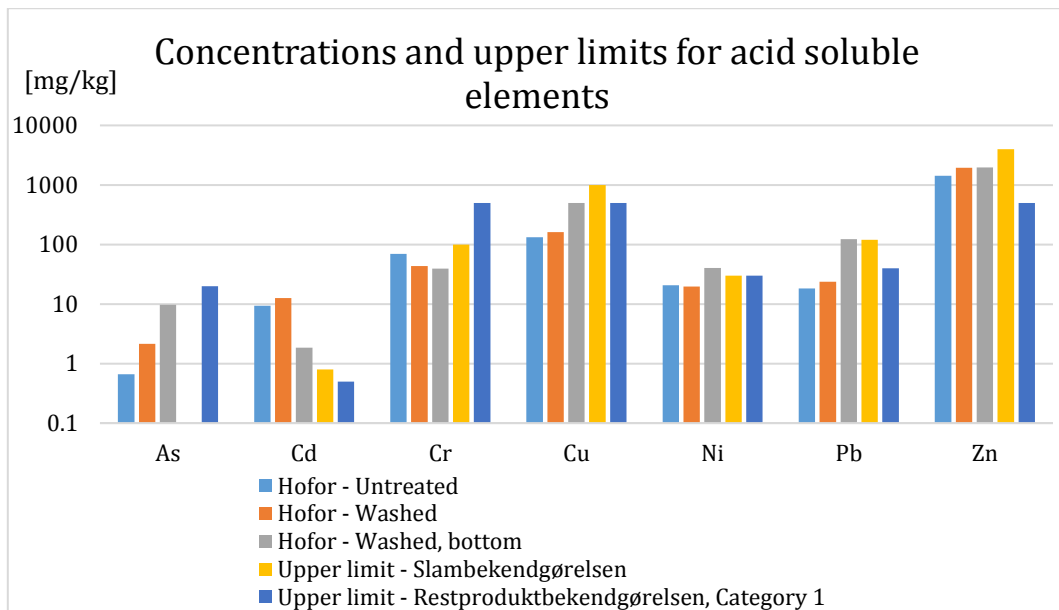
Figure 8-5 - Concentrations and upper limits for acid soluble elements

The limits come from two different decrees from the Danish government. The only breach on Slambekendgørelsen [11] are Cd and Ni in the bottom ash, while Restproduktbekendgørelsen [12] is breached multiple times.

It can be seen, that the concentrations of Al, As, Cu, Fe, Na, Ni, P and Pb are higher in the bottom ash, meaning that the form they are in, is denser than the average ash.

Given these high concentrations, it is hoped, that the LWA blocks will bind the harmful elements hard enough, so as to make sure that they stay bound. The raw data can be seen in appendix P1-DA-10.

8.2 MIX DESIGN

As described previously, a preliminary mix design was known based on Webers mix design, and a volume of the machine. Additional water was to be added to the mix, to soak the LWA. In the following this amount is found, and the rest of the process to finding a final mix design is described.

8.2.1 Absorption rates

From the tests done on the pycnometers, the absorption rates were found. These can be seen on Figure 8-6.
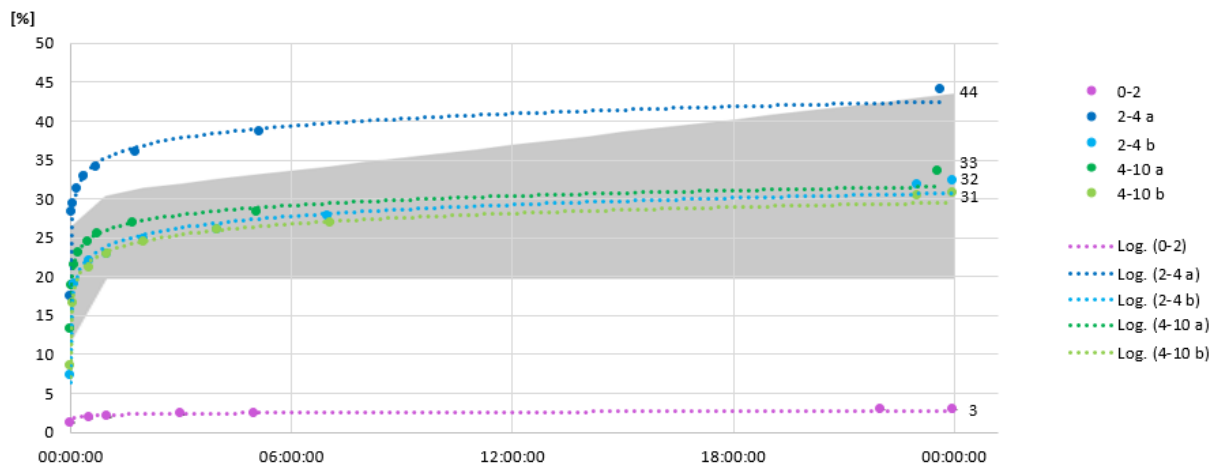
Figure 8-6 - Absorption rates compared to that of LWA 10-20

As can be seen, the LWA 0-2 has a really low absorption rate, compared to the others, which is to be expected, as it is mostly made of small crushed pieces of LWA. The other two sizes of LWA are places relatively close, apart from one of the measurements of the 2-4.

The gray area on the plot, is the range of the absorption rate given for LWA 10-20, as this was the only absorption rate given by the manufacturer. The data sheet for LWA 10-20 can be found in P1-DA-11, Based on this, the difference between the two measurements of LWA 2-4, seem more acceptable. The raw data for all of the curves can be seen in appendix P1-DA-12-14.

As can also be seen for all of them, the absorption is by far most active in the first hour, after which it starts levelling out. Based on this, it seems fair to base the water consumption of the LWA on the absorption for the first hour. The absorption rates for each size of LWA was found using curve fitting, and from this, the extra water that needed to be added were found. It can all be seen in Table 8-2.

Table 8-2 - Water added due to absorption from the LWA

Sample	Formula, found with curve fitting, where x is the time in days	R^2	Absorption after 1 hour	Average absorption after 1 hour	Amount of LWA in mix based on table densities	Water added to the mix
		[-]	[%]	[%]	[kg]	[kg]
0-2	$y = 0.153 \ln(x) + 2.7214$	0.8492	2.35	2.35	6.40	0.143
2-4	$y = 2.2541 \ln(x) + 42.523$	0.9923	35.36	29.65	0.96	0.285
	$y = 2.1403 \ln(x) + 30.739$	0.9843	23.94			
4-10	$y = 1.7467 \ln(x) + 31.591$	0.9666	26.04	24.64	7.19	1.770
	$y = 1.9724 \ln(x) + 29.514$	0.9791	23.25			
					Total	*2.198 kg*

8.2.2 Test cast and new mix design

A new mix design can now be found and tested. The original mix design can be seen in Table 8-3.

Table 8-3 - Original mix design

For 25.62 l		
LWA 0-2	9.48	l
LWA 2-4	3.84	l
LWA 4-10	21.78	l
Rapid Cement	2.82	kg
Coal Fly ash	1.15	kg
Water	3.20	l

Based on this, and the absorption rates found previously, a new mix design can be made. Take note that the amount of water in this design includes 0.64 l for soaking the LWA, and so this has to be taken out, before the newly found soaking water is added. The new mix design can be seen in Table 8-4.

Table 8-4 - New mix design based on absorption rates

For 25.62 l		
LWA 0-2	9.48	l
LWA 2-4	3.84	l
LWA 4-10	21.78	l
Rapid cement	2.82	kg
Coal Fly ash	1.15	kg
Water	4.76	l

A test cast was made based on this design, and it was better than the first test cast, but still came out too dry. As mentioned, this was to be expected, as the available machine could not press as hard, as Webers can, and so the mixture had to be wetter. Water was added, until the mix had the desired consistency, and from this water addition the new mix design was found. This can be seen in Table 8-5.

Table 8-5 - Final mix design

For 25.62 l		
LWA 0-2	9.48	l
LWA 2-4	3.84	l
LWA 4-10	21.78	l
Rapid cement	2.82	kg
Coal Fly ash	1.15	kg
Water	6.40	l

The cement used in the final mix, is a Rapid Cement from Aalborg Portland, CEM I 52.5 N (LA), and the PFA is from Nordjyllandsværket.

As can be seen around 1.6 l of water needed to be added, to get the right texture. A number of test blocks were cast with this amount of water, and as the mix came out consistently good, it was kept.

In ordinary concrete, it would be unthinkable to add this amount of water, as it would have a huge impact on the strength, but seeing as it is the LWA that is the strength determining factor in the LWA blocks, this is not thought to be a problem.

8.2.3   Activity factor

Once the final mix design was found, all that was left, was to find the activity factor for the ash, and casting could begin.

As stated previously, a number of different methods was tried to find the water need for the ash.

The methods involving a vicat apparatus, are designed for cement, and so reference testing was done on cement. From Aalborg Portland [13] it is known, that for their basis cement the water need is 0.28 and for rapid cement it is 0.29.

The first method tried was the one described in ASTM c187. The tests were done on basis cement, and the results can be seen in Table 8-6.

Table 8-6 - Experiments done according to ASTM c187

W/C aim	Cement	Water	W/C real	Distance from bottom	Comment
-	[g]	[g]	-	[mm]	
0.5	650.80	325.01	0.499	-	Way too liquid
0.29	650.00	188.50	0.290		Needle went straight through
0.25	650.03	162.48	0.250		Needle went straight through

As can be seen the needle of the vicat went straight through the mix, even when it was notably dryer than it should. It was also a problem with the method that it included very little time from when the mixing stopped, to when the arm was to be released. As it was not possible to stay within the timeframe, and the results varied too much form the expectation, this method was discarded.

The next method was the one described in DS/EN 193-3, and it was done on basis cement as well. As can be seen from Table 8-7, the method yields wildly different results for the same mixes.

Table 8-7 - Experiments done according to DS/EN 193-3

W/C aim	Cement	Water	W/C real	Distance from bottom	Comment
-	[g]	[g]	-	[mm]	
0.29	500.00	145.03	0.290	0	Needle went straight through, too slow
0.25	500.02	125.04	0.250	17.5	10 seconds too slow
0.27	500.02	135.01	0.270	0	Needle went straight through
0.26	500.03	130.05	0.260	3.5	Too slow
0.257	500.00	128.52	0.257	4	Too slow
0.253	500.02	126.52	0.253	19	8 seconds too slow
0.26	500.00	130.00	0.260	21	7 seconds too slow
0.27	500.00	135.01	0.270	0	Needle went straight through

In Figure 8-7 the data is plotted. It was expected to show some kind of correlation between the W/C and the penetration depth, but as can be seen it does not.
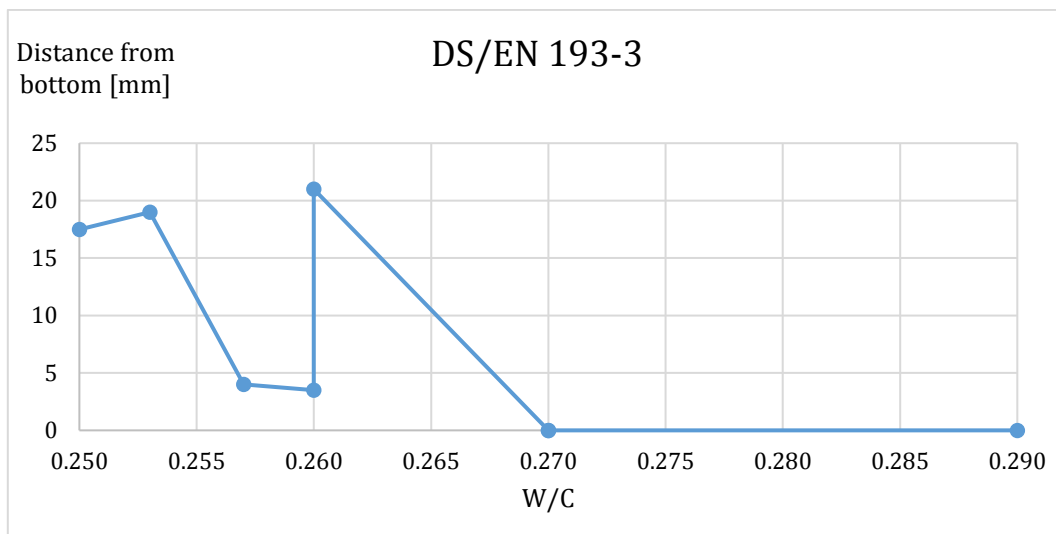

Figure 8-7 - Plot of data from DS/EN 193-3

Based on this, the method was discarded.

The next method tried, was the self-developed one. It was a mix of the previous two, taking the best from each. The data can be seen in Table 8-8.

Table 8-8 - Own method

W/C aim	Cement	Water	W/C real	Distance from bottom
-	[g]	[g]	-	[mm]
0.28	500.03	140.04	0.280	11
0.28	500.01	140.01	0.280	0
0.28	500.00	140.00	0.280	12
0.28	500.15	140.04	0.280	0

As can be seen, four identical mixes, yielded wildly different results. Based on this, this method was discarded as well.

The conclusion on these three experiments is, that producing reliable data on a vicat apparatus, is exceedingly hard. First off, it all has to be done in a very short time, and secondly the lowering of the needle, to where it just touches the sample, is next to impossible. A fraction of a millimeter too high or too low, affects the results a lot. Because of this, testing with the vicat was discontinued.

The next set of tests were done on the flow table. First up was the TI-B 18, and the results can be seen in Table 8-9.

Table 8-9 - Results from tests done according to TI-B 18

	W/C	Cement	Water	Average radius	Deviation
		[g]	[g]	[mm]	[mm]
ref1	0.28	600.02	168.02	69.1	2.0
ref2	0.33	600.01	198.00	80.5	2.1
ref3	0.38	600.03	228.02	90.6	2.9

As can be seen, the results are very consistent, and there is a low deviation, for all three different mixes. This voted well for this method.

Next up is the self-developed method, in which the mix is designed, to be identical to that used for the blocks, the two larger sizes of LWA not included. The results can be seen in Table 8-10.

Table 8-10 - Results from tests done according to Own method

W/C_{eq}	Cement	Coal fly ash	LWA 0-2	Water	Average diameter	Deviation	Deviation between averages
	[g]	[g]	[ml]	[g]	[mm]	[mm]	[mm]
					88.8	7.8	
1.50	106.47	43.54	358	192.36	93.8	6.6	5.8
					100.4	2.1	

Here, a large mix is made, and then divided into three and tested. It can be seen, that the deviation is high, both in the separate tests, and between the tests, and for this the method was discarded.

The last method is the one described in DS/EN 450-1. Again a large batch is made, divided into three and tested. The results can be seen in Table 8-11.

Table 8-11 - Results from tests done according to DS/EN 450-1

	V/C	Cement	Sand	Water	Average radius	Average between samples	Deviation Of all measured radiuses
	-	g	g	g	mm	mm	mm
ref1	0.5	450.03	1350.04	224.99	74.5	74.54	2.43
ref2	0.5	450	1350.02	225.03	74.6		

Here it can be seen, that the deviation between two separate tests is really small, especially taking into account that it is a deviation of 24 separate measurements.

Based on this, and on the fact that this method uses less cement than TI-B 18, this one was picked.

Testing was then done, with three different amounts of cement replaced with ash, and the results can be seen in Table 8-12.

Table 8-12 - Activity factor found using DS/EN 450-1

W/C$_{eq}$	Cement replaced	Factor	Cement	Sand	Ash	Water	Average radius	Average of averages	Difference from reference results		Comment
-	[%]	[-]	[g]	[g]	[g]	[g]	[mm]	[mm]	[mm]	[%]	
0.50	10	2	405.03	1350.05	45	247.46	78.0	76.0	1.5	2.0	Accepted
							74.3				
							75.8				
0.50	25	3	337.52	1350.07	112.48	337.5	105.3	105.3	30.7	41.2	
							N/A				
							N/A				
0.50	25	2	337.53	1350.05	112.53	281.38	77.0	75.5	1.0	1.3	Accepted
							75.8				
							73.8				
0.50	40	2	270.06	1350.01	180.03	315.03	83.5	82.3	7.8	10.5	
							83.0				
							80.5				
0.50	40	1.8	270.08	1350.04	180.02	297.1	72.8	72.2	-2.4	-3.2	Accepted
							71.5				
							72.3				

Here it can be seen, that for 10 % and 25 % cement replacement with WA an activity factor of 2 yields acceptable results, while for a 40 % replacement, an activity factor of 1.8 is more suited. It is theorized that a factor of 1.9 would have made acceptable results for all three replacements, and so a factor of 1.9 was used. The full set of data used to come to this conclusion can be seen in appendix P1-DA-15.

The casting is done in such a way, that the water added, due to the activity factor of the ash, is put in last, and not without judging the consistency first. This is due to the fact that the LWA concrete is somewhat unreliable, and a good consistency was preferred over using the calculated amount of water.

8.3 CASTING, STORING AND CUTTING BLOCKS

As the mix design was done, blocks could be cast. For the most part, it went according to plan. However one of the mixes came out really dry and water had to be added. All of the mixes, and what they consist of, can be seen in Table 8-13.

Table 8-13 - Castings

Name	Cement replacement	Activity factor	Water total	Cement	Fly ash	Bio Ash	LWA 0-2	Actual LWA 0-2	LWA 2-4	Actual LWA 2-4	LWA 4-10	Actual LWA 4-10
	[%]	[-]	[kg]	[kg]	[kg]	[kg]	[l]	[kg]	[l]	[kg]	[l]	[kg]
Ref.01	0	1	7.17	2.82	1.15	0	9.48	-	3.84	-	21.78	-
Ref.02	0	1	6.40	2.82	1.15	0	9.48	7.32	3.84	1.23	21.78	6.26
Ref.03	0	1	6.40	2.82	1.15	0	9.48	7.27	3.84	1.22	21.78	6.33
Ref.04	0	1	6.40	2.82	1.15	0	9.48	7.73	3.84	1.40	21.78	6.90
Ref.05	0	1	6.40	2.82	1.15	0	9.48	7.15	3.84	1.41	21.78	6.67
Ref.06	0	1	6.40	2.82	1.15	0	9.48	7.15	3.84	1.52	21.78	6.28
Ref.07	0	1	6.40	2.82	1.15	0	9.48	7.15	3.84	1.35	21.78	6.30
WA.15	15	1.9	6.68	2.40	1.15	0.42	9.48	7.15	3.84	1.37	21.78	6.44
WA.20	20	1.9	6.78	2.25	1.15	0.56	9.48	7.15	3.84	1.34	21.78	6.45
WA.25	25	1.9	6.87	2.11	1.15	0.70	9.48	7.15	3.84	1.37	21.78	7.02
WA.30	30	2.38	7.27	1.97	1.15	0.85	9.48	7.15	3.84	1.28	21.78	6.54
WA.35	35	1.9	7.06	1.83	1.15	0.99	9.48	7.15	3.84	1.29	21.78	7.04
WA.40	40	1.9	7.16	1.69	1.15	1.13	9.48	7.15	3.84	1.38	21.78	6.69

As described earlier, the casting method went through some iterations, before it was settled. This is the reason why there are so many reference blocks, and why Ref01 has a higher water content.

With the LWA 2-4 and 4-10, measuring out a volume and getting a relatively consistent result was easy enough, but with the LWA 0-2, it proved harder. Due to this it was at a point decided to add in a fixed weight instead of a volume.

As can be seen WA30 needed a lot more water to reach the right consistency than expected. The reason for this is unknown.

After the blocks were cast, they were set to harden in a climate controlled room. As can be seen on Figure 8-8 and Figure 8-9, the conditions were within the acceptable limit for most of the project period.
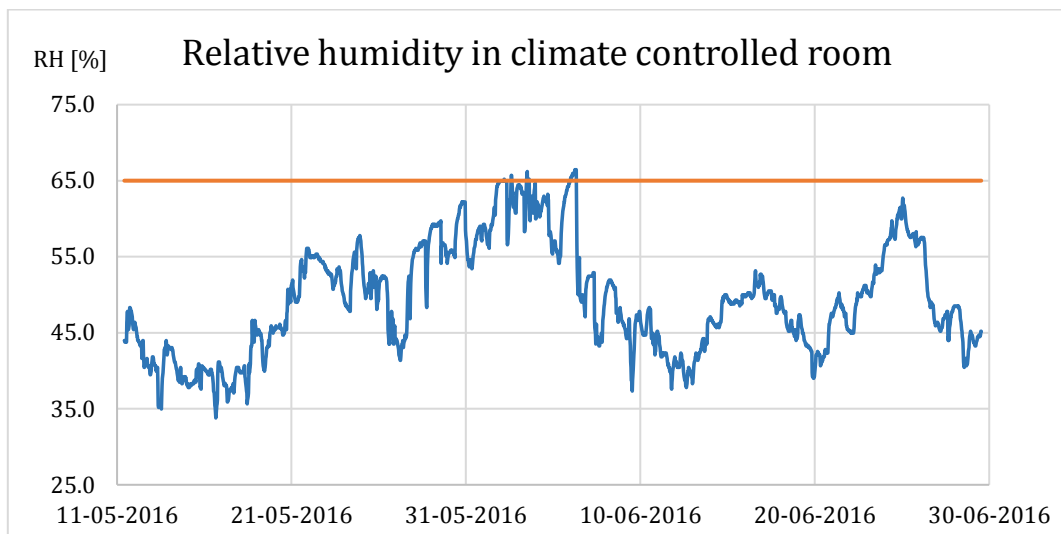
Figure 8-8 - Relative humidity in the climate controlled room during storing of blocks and samples
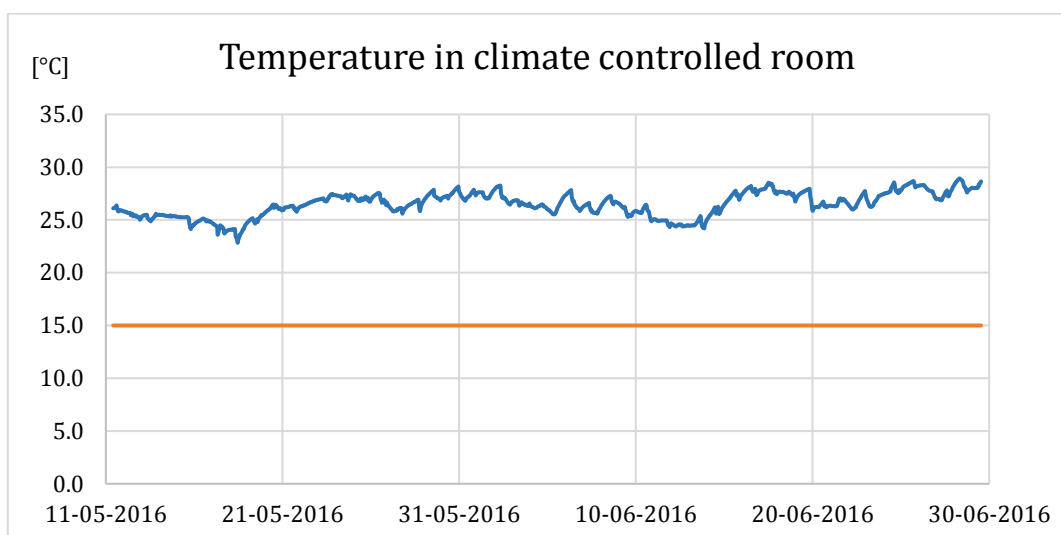

Figure 8-9 - Temperature in the climate controlled room during storing of blocks and samples

The horizontal lines mark the limit, and as can be seen the humidity was very briefly a bit too high, due to the fact that the room were used for other experiments, but this is not thought to be a problem.

After the blocks had been stored a suitable amount of time, they were cut. In Table 8-14 is a list of all of the blocks and the samples cut from each block, together with a description of what they were used for.

Table 8-14 - List of blocks cast, and how their cores were used.

	Cast	Cut	Number of cores	Size in mm	Cores for 14 days compressive strength	Cores for 28 days compressive strength	Cores for compressive strength at other maturities	Cores for other uses
Ref.1	05/04	27/04	3	100	-	-	3 (64)	-
Ref.2	06/04	28/04	2	100	-	-	3 (63)	-
Ref.3	06/04	20/05	12	75	-	-	10 (49)	2
Ref.4	25/04	20/05	12	75	-	-	10 (30)	2
Ref.5	09/05	20/05	11	75	4 (16)	5	-	2
Ref.6	20/05	27/05	23	75	10	9	-	4
Ref.7	30/05	09/06	24	75	10	11	-	3
WA.15	26/05	03/06	21	75	9	9	-	3
WA.20	26/05	03/06	21	75	9	9	-	3
WA.25	25/05	03/06	18	75	8	7	-	3
WA.30	25/05	03/06	19	75	8	8	-	3
WA.35	31/05	10/06	24	75	11	10	-	3
WA.40	01/06	10/06	13	75	5	5	-	3

The numbers in parenthesis are the maturities at which the samples was tested, if this was not done on the planned day. As can be seen the first 4 reference blocks were not tested at the 28 day maturity, as the test method was not yet determined at that point. Also those 4 were used to test different methods for casting, so it is not sure that they are comparable to the rest of the samples.

To have something to compare the results with, samples were also cut and tested from some of Webers own blocks. These can be seen in Table 8-15.

Table 8-15 - Cores cut from weber blocks

	Number of cores	Size in mm	Cores for compressive strength	Cores for other uses
W.1	3	100	3	
W.2	5	75 & 100	2	3
W.3	5	75	5	
W.4	5	75	5	
W.5	5	75	5	
W.6	5	75	5	
W.7	5	75	5	

In addition to these, a sample was taken from W.8, to be used in a leaching test.

8.4 TESTING CORES

8.4.1 Density and Porosity

The samples that were not tested for compressive strength, were among other things, tested for their density and porosity. The density, found using the water method, were held against the density found by measuring the samples and weighing them. Out of 33 samples only four had a difference between the two densities larger than 5 %. Two of these four were from the WA.40, and as can be seen on Figure 8-10, it was most likely the geometrical method that was off, as it does not account for the uneven surface, and so the volume is overestimated.

Figure 8-10 - One of the samples where the two densities were more than 5 % apart

Because of this, the densities found with the water method was picked to be presented, and they can be seen in Figure 8-11.
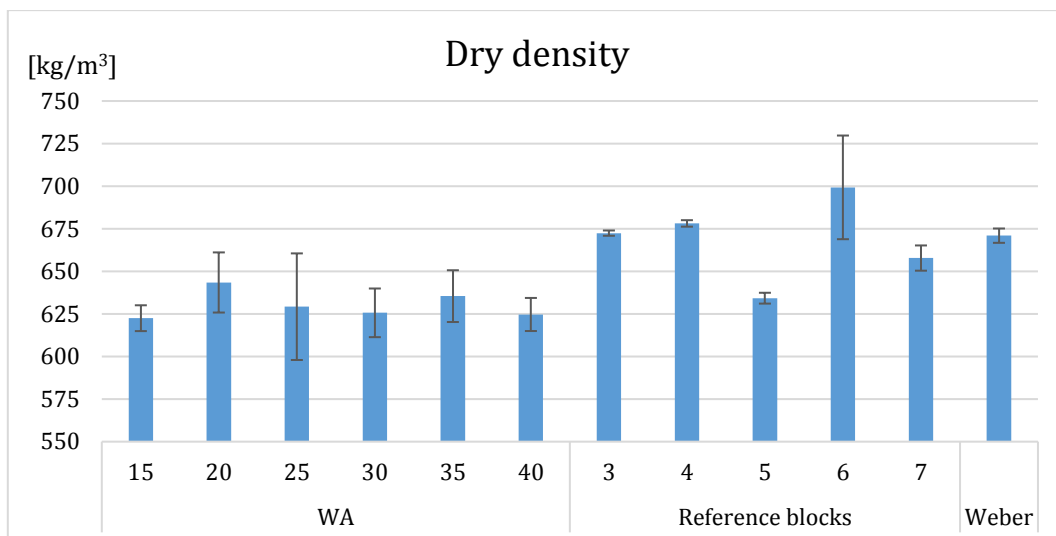

Figure 8-11 – Densities of samples measured two different ways

As can be seen the densities for the blocks containing WA are very similar, all within around 20 kg/m^3 of each other. Generally they are lower than that of the Weber block, which is good, as it means that the block will be lighter, and therefore cheaper to transport. It can also be seen, that the density of Ref.6 is the highest measured. Why it is so different from the other references, is not known, but maybe is has been compacted better. The data that this figure is made from can be seen in appendix P1-DA-16.

From the same measurements that gives the densities, the porosities of the blocks can also be found. These are presented in Figure 8-12.
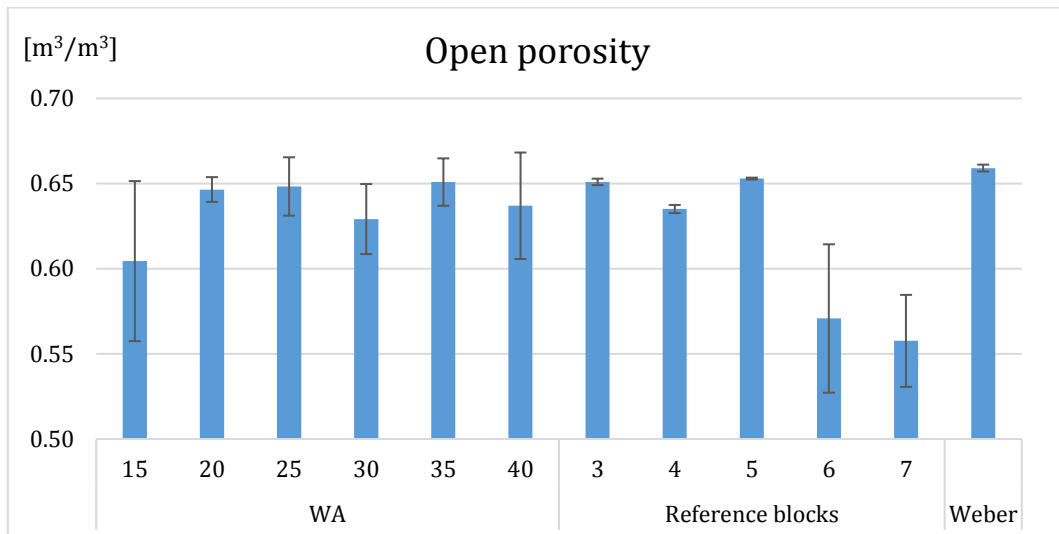
Figure 8-12 - Open porosity of blocks

Here it is seen, that all of the blocks have a porosity higher than 0.5, meaning that they are actually more air than anything else. It is also seen, that most of the blocks containing WA are very similar to the Weber block, when it comes to the open porosity. This is deemed a good result.

8.4.2   Capillary suction

The capillary suction of all of the different blocks were also tested, and the results can be seen in Figure 8-13.
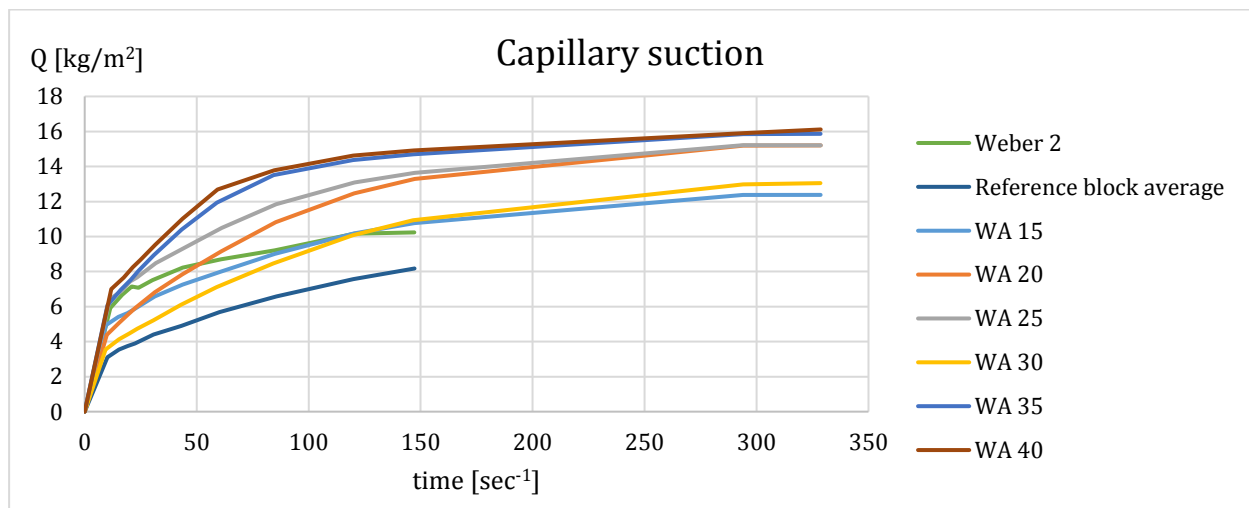

Figure 8-13 - Capillary suction

The lowest values are the ones for the reference blocks, and while WA15 and WA30 behave similarly to the Weber blocks, the rest of the blocks containing ash, lies higher. Apart from WA30, there is a clear tendency that the more ash is added to the block, the faster is sucks up water. This could be seen as a bad result, but as Weber does not try to stay within any limits

when it comes to capillary suction, this does not affect the usability of the blocks. The data that the figure is based on can be seen in appendix P1-DA-17.

8.4.3   Compressive strength

In the following the compressive strength for all of the different samples will be presented and discussed.

8.4.3.1   Weber samples

The Weber samples were tested first, both to figure out the correct testing procedure and to see if they were as strong as they claimed to be. The results can be seen in Figure 8-14. Inside the bar is first the water content in % at testing, and then the testing conditions. Soft and hard refers to the type of plate used to distribute the force. Soft is a 12 mm light density fiberboard, and hard is a 3 mm masonite plate.
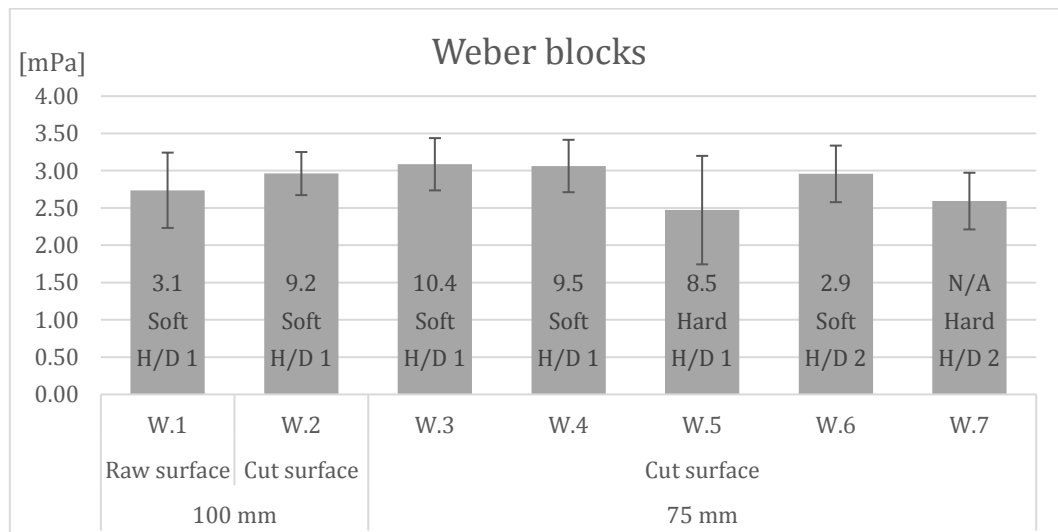

Figure 8-14 - Compressive strength of the Weber samples.

As can be seen, a lot of different things were tested on the Weber samples.

- Firstly is was desired to know, if it affected the strength whether the sample had cut or raw surfaces. As can be seen by comparing W.1 and W.2 that the cut surfaces yields a bit higher strength, albeit, not that much.
- Comparing W.2 with W.3 and W.4 it can be seen, that there is not much difference between testing on samples of 100 mm and samples of 75 mm.
- Comparing W.3 and W.4 with W.5, it can be seen that using a light density fiberboard yields a higher strength and a lower deviation. The same can be seen, when comparing W.6 and W.7 although it is not as pronounced.
- Lastly comparing W.3 and W.4 with W.6 it can be seen, that the H/D does not play a huge role in the measured strength.

Based on this the samples were cut 75 mm in diameter, with a H/D of 1, as this gave the largest amount of samples, and they were pressed between light density fiberboard plates.

It should be mentioned that according to DS/EN 1354, W.2 is not a valid sample, as it only consists of data from two samples, and there needs to be at least three. All of the data, from all of the Weber samples can be seen in appendix P1-DA-18.

8.4.3.2 Reference samples

On Figure 8-15 the compressive strengths all of the reference samples can be seen.
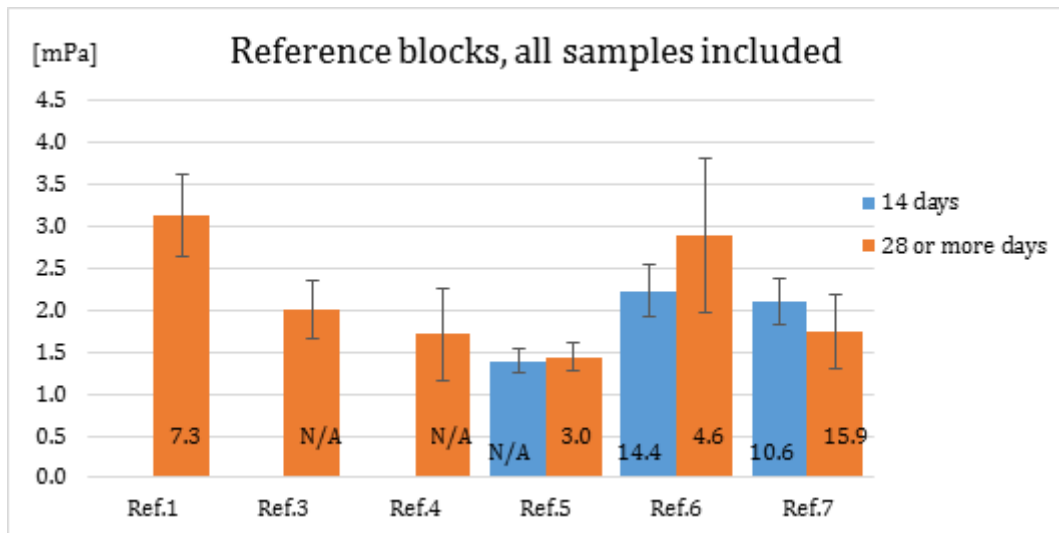

Figure 8-15 - Compressive strength of reference blocks, all samples included

As ca be seen for some of them, the deviation is rather high. This is the case with all the cast samples, as there are apparently large variations in the blocks. All of the raw data can be seen in appendix P1-DA-19-21. It was therefore decided to remove up to 1/3 of the samples, if their strength were more than 25 % different from the average. This decision was based on DS/EN 196-1 [14] in which 1/3 of the samples can be removed, given that their strength is more than 10 % from the mean. From looking at the data, it was clear that if a limit of 10 % were picked, not many data points would remain, and so it was set to 25 % instead.

On Figure 8-16 the compressive strength can be seen again, this time without the outlying data included. The only real change, apart from the smaller deviations, is that Ref.4 is considerably lower.
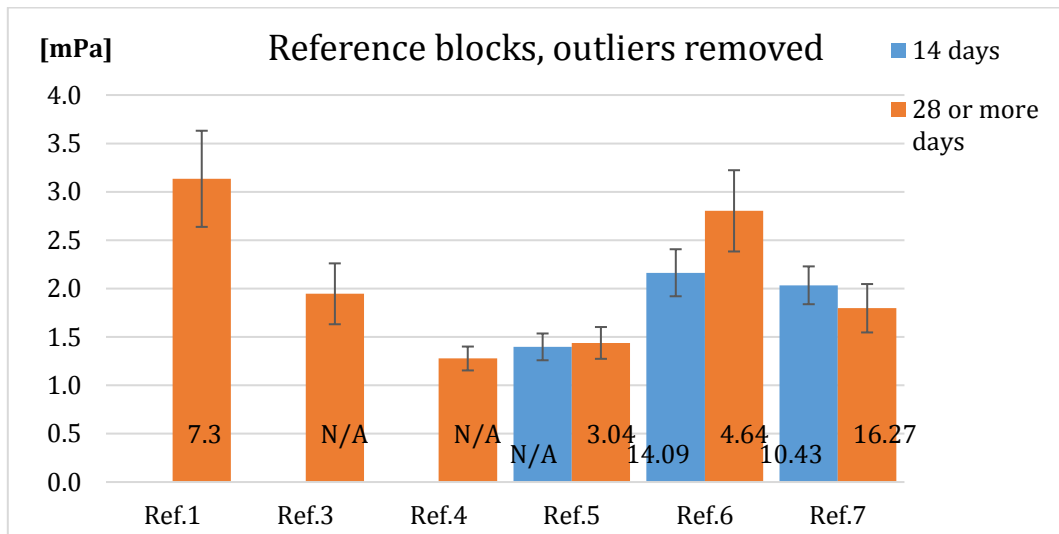
Figure 8-16 - Compressive strength of reference blocks, samples more than 25 % from average excluded

As can be seen, the different samples have very different compressive strengths.

- Ref.1 was seen to have a much tighter structure, probably due to the higher water content, and this could be the cause of the higher strength.
- Ref.2 is not part of the plot, as it did not qualify to be taken into consideration. One of the three samples were by accident tested wrong, and according to DS/EN 1354 a test must consist of at least 3 samples.
- Ref.3 does not seem to have developed extra strength, even though it had 49 days to harden. This again proves, that it is the LWA that is the strength limiting factor for the blocks.
- Also it can be seen that the difference between 14 day 28 days strength are not significant, which implies that the strength of the paste surpasses that of the LWA sometime before the 14 day mark.
- Ref.4 and Ref.5 are the lowest two of the samples. This could be explained by the fact that these were compressed instantly instead of gradually. This could cause the LWA to pack badly, and subsequently give a lower strength. Also, when compressing Ref.5, the machine tilted due to overload, and this can have damaged the block.
- The strength of Ref.6 and Ref.7 at 28 days is very different. It was seen earlier that Ref.6 had a density somewhat higher than the rest of the references, and this is probably the cause of the higher strength.

Based on this, the reference strength is decided to be the average of Ref.3 at 49 days, and Ref.6 and Ref.7 at both 14 and 28 days. This comes out to 2.15 mPa, and as the prescribed strength of the Weber blocks is 3 mPa, it is found that the lower quality of casting reduced the strength with about 28 %.

8.4.3.3 Wood ash samples
On Figure 8-17 the compressive strengths found for the wood ash samples can be seen. In this figure, all of the samples are included. In appendix P1-DA-22, the data that it is based on can be seen. On Figure 8-18 they can be seen only including the ones within 25 % of the average.
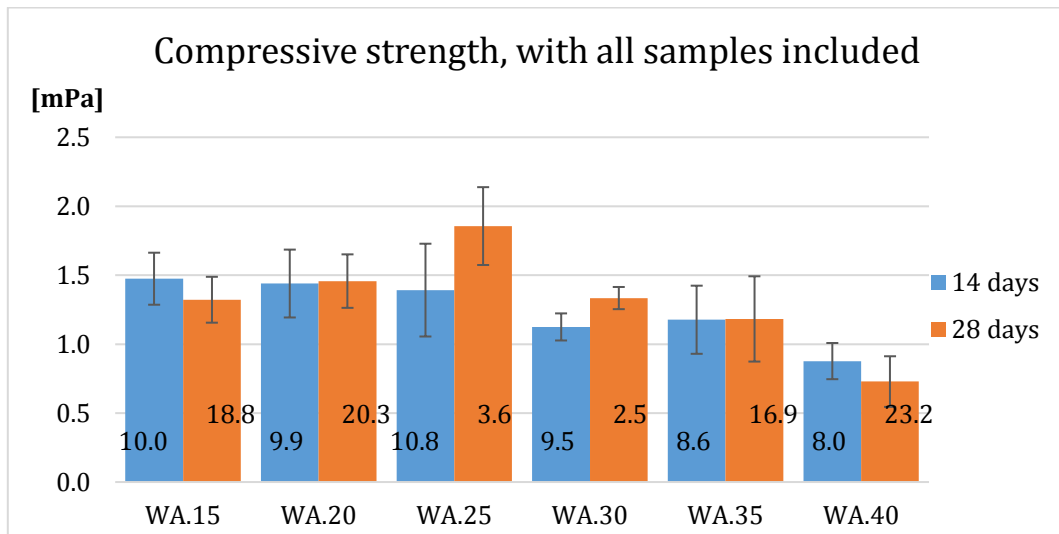
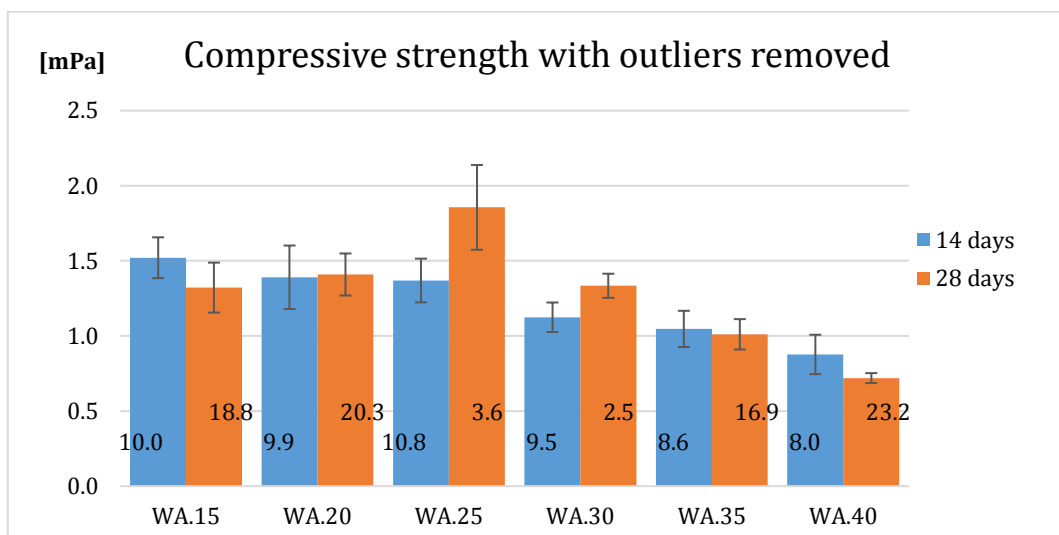*Figure 8-17 - Compressive strength of samples containing wood ash*

Figure 8-18 - Compressive strength of samples with wood ash, excluding samples more than 25 % from average

Here it is again clear, the there is no significant difference between the 14 and 28 days strengths, as the strength of the cement paste has surpassed the strength of the LWA before the 14 day point.

It is also clear, that the strengths for the blocks from WA.25 and down are pretty similar, with the exception of WA.25 at 28 days. Again, it seems that it is the LWA that is the strength limiting factor, as long at the cement paste can hold it together. At 30 % WA the fraction of ash has become too great, and the strength suffers.

The overall strengths are lower than those of the references. It could be that the addition of ash impacts the adhesion between the paste and the LWA, and that this lowers the strength to a point where another factor takes over being strength defining. This keeps the strength steady until the addition becomes great enough, at which point it is again defining the strength.

To see how strong the WA block could potentially be, had they been cast on Webers block machine, the strengths have been corrected for the casting error of 28 % found from the references. The resulting strengths can be seen on Figure 8-19.
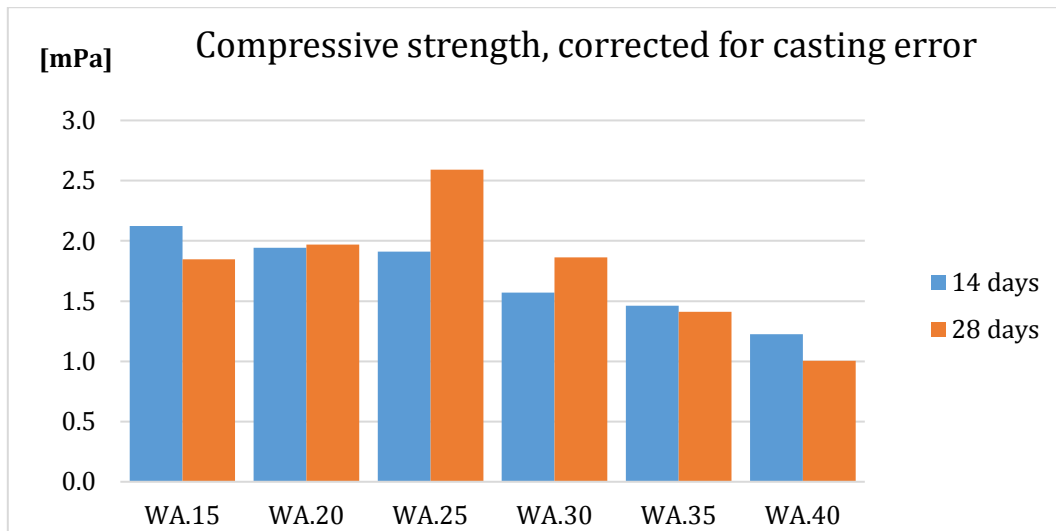

Figure 8-19 - Compressive strengths, corrected for casting error

As can be seen the three first replacements reach a strength of around 2 mPa, which according to Weber is an acceptable result. Given that the compensation for the casting error is fair, it seems that around 25 % of the cement used for the blocks could be replaced with wood ash, and the resulting block would still be usable.

It was earlier found, that the LOI might be as high as 11 %. In ordinary concrete, this would be unacceptable, the reason being, that the organic matter would decay over time, causing voids. But seeing as the LWA blocks are already made up of mostly voids, a few more would not affect the overall integrity of the block.

As the large LOI did not interfere with the casting, this could actually mean that all the ash, unusable for ordinary concrete based on LOI, could potentially be used for LWA concrete.

8.4.3.4 Difficulties testing blocks

All of the compressive strengths are calculated based on the force it took to break the sample in kN, and the area of compression in mm². In all of the calculations, the area is set to precisely that of a circle with a diameter of 75 mm, and in most cases this gives perfectly good results. However, when dealing with some of the blocks with a high ash content, this is not a perfect approach. As these blocks tend to be harder to cut, a lot of them have broken edges, which leads to a smaller area of compression, as can be seen on Figure 8-20. This is not just a problem for the blocks with a large fraction of ash, even though it is more commonly seen there.
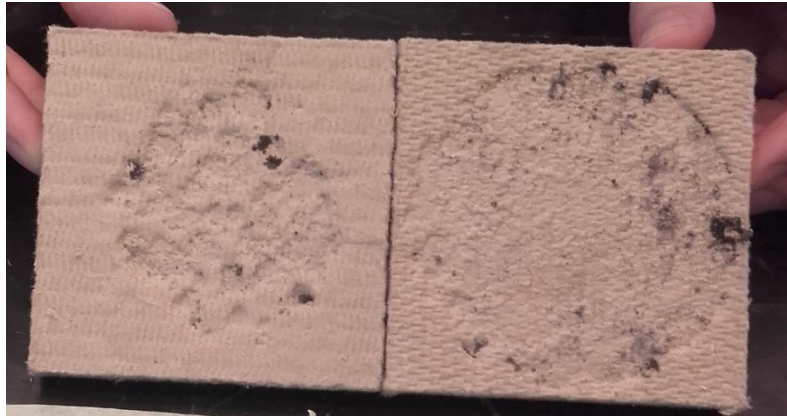
Figure 8-20 - Comparison of area of compression. On the left is the mark from a sample with broken edges, on the right is the mark from a complete sample

This is of course a problem, and a solution could be to press the plate down into an inkpad, take a picture, and use image recognition software to find the precise area of compression. The pressure place could even be used as a stamp, and stamp the area onto a piece of paper, to be analyzed later.

8.4.4 Leaching

Once the samples had been tested for compressive strength, and dried out to find the water content, one from each block was taken out, crushed and tested for leaching. The results can be seen in Figure 8-21 and in appendix P1-DA-23.
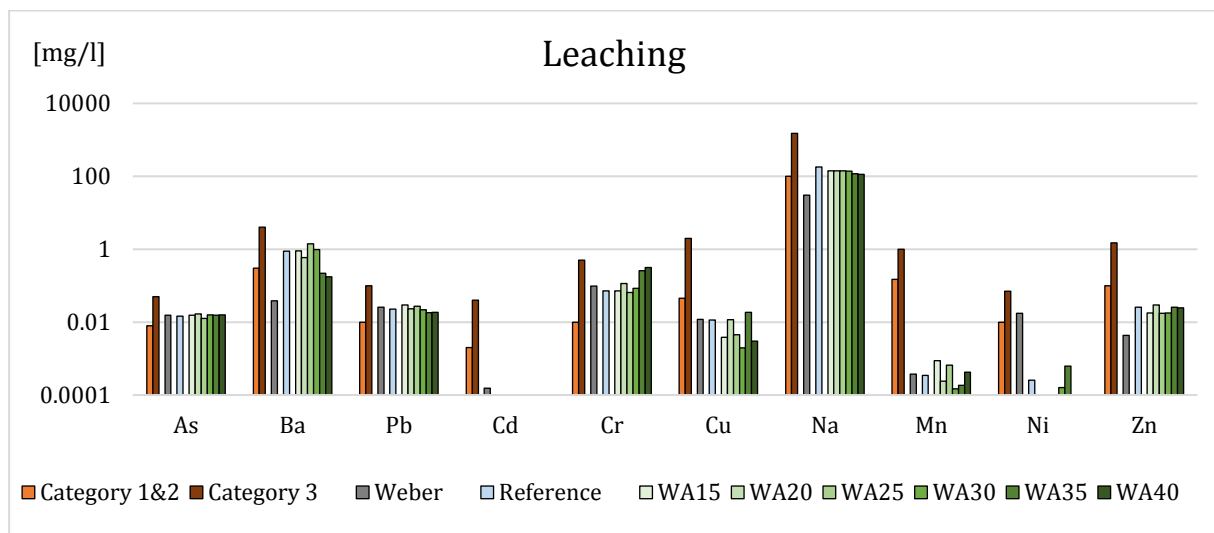
Figure 8-21 - Concentrations found in leaching experiment

The concentrations shown are in mg/l made from a mix of one part solid to two parts liquid, L/S 2. The Category 1&2 and 3 columns refer to the limits set by "Restproduktbekendgørelsen" [12], regulations set by the Danish ministry of environment. Category 1 and 2 are identical in this aspect, and so are presented together.

As can be seen, all of the samples stay within the limits for category 3, while many of them breach category 1&2. However in most of the places where the WA blocks breach the limit, so does the Weber block. So given that the measured values are correct, and that Weber has all of their permits in order, the WA blocks can be used in all the same places that Weber blocks are used today.

9. CONCLUSION

In this part, two different ashes were considered to be used as cement replacement in the making of lightweight concrete. They both underwent a preliminary examination, and based on solubility in water and loss on ignition, one of them were picked. It did however, still need to be washed, as it had a solubility of around 13 %. The washing was done in a big custom made pot, and resulted in the heaviest particles separating at the bottom. Both these, the main part of the washed ash, and the untreated ash were examined for their concentrations of heavy metals, and it was found that some of them were too high. The washing somehow increased the loss on ignition to over 12 %. No explanation has been found, and the ash was used anyway.

A lot of work went into refining the mix design, and this included finding the absorption rates for the LWA, and the activity factor for the ash. Both of these meant an increase in water to the mix, but the addition of water ended up having to be even higher, due to differences in casting methods.

The casting was done on a block machine and a lot of time was spent, figuring out how to get the best results in casting, as there were multiple variables. After the blocks had been cast and had cured they were cut into smaller samples and tested, both for compressive strength, density and porosity, capillary suction and leaching. It was found that they performed comparable to commercially available blocks in both leaching, density and porosity. The capillary suction was higher, and seem to be linked to the amount of ash added.

The method for testing compressive strength was first refined on commercially available blocks with a compressive strength of 3 mPa, before testing started on blocks cast in the project. After finding the right set of parameters for testing, a lot of reference samples were tested, and it was found that the lower quality casting had made a difference in compressive strength of about 28 %. Next the blocks with wood ash in them were tested, and it was firstly found that there was no difference between the 14- and 28-day strengths. Secondly it was found that the ash caused the strength to drop a bit, but it was almost the same for all the blocks with 25 % or less ash. At 30 % the strength started to drop. Theoretical compressive strengths, based on the casting error of 28 % were calculated, and it was seen that from 25 % and below they were around 2 mPa, which makes them interesting for the block manufacturer.

The high loss on ignition did not seem to effect the block in a negative way, and it is speculated that any ash that is discarded for other used due to a high loss in ignition, might be usable in lightweight concrete.

10. FURTHER RESEARCH

- Ideally, the next step is upscale testing, in an industrial setting. It would be interesting to see, if the same results could be repeated.
- A verification of activity factor, by testing on concrete mix instead of mortar, would be in order.
- It was seen that the compressive strength was more or less the same for 14- and 28-day tests, which would imply that the final strength was reached sometime before the 14-day mark. It would be interesting to see, at what point within the 14 days the strength is reached, and what effect the ash has on this.
- It was found in the wood ash, that the LOI was very high, which would imply a large amount of organic matter. Tests ought to be conducted in what effect the decay of the organic matter has on the compressive strength.
- Around 30 % of the binder in the investigated lightweight aggregate concrete is coal fly ash. Replacing some of this with a bio ash might prove interesting.
- The blocks were drilled into cores at different maturities, and it has not been looked into what effect this has on the final compressive strength. It should be examined.
- The hardening in these projects have been done in a climate-controlled room, but there were still problems with samples drying out. It could be looked into if hardening the samples under water would yield results that are more consistent.

11. SOURCES OF ERROR

Though calculations were carried out to estimate the amount of water required for the LWA and bio ash, it became clear when mixing the concrete that extra water had to be added in order to obtain a desirable consistency. It is assumed that the extra water does not change the W/C_{eq}, but it is impossible to know for sure.

Lifting the block machine after creating a lightweight aggregate concrete block was not always conducted as vertically as intended, mainly because of it being difficult to steer a forklift gently and precise. In various cases the top of the block was damaged and had to be cast aside, but the remaining coherent block was used even though the structure might have been damaged during the lifting of the machine.

After drilling and cutting samples some specimens were partially filled with a mixture of dust and water from the process. The dust and water mix was removed as much as possible, but it is plausible that some was left causing an increasing of the mass.

DS/EN 1354 states that the water content of a specimen should not be lower than 4 % when tested for compressive strength. However, this was the case in several cases of the 28-days strength tests. Though the test results do not give reason to believe it, the low water content might have affected the compressive strength. DS/EN 1354 also states that a block should be cut into smaller specimens, at the earliest, 7 days before the testing date. As the cylinders for testing both 14 and 28 days strengths were cut from the same block, on the same date, all the specimens for 28 days strengths have been cut too early.

Some blocks turned out to have poor resistance when drilling cylinders, causing edges to break of the specimens. This made the measuring of the volume for some samples very cumbersome leading to imprecise densities and perhaps a misleading compressive strength.

Part 2

12. INTRODUCTION

While working on an experiment for Part 1 of this report, the water content of some LWA 0-2, was needed to be known in a hurry. For this reason, some of it was weighed off, and put in a microwave oven for 10 minutes, after which it was left to cool down for 10-20 minutes. When it was again looked at, it was clear that something unexpected had happened. Some of it had formed a large ball, as can be seen on Figure 12-1.

Figure 12-1- LWA 0-2 sintered together

When the ball was broken open, it looked a lot like a regular piece of LWA, only a lot bigger.

This indicates that the LWA has gotten hot enough to not only sinter together, but also release gasses, that was not already released when it was sintered at the factory. This would imply that it got hotter than the 1150° C it had previously been heated to.

This could mean, that it is possible to make LWA, using microwave heating instead of conventional heating. And given that it is possible, it would also be possible to investigate other materials to be used to make LWA, such as ash, in small scale using only a microwave oven. The investigation of these possibilities is what makes up Part 2 of this report.

13. THEORY

To figure out if it is possible to make LWA using a microwave oven, it is first important to know how LWA is made conventionally, and equally important to know what is causing this behavior.

13.1 PRODUCTION OF LWA FROM CLAY

Most LWA including that from Weber is made from clay, in a process not unlike the one used for cement production. The clay is fed into one end of a large cylindrical rotating oven that has a heat gradient over its length. In the start of the oven, the moisture is driven off of the clay, and it forms large clumps. These are crushed by the rotation, and the smaller pieces are moved along the length of the oven. In the end, the oven is around 1100-1150° C, and this causes the clay to sinter together, and all of the organic material to burn off, creating CO_2, that forms a lot of voids. It is these voids that cause the aggregate to be so relatively lightweight. In some cases, depending of the composition of the clay, organic matter is added to increase the amount of CO_2 produced.

13.2 PRODUCTION OF LWA FROM ASHES

Studies have been done, in the production of LWA from incinerator bottom ashes (IBA), among others. S. Bethanis and C. R. Cheeseman [15] have made LWA from IBA, enriched with pulverized fuel ash (PFA) with promising results. The method for making the LWA from IBA is a lot different than the method used for clay. What they did, was to first mill the ash to have a similar particle size to that of clay, after which they dried it, crushed it and then pelletized it using a revolving drum. The pellets of the desired size were then rolled in PFA, before they were dried again and then sintered in a revolving furnace at 1040-1140°C.

This method is very cumbersome, because the IBA has to be made claylike for the method to work.

13.3 THERMAL RUNAWAY

Thermal runaway is a mechanism where an increase in temperature changes some properties that leads to a further increase in temperature; a positive feedback loop that can lead to extreme temperatures. The phenomenon is seen in many different fields, such as chemistry and electronics. For a civil engineer it will be best known from the heat release of large quantities of curing concrete.

This study however is concerned with the thermal runaway that can occur to some ceramics in a microwave oven.

The way a microwave oven works is by emitting electromagnetic waves, causing some molecules to warm up. The molecules affected by microwaves are what is known as dipole molecules. These will want to align their poles with the poles of the waves, and as these keep changing, the molecules start spinning in what is known as molecular dipole rotation. This rotation cause them to bump into other molecules, and thus make heat. This mechanism is called dielectric heating. In materials normally heated in a microwave i.e. food, the most predominant dipole molecules are water and fats.

Some molecules get more susceptible to dielectric heating, as they get hotter. This can cause thermal runaway. This is a known phenomenon, and is why not all ceramics and glasses are microwave safe. According to Kenkre et al [16] the components in ceramics that are prone to thermal runaway is especially iron-oxide, zinc-oxide, alumina, chromia and silica.

14. MATERIALS AND METHODS

To figure out whether this method can be used, for small scale experiments with ash, it is first important to know if the first time was a random occurrence, or not.

A glass jar is filled with LWA 0-2 and put in the microwave oven for 10 minutes, and then taken out immediately. It was clear that it had reached a very high temperature, as it was glowing red, but no nut had formed. It was concluded that the stirring immediately after the jar had been taken out of the oven, had popped the bubble, before it had time to set. The test was repeated, but this time, the jar was left for 20 minutes, after the oven had stopped. This time a nut formed once again, a bit smaller than the first one, but other than that, exactly the same. As it was now confirmed that the method was repeatable, the work with the ash could begin.

14.1 FINDING A SUITABLE ASH

As discussed in chapter 13.3, the molecules that are best known for causing thermal runaway in ceramics are iron-oxide, zinc-oxide, alumina, chromia and silica. For these tests, it was deemed a good idea to pick an ash that was known to have a high concentration of at least one of these. The other project running parallel with this one, was working with a sewage sludge ash (SSA). It had the same red color that most SSA's have, due to the fact that iron-oxide is used in the cleaning process of the sewage sludge. This implies that is has a high iron-oxide content, and for this it was picked.

14.2 TESTING THE ASH

There are two things that needs to be present in the ash for it to be capable of making LWA using thermal runaway. There need to be a sufficient concentration of a material that can go into thermal runaway, and there need to be a sufficient amount of material that can produce gas when heated up.

To find out what a sufficient concentration is, the easiest approach is to test the LWA and the nut formed from the LWA, to see what was in it before it sintered together, and to see what was used up.

The concentrations of the different metals are found in the same manner as discussed in chapter 7.1.1.5, using strong acid and high temperature to dissolve the metals, and then analyzing them using ICP. The analysis was done, on the untreated LWA 0-2, as well as on the ash.

The concentration of materials that produce gas when heated to a high temperature was tested in two different methods.

Firstly the concentration of organic matter was found, using loss on ignition, in the same way as described in 7.1.1.3. This was only done on the ash, as the LWA has previously been heated to 1150° C, and so no organic matter is expected to be left.

Secondly the content of carbonate was determined. This was done by adding hydrochloric acid to a sample, and looking at the amount of CO_2 it produces. A more detailed description can be found in appendix P2-ME-01. This was done on the untreated LWA 0-2 and on a crushed sample of nut. Why it was not done on the ash, will become clear in the following.

14.3 EXPERIMENTS WITH THERMAL RUNAWAY

As no one seems to have done this before, there is no predetermined test method to lean on. So the testing with the ash started out using the exact same method as had been used with the LWA, and was refined from there.

The first test was done using approximately 500 g of ash in a glass jar, which was simply put in the microwave for 10 minutes. This proved to be an unsafe method, as the microwave oven broke.

The second method tried, was where a small amount ~5-6 g of ash was incased in sand, as can be seen on Figure 14-1.
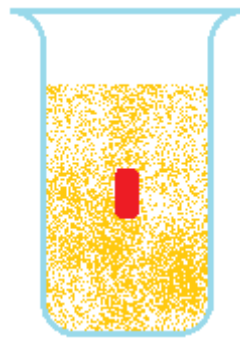
Figure 14-1 - principle sketch of ash incased in sand

The sand was both to protect the oven from the hot ash, but also to insulate the ash, so it had time to set, before losing too much heat to the surrounding air. This method was tried a number of times, with many different setups, but to no success.

The third method involved different amounts of ash, placed in a ceramic crucible, on top of a petri dish full of sand, as can be seen Figure 14-2

Figure 14-2 - Setup with ash in crucible

Using this method, the microwave radiation can go straight to the ash, and there is still some sand to protect the oven from the hot ash. This method worked, and a number of experiments was done using it.

15. RESULTS AND DISCUSSION

15.1 CONCENTRATION OF METALS

The concentrations of different metals in both LWA 0-2 and SSA was found and can be seen in appendix P2-DA-01. The important one for this are the aluminum, chrome, iron, silicon and zinc, and the respective concentrations of these can be found in Table 15-1.

Table 15-1 - Content of relevant metals in mg/kg and % of sample B.W.

			Aluminum	Chrome	Iron	Silicon	Zinc
SSA	Average	[mg/kg]	36613.17	43.41	65052.00	1018.68	2340.31
	Deviation	[mg/kg]	1424.75	0.14	1434.66	240.06	24.13
	% of sample	[%]	3.66	0.00	6.51	0.10	0.23
LWA 0-2	Average	[mg/kg]	11211.53	13.84	15115.00	1031.80	56.44
	Deviation	[mg/kg]	1737.59	0.24	579.50	690.68	14.74
	% of sample	[%]	1.12	0.00	1.51	0.10	0.01

As can be seen, the concentration of all of the relevant metals are higher in the SSA than it is in the LWA, apart from Si that is basically even. It can also be seen, that the iron concentrations are highest in both cases. The leads to the conclusion that it is probably the iron-oxide that are responsible for the thermal runaway.

The method used to measure the concentrations, cannot tell how the metals were bound before being dissolved. As thermal runaway only occur if they are in the form of an oxide, there is no way of knowing if these concentrations actually correspond with the reactivity regarding thermal runaway. For the iron however, there is a clue in the color, as iron-oxide is red and the ash is reddish brown.

Seeing as it was possible to get the LWA to produce thermal runaway, and given that it is the iron-oxide that is responsible, it is assessed that it should be possible to get the ash to do the same, even if it is diluted to a 1/4 concentration with some other material. This gives rise to the opportunity to dilute it with something containing a high amount of carbonates, or other things that can produce gas at high temperature. Because of this, an iron oxide rich ash can be used, even if it does not contain carbonates in very large amounts.

15.2 LOSS ON IGNITION AND CARBONATE

The LOI for the ash were measured, and was found to be around 0.23 %. This is relatively low, but could contribute to the formation of gasses at high temperatures.

The carbonate content was measured for the LWA and the nut respectively, and the results can be seen in Table 15-2 below.

Table 15-2 - Carbonate content in nut and LWA

Sample	Sample [g]	CO_2 [mL]	$CaCO_3$ [%]	Average [%]	Standard Deviation [%]
Nut					
A	2.5004	12.2	1.9		
B	2.5012	8.4	1.3		
C	2.5009	8.4	1.3	2.07	0.67
A	1.0018	7.1	2.8		
B	1.0018	7.1	2.8		
C	1.0039	5.4	2.1		
Leca					
A	2.5008	9.8	1.6		
B	2.5001	8.8	1.4		
C	2.5009	8.0	1.3		
A	1.0016	7.2	2.9		
B	1.0012	7.2	2.9	2.00	0.65
C	1.0002	7.0	2.8		
A	2.5000	11.0	1.8		
B	2.5000	11.0	1.8		
C	2.5000	11.0	1.8		

The carbonate content of the nut, appears to be larger than that of the LWA, which is counterintuitive. But as can be seen, there is a very large deviation between the different groups of experiments. The reason is probably that there is so little carbonate in both the LWA and the nut, that the measuring technique is not very accurate.

If nothing else, the analysis show, that it does not require very much carbonate for the sintered LWA to blow up, and create additional voids. For this reason the test was not conducted on the SSA.

15.3 EXPERIMENTS WITH THERMAL RUNAWAY

15.3.1 Method one
As described earlier, the first method used simply, consisted of a jar filled with ash, and put in the oven. It worked, in the sense, that a nut was formed, however it broke the oven. What happened was, that the nut formed on the bottom of the jar, making both the jar and the glass plate of the oven, hot enough to fuse together and break, as can be seen on Figure 15-1.

Figure 15-1 - Broken glass jar and microwave plate. The ash and plate is wet in an effort to cool it down.

In addition the heat also caused the plastic part of the oven, the makes the glass plate rotate, to melt. For this, the method was deemed unsafe, but it did succeeded in making a nut, as can be seen on Figure 15-2.

Figure 15-2 - First nut made from SSA

It is around 3-4 cm in diameter, and black and shiny on the inside as can be seen on Figure 15-3. Opposed ordinary LWA it mainly consists of one large void, instead of many small ones.
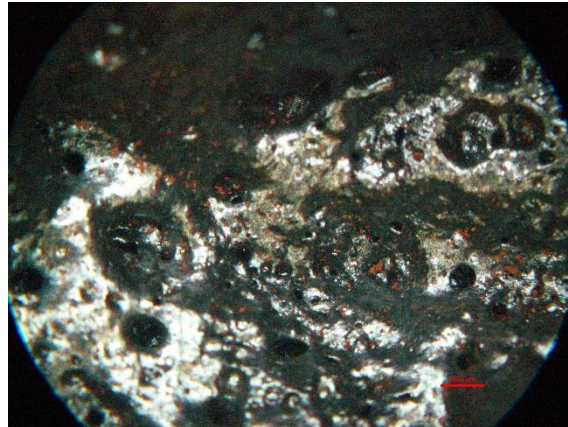
Figure 15-3 - Inside of the nut made from SSA

The outside is covered in unchanged ash, but is has some cracks, as can be seen on Figure 15-4.
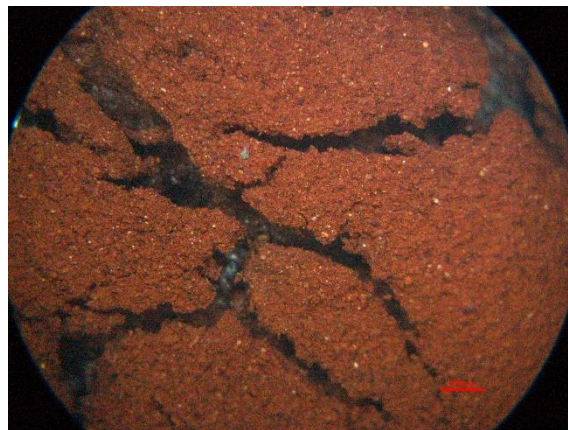

Figure 15-4 - Outside of the nut made from SSA

15.3.2 Method two

The second method used for the next five experiments, did as described above, involve packing a small amount of ash in sand. The results are summed up in Table 15-3.

Table 15-3- Summation of experiments using method two

Experiment Number	Amount of Ash	Time	Sand	Result
[-]	[g]	[Minutes in / Minutes rest]	[-]	[-]
2	5.85	10/20	Sea sand 0-4 mm	The ash formed a loose ball that fell apart when touched.
3	6.4	10/20	Sea sand 0-4 mm	Same result as 2.
4	6.59	10/20	Great belt sand, 0-2 mm, class E	Same result as 2.
5	~12	10/20	Great belt sand, 0-2 mm, class E	Same result as 2.
6	6.15	15/20	Great belt sand, 0-2 mm, class E	Same result as 2.

As can be seen, none of these experiments seemed to work. Whether the sand took up to much of the microwaves, or just removed too much heat due to the large area of contact is unknown. Because of the lack of positive results, the third test method was taken into use.

15.3.3 Method three

As described above the third test method involved placing the ash in a crucible. The results from these experiments can be seen in Table 15-4.

Table 15-4 - Summation of experiments using method three

Experiment Number	Amount of ash	Time	Result
[-]	[g]	[MM:SS]	[-]
7	5.36	10	No result
8	~20	7	The ash sintered together to form a crucible shaped nut.
9	14.6	4:51	Started glowing at 4:30, oven opened at 4:51, ball deflated.
10	10.55	7	No reaction.
11	11.55	6:11	No reaction, oven stopped by it self
12	12.77	8	No reaction, oven stopped by it self
13	13.54	5	No reaction, oven broke.

As seen in the table, the experiments stopped when the second microwave oven broke. It had turned off multiple times, most likely because it was getting too hot, but after the last time of turning off, it would not turn on again.

Apart from that, it seems that there is a critical mass of ash for this setup, somewhere between 13.54 g and 14.6 g, under which the thermal runaway does not happen. This is most likely due to the fact that the process does not start before a certain temperature is reached. Thermal runaway works differently on different materials. For some the heat increase over time will go up fast and level out, for others it will heat up slow until a certain temperature where it then accelerate fast. It would seem that the SSA falls in the second category.

If the mass of ash is too small, it might conduct the heat away faster, than it receives it from the oven, and so never reach critical temperature. This could also mean, that method two could potentially work, if only there had been enough ash. The amount might need to be higher than in method three, as the contact area with the sand is very large, and so a lot of heat will be directed away.

It also means that in a bigger production, it might be possible to heat the ash to critical temperature using any method, and then only using the microwaves for the actual thermal runaway. As the critical temperature is not known, it is unknown if this would be more efficient than just using microwaves.

15.3.4 Alternative reason for expansion

As seen above, the inside of the nut created from SSA is dark and shiny, and could almost look like metallic iron. As the iron transitioned from being iron-oxide to pure iron, some oxygen must have been released, and this could be the cause of expansion.

In experiment number 9, it was seen that 14.6 g of ash went into thermal runaway. It is known that the sample is 6.51 % iron, which means that there were 0.95 g of iron present in the ash. Assuming that all the iron is in the form of iron(III)oxide, and that all of it changed form, the amount of oxygen released can be found. First the amount of iron in the sample in moles is found using:

$$n = \frac{m}{M}$$

where:

n the number of moles of an element

m mass of element in g

M molar mass of element in g/mole

$$n_{Fe} = \frac{m_{Fe}}{M_{Fe}} = \frac{0.95g}{55.845g/mol} = 0.017 \; mole$$

Iron(III)oxide consists of two iron atoms, and three oxygen atoms, meaning that the amount of oxygen moles is:

$$\frac{0.017}{2} \cdot 3 = 0.026 \; mole$$

The ideal gas law goes as follows:

$$p \cdot V = n \cdot R \cdot T \rightarrow V = \frac{n \cdot R \cdot T}{p}$$

where:

p pressure in Pa

V volume in m^3

R gas constant, set to $8.314 \; J \cdot K^{-1} \cdot mol^{-1}$

T temperature in kelvin

Assuming that the temperature during thermal runaway is around 1200° C, and that the pressure is equal that of the atmosphere, the volume of the released oxygen would be:

$$V = \frac{0.026 \; mol \cdot 8.314 \; J \cdot K^{-1} \cdot mol^{-1} \cdot (273.15 + 1200)K}{101325 \; Pa} = 0.003 \; m^3 = 3.08 \; l$$

This is clearly grossly overestimated in almost every step, but even a fraction of that amount would be more than plenty to inflate the nut. It could also be that this is the mechanism that inflates the LWA

As the nut has not been analyzed, it is unknown if this is the reason for the inflation or not.

16. CONCLUSION

Investigation has been done on the possibility of using thermal runaway in the production of lightweight aggregate. As if was first seen by accident in LWA, it was first proven to be reproducible. As it was, the concentrations of certain metal-oxides along with carbonate were measured to see, what would be sufficient amounts to cause thermal runaway. After this a sewage sludge ash was found, that met the newly found requirements.

A preliminary test method was developed, and using this, it was proven that it is possible to make LWA from SSA. The investigation did not get further than this due to time constrains.

17. FURTHER RESEARCH

There is a large work of research being done at the institute, in cleaning out the phosphorus from SSA by means of electrodialysis, to reuse it for fertilizer. After the cleaning, a lot of ash is left, and as before it was cleaned, it is still an unused resource. If it was possible to use this ash for production of LWA, a whole category of waste will be turned into a double efficient resource. From one of the PhD students working on this project, the concentrations seen in Table 17-1 are known. These were sent in a mail, seen in appendix P2-MA-01.

Table 17-1 - Concentrations of Iron in cleaned ashes

Type of ash		Gasification				Incineration					
Experiment	[-]	1	2	3	4	1	2	3	4	5	6
Iron content	[%]	8.00	8.20	8.00	8.10	10.20	9.80	7.10	6.80	9.60	10.30

As can be seen, all of the different experiments in cleaning methods he has been working on, leaves an ash with a more than sufficient iron content for the thermal runaway process to be theoretically possible. The PhD reported that the incineration ash is red, indicating that it could hold iron-oxide. The gasification ash on the other hand is gray, so it is not certain that it would work.

In addition the experimental setup should be refined to a point where it produces consistent results, without breaking microwave ovens.

18. SOURCES OF ERROR

As testing did not get very far, there are no large sources of error to mention. As could be seen the analysis of the carbonate content did not conclude anything, as the concentrations were too small. Another test method should be employed.

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