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Exploring the Viability of Using WtE Incineration Ash as a Cement Replacement in Concrete

Master's thesis in Materials Chemistry

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CHALMERS UNIVERSITY OF TECHNOLOGY
Gothenburg, Sweden 2024

MASTER'S THESIS ACEX30

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ABSTRACT

The increasing importance of reducing greenhouse gas emissions demands new environmentally sustainable building materials. In the concrete industry most of the emissions comes from the production of cement. Research on materials that can be used as replacements for cement (SCMs) could help reduce the climate impact of the concrete industry. One of the possible SCMs that could be used are ash from WtE incineration. In this report the viability of the use of three different ash fractions from WtE incineration as cement replacement are be evaluated. These are a fly ash sample, an incinerator bottom ash sample and a mineral fraction incinerator bottom ash sample. Chemical and mechanical properties was evaluated using different techniques including a modified R3 method, compressive strength tests, PXRD and leaching tests with ICP-MS. It was found that all three of the ash fractions had chemical reactivity and the mechanical strength tests suggested that two of the ash fractions fly ash and MIBA had effect on the mechanical strength while the slag did not have ant effect. The results from the leaching tests were compared to thresholds on allowed limits of leaching from monolithic concrete samples and found to be below these thresholds. However, the limited knowledge and regulations on monolithic samples made the interpretation of these results difficult. The result from this thesis suggests that the use of WtE incineration ash a SCM is a possibility but that more research is needed. It is also concluded that the need for more and less complicated to understand regulations regarding leaching from monolithic concrete is needed in Sweden and that the current lack of regulations might be hindering the adoption of new sustainable building materials.

Contents

Preface

In this project mechanical and chemical tests have been carried out to investigate if ash from WtE incineration can be used as a cement replacement and/or filler. The project is done in cooperation with Renova AB and Chalmers University of Technology. The project investigates the possibility of using ash to create new more environmentally sustainable building materials.

Mechanical test has been done to investigate the mechanical properties of concrete containing WtE incineration ash. These tests were carried out at the Department of Architecture and Civil Engineering, Division of Building Technology, Chalmers University of Technology, Sweden. Leaching tests and chemical analyses have also been carried out to investigate the leaching of metals from monolithic concrete cubes containing WtE incineration ash. These tests were carried out at the Department of Architecture and Civil Engineering, Division of Water Energy Technology, Chalmers University of Technology, Sweden. I would like to thank Bruno Oliveira Goncalves and Amir Saeid Mohammadi for their help and supervision in the laboratory work.

Göteborg June 2024

Joel Eriksson

List of Acronyms

- WtE – Waste to Energy
- SCMs – Supplementary cementitious Materials
- MSW – Municipal Solid Waste
- PXRD – Powder X-ray Diffraction
- ICP – Inductively Coupled Plasma
- MS – Mass Spectrometry
- FA – Fly Ash
- IBA – Incinerator Bottom Ash
- MIBA – Mineral fraction of Incinerator Bottom Ash
- R3 test – Rapid, Robust, Relevant Reactivity Test
- ASTM – American Society for Testing and Materials
- EN – European Norm

1 Introduction

In Sweden the concrete industry is responsible for about 4% of the total emissions of CO_2 equivalents annually. Around 90 % of these emissions comes from cement which is the main component of concrete. The industry has set up goals to mitigate the emissions caused by concrete production. By 2030 there should be climate neutral concrete on the market and by 2045 all concrete on the Swedish market should be climate neutral [1]. One way of reducing the climate impact of the concrete industry could be to use other materials as partial replacement for cement. These materials are often referred to as supplementary cementitious materials (SCMs) and are materials that have similar properties to cement. One material that could possibly be used is ash from waste-to-energy incineration (WtE). These ashes are by product of the production of electricity and heating from incineration of waste. At one waste to energy incineration plant in southwestern Sweden 550 000 metric tonnes of waste are incinerated yearly meaning that there is also a lot of waste ash that could be used. The use of ash as a cement replacement would be mutually beneficial for both the concrete industry and the ash producers. For the concrete industry it would lead to a decreased need of mining new raw material from nature and a decrease of emission from cement production. The ash producers would also be benefiting since the ash materials could be used in more areas and therefore get new value. This would also in the case of the fly ash reduce the need for landfilling. Using ash from the WtE incineration process as replacement in concrete could contribute to the adoption of new more sustainable building materials. It would also contribute to a more circular society.

As previously mentioned, the ash materials used in this project come from a WtE incineration plant in southwestern Sweden. The plant uses waste both from the local area and imported waste as fuel in the process. In the end the fuel is converted into electricity and heating for the surrounding area. About half of the waste comes from households and the other half comes from businesses [2]. This means that there is a wide arrange of different combustible materials that are being incinerated. These could be wood, paper and plastic but also different metals that can be found in the mixed waste. Below an overview of the WtE incineration process can be found in figure 2.1. Three different ashes from this process will be investigate during this project. These being fly ash, slag and MIBA that are taken out from different parts of the waste incineration process. The ash fractions are further explained in the section 3.1.

As previously mentioned, the source of the waste comes from mixed waste from household, industry and are imported. This means that the ashes that are taken from the process can contain a variety of different materials. Inorganic minerals such as SiO_2, CaO, Al_2O_3 and Fe_2O_3 that are the reason that this material might be a suitable replacement for cement [3]. But it also contains a wide range of different metals which is one of the challenges in the adoption of concrete containing ash as a new building material [4]. To be able to use as a building material it will need to meet certain guidelines. One of them being the amount of metals that leaches out of the building material. The current regulations

describe how much metals are allowed to leach out of a ground up powder form of the material and there are no guidelines for leaching from monolithic samples. The aim in using ash in concrete is to use the material as a building material meaning that the material will be found in monolithic form. It is therefore of interest to investigate the leaching from monolithic concrete samples since this is a better representation of how the leaching could occur when the material is used. Regulations that do not fairly investigate the material may hinder the adoption of new more environmentally sustainable building materials.

1.1 Aim and goal

There are two aims for this project.

- To investigating if ash from WtE incineration could be used either as a cement replacement material or as filler in concrete.
- To investigate the leaching of metals from monolithic samples of concrete containing ash as cement replacement.

The goal of this project is to make an initial investigation into the possibility of using WtE ash in the production of more environmentally sustainable concrete. Also to investigate if regulatory hurdles hinder the use of ash in concrete.

1.2 Formulation of research questions

The report tries to answer the following question:

- How does the addition of ash effect the mechanical properties of the concrete?

- Which metals leaches out of monolithic concrete containing WtE ash as cement replacement?

- Do the amount of leached metals exceed current available Swedish regulations?

1.3 Limitations

Due to the extensive time requirement of the material preparation process needed to get enough material for the experiments repeated tests where not made. This was due to the low scale of the lab equipment for grinding the material which did not make the production of enough powder possible. The test made in this report will instead be seen as initial testing. The time constraints also limited the curing time of concrete samples and affected the time and choice of which leaching test was carried out. To measure reactivity the modified R3 method was chosen because it was expected that the ash samples would not be substantially reactive enough to get results from methods designed for more reactive materials like the Frattini test.

2 Background

2.1 Concrete

Concrete is a material which consists of aggregates water and cement. The Aggregates can be sand and/or gravel that make up the biggest part of the concrete material. The aggregates size and shape influence the properties of the concrete this can be strength, stability and workability [3]. Another and perhaps the most important ingredient to concrete is cement which consists of limestone (calcite), clays and shale. The cement is what binds the concrete material together. When the cement is mixed with water it will undergo chemical reactions called pozzolanic reactions to form a paste that binds the material together. In this project it will be investigated if WtE ash can be used as either an aggregate filler in concrete or as a Supplementary Cementitious Material (SCM). For the WtE ash to be able to be used it needs to have properties like the normally used aggregate materials or the normally used cementitious materials [5]. If used as an aggregate filler material it should have similar properties to sand or gravel. Therefore, the size and shape of the SCM is of interest. Another important factor of the aggregate material is that it is inert and does not react with water or cement in a negative way which could decrease the quality of the concrete. One thing that has been shown when using inert filler materials however is that above 20% filler the strength of the concrete gets reduced [4]. SCMs that inherit pozzolanic activity can be used at higher concentration of filler due to reactions that strengthen the concrete matrix. The aggregate material should also be non-toxic which is one of the concerns of using WtE ashes since it is known that it can contain different metals.

2.1.1 Concrete ingredients

Aggregates makes up the largest part of concrete it serves as an inert filler which both occupies volume and give the concrete stability [5]. Aggregates typically consist of sand, gravel and/or crushed stone. Properties of the aggregates will also have big impact on the properties of the concrete. Size, shape surface texture and area will impact workability, density, durability and strength among others [3]. The aggregates will act as the skeleton of the concrete matrix that bonds with the cement paste. Important factors for the aggregate material are properties like size, surface area and texture as previously mentioned but another important factor is that it is chemically inert. When investigating WtE ash as possible filler aggregate material. Another important factor is the presence of metals in the material which could cause leaching and environmental damage.

Cement is the most important component in concrete and normally consists of a mixture of different minerals with different elements. The elements are among others Si, Al, Fe, Ca, O and are mostly found in limestone, clay or shale [5]. In production of Portland cement high temperatures (1300-1450 °C) is used to transform the raw material into various minerals including Alite (Ca_3SiO_5), Belite (Ca_2SiO_4), Aluminate ($M(Al_xO_y)$), Ferrite ($M(Fe_xO_y)$) and notably a large amount

of CO_2 is created in the process. A net reaction of the production of Portland cement can be seen in Figure 1 below. The dry cement mix powder is then mixed with aggregates to create dry mix concrete powder and when water is added several chemical reactions will start. The reaction between the cement minerals such as alite and belite with water will result in the formation of calcium silicate hydrate gel which will work as a glue and hold the concrete matrix together [5]. An example of one chemical reaction that could occur can be seen in Figure 2 below where Alite reacts with water to form calcium silicate hydrate gel and slaked lime ($Ca(OH)_2$) [3].

$$Calcite + Clay\ mineral + Shale \xrightarrow{1300-1450\ °C} Alite + Belite + Aluminate + Ferrite + CO_2 \quad (1)$$

$$Alite + Water \rightarrow Calcium\ silicate\ hydrate\ gel + Slaked\ lime \quad (2)$$

Figure 2.3 *Chemical formula to produce cement and concrete [5].*

As previously mentioned, the cement is the part of the concrete that is responsible for most of the carbon emissions created in the cement industry at around 90% of the total emissions [1]. This is caused by the high temperature requirement and the formation of CO_2 in the reaction which can be seen in figure 2.3. By replacing the cement with another material that could have similar properties could lead to a large reduction in the carbon footprint of concrete productions. This is as previously mentioned why one of the goals with this thesis is to investigate if WtE ashes could be used as a replacement. One of the reasons that WtE ash is seen as a possible replacement or additive in cement is that they contain minerals with elements that are important in the creation of cement. This will be further explained in the section selection of WtE ash materials.

2.2 Pozzolanic activity

One important property investigated was the pozzolanic activity of the WtE ashes. Pozzolanic activity is the ability of a material to react with lime (calcium hydroxide) and water to form a cementitious material. This affect both the strength and durability of the concrete which makes the pozzolanic activity an interesting property to investigate to be able to evaluate if the WtE ash could be used as an SCM. Pozzolanic materials can contain chemicals such as SiO_2, Al_2O_3 and CaO [6]. These can react with lime to form calcium silicate hydrate (CaH_2O_4Si) and calcium aluminate ($CaAlO_4$) which are important components in cement. The pozzolanic activity which can give an estimation how the material will perform as cement can be measured using different methods. Chemical methods include among others the lime saturation method where the SCM is added to saturated lime solution and after the pozzolanic reaction has been going for a certain amount of time the residual amount of lime is measured [7]. Another test that can be used is the "Frattini" test in which the concentration of OH^- and Ca^+ ions is measured using titration methods [8]. Another method is to use the modified R3 method which measures the amount of bound water that is in a paste made with the SCM [9]. Out of the methods the modified R3 method

was chosen and used to evaluate the pozzolanic activity of the WtE ash concrete specimens. This is because it was deemed to be the most suitable as the other methods are more suitable for materials that exhibit high degrees of pozzolanic activity. The modified R3 method measuring bound water is more suited to be used for the WtE ash materials that will probably have relatively low degrees of pozzolanic activity because of the lower concentrations of SiO_2 and Al_2O_3 [10]. The other methods will probably either not work or require much longer time when dealing with the ash materials that are expected to have a low degree of pozzolanic activity. The purpose of the method is to investigate how much chemically bound water there is in the paste. Earlier research has shown that the amount of chemically bound water is directly proportionate to the cement hydration process [10].

The "Frattini" test based on calculating the concentrations of OH^- and CaO when mixing the sample with water. By titration these concentrations can be calculated and be compared to references to see if the sample shows any pozzolanic activity [8]. The Frattini test is not suitable for materials which may only contain only a small amount of pozzolanic activity. This test was therefore not used in the project since the ash samples was expected to have low activity.

2.3 WtE incineration process

An overview of the WtE incineration plant is given in Figure 2.1 [11]. The waste is collected and put into a bunker and fed to ovens that incinerate the waste at the lowest 1000˚C under constant supply of air. The heat from the ovens can then be used to heat water and drive turbines to make electricity and be used for district heating. Using an electrostatic filter, 99.5 % of the ash from the flue gas can be separated and sent to washing and zinc recovery [11]. During zinc recovery the ash is washed with acid from the flue gas cleaning to recover the metal and after this the ash is washed with water, Figure 2.2 [11]. The residual ash was tested in this project. The other ash type used in this project is slag that is collected from the bottom of the furnace. The difference between these two is that the slag is more reactive as it has had less contact with air and water and metals pieces are still present. The slag sample is collected directly after the water lock, while the MIBA sample is slag that has been metal sorted and put into piles to dry for around six months [4]. The different ash samples are further described in 3.1.

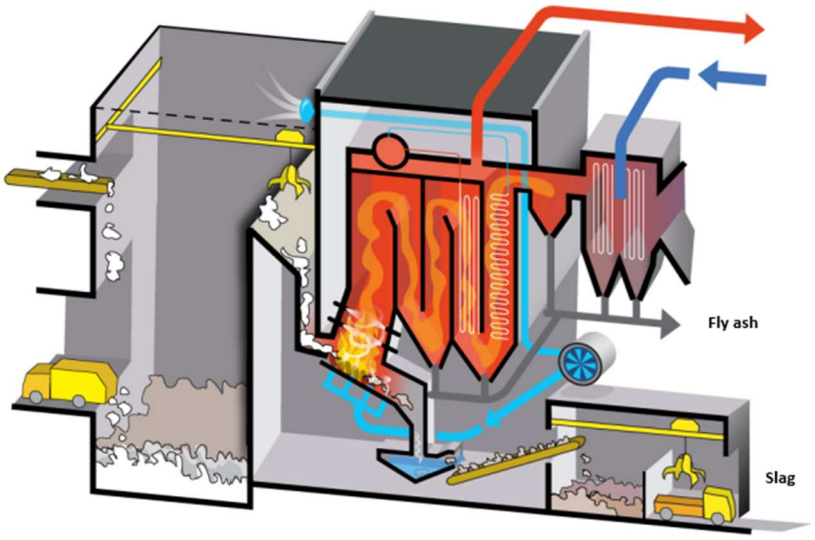

Figure 2.1 Overview of the WtE incineration process [11].

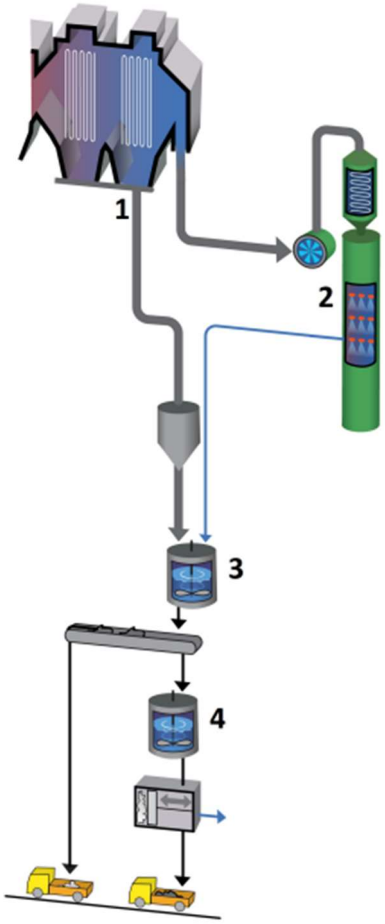

Figure 2.2 Overview of the zinc recovery process in the WtE incineration processes were 1 is the electrostatic filter and 2 is a silo where the ash is collected. 3 is where the fly ash is washed with HCl and 4 is where the zinc is recovered using NaOH [11].

3 Materials and Methods

3.1 Characterisation of ash

All the ash materials used in this project was collected from a WtE incineration plant in the southwest of Sweden. In table 3.1 indicative elemental contents of each ash material is listed.

3.1.1 Slag

Slag or IBA short for incinerator bottom ash stands for about 80 w% of the residues from the incineration of MSW [13]. It is a heterogenous material with a large range of different particle sizes. It has been shown that the composition of the ash varies with the particle size and that smaller particles has more portlandite ($CaCO_3$) but also more metals than larger particulates [13]. The ash can contain minerals, metals, glass and metals among other materials. In the end the composition of the ash material depends on the MSW raw material and the incineration process conditions.

3.1.2 MIBA

MIBA short for mineral fraction from incinerator bottom ash is the same material as the slag but is further treated [14]. Parts bigger than 40 mm are removed and metallic pieces are removed. Another big difference to the slag is that the material is left to stabilize outside for about 6 months. During this time the material will be exposed to gases like CO_2 and O_2 and to water from rain. These chemicals react with the ash and the material become less reactive [4].

3.1.3 Fly ash

Fly ash consists of the solid materials that are collected from the cleaning of the combustion gases. The main components of fly ash are alumina, silica and iron oxides, but it also contains metals [12]. The specific ash that is used in this project has been collected after the electrostatic filter and washed with acid and water as discussed in 2.3 [11]. The material is homogenous and unlike the slag and MIBA does not have any larger pieces in it.

Table 3.1 Total expected elemental content of the three WtE ash samples in mg/kg dry mass [4].

Element	Fly Ash (mg/kg DS)	Slag (mg/kg DS)	MIBA (mg/kg DS)
Al	38 000	26 450	26 450

As	350	33	33
B	190	-	-
Ba	2 000	2 250	2 250
Be	0.66	-	-
Ca	190 000	115 000	115 000
Cd	35	2.3	2.3
Co	38	92	92
Cr	690	450	450
Cu	1 800	3 300	3 300
Fe	25 000	61 500	61 500
Hg	2.9	<0.05	<0.05
K	7 900	14 000	14 000
Mg	15 000	13 000	13 000
Mn	1 200	1 100	1 100
Mo	170	23	23
Na	9 600	29 000	29 000
Ni	250	195	195
P	7 200	5 350	5 350
Pb	6 300	820	820
S	68 000	7 150	7 150
Sb	3 500	78	78
Se	-	10	10
Si	95 000	-	-
Sr	-	465	465
Ti	15 000	8 350	8 350
V	77	57	57
Zn	22 000	5 100	5 100

3.2 Raw material preparation

Before evaluating the ashes as SCMs in the experiments some pre-treatment steps were carried out including drying and milling the SCMs. Around 300 g of each ash samples were weighed and dried in an oven at 105 °C for 24h to remove water. After 24h the beakers were again weighed and the SCM was mixed around using a glass rod. After another 24h the beakers were again weighed, and it could be concluded that the weight had stabilized and that the samples where dry. The difference in weight between weighing at 24h and at 48h drying was less than 0.5%. When more material was needed it was decided that drying for 24h would be sufficient to dry the material based on this.

The particle size distribution of the SCMs was not homogenous and could have a large difference in particle sizes. A particle size distribution of the original ash samples was also calculated using sieves of different sizes. The three different ash samples where sieved using two different sieve sizes. These where sieves with holes of 1 and 2mm. This gave three different fractions one with particles that where bigger than 2mm the second being particles that where between 2

and 1mm and a third fraction that was particles smaller than 1mm. The different fractions where weighed and the weight percentage of each fraction where calculated. This was done twice to get an average of the particle size distribution and how much of the material that could be used for further tests. This was the case because the milling machine used a Retsch PM 100 could not handle particle sizes bigger then 1mm.

To get a decent comparison to cement it was decided that the ash samples that were used should be sieved and milled into a fine powder. After the samples had been sieved the fractions containing particle sizes bigger then 1mm was discarded. The remaining material was milled in a Retsch PM 100 rotating ball mill. About 20g of MSW ash sample were put in a stainless-steel container along with 31 stainless-steel balls and was tightly secured into the milling machine. The counterweight was set to match the total weight of the sample holder and the contents inside. After this the milling machine was run for 20 minutes at 500 rpm and switching rotating direction every 5 minutes. This was done a total of 8 times for each ash sample to attain enough material to be used in the R3 test, for casting concrete cubes and the PXRD test.

3.3 Modified R3 method

The modified R3 method measures the pozzolanic activity of a material. This is done by measuring either the amount of cumulative heat through thermogravimetric methods or the amount of bound water content in pastes made from SCMs [15]. In the case of WtE ashes that contain substantial amounts of aluminium the bound water content method should be the most suitable. This because aluminium can react with pozzolanic materials and form heat. There will therefore be difficult to distinguish between heat coming from the reactions with aluminium and the heat from other reactions of constituents of the SCM. This method is also better suited to measure small degrees of pozzolanic activity. The method used follows method B in the ASTM C1897-20 standard and is carried out by mixing the SCM ash into a paste. The paste contains porlandite ($Ca(OH)_2$), WtE ash and 0.5 M potassium hydroxide (KOH) solution. After the paste has been mixed it is cured for around 24 h before the bound water measurement is carried out [15]. To be able to calculate the bound water content the paste is first dried at a temperature of 110 °C for 24 h to remove all the absorbed water that is not chemically bound in the paste. After this the paste is heated at 400 °C in a furnace and the weight before and after heating is recorded [15]. Using the formula below the amount of bound water can be calculated as grams per 100 grams of dry material.

$$H_2O_{bound,dried}\left(\frac{g}{100\ g\ of\ dried\ paste}\right) = \frac{w_0 - w_h}{w_0 - w_c}\ x\ 100 \quad (4)$$

Figure 2.4 Formula for calculating bound water where w_0 is the weight of the dried paste and crucible before entering the furnace, w_h is the weight after the furnace and w_c is the weight of the aluminium container [16].

When enough sample material had been milled the modified R3 method was used to check if the sample ashes had any degree of pozzolanic activity at all and if it was any idea to carry on with casting concrete cubes with them as a cement substitute. The test was carried out by mixing a paste containing 10g of SCM, 30g $Ca(OH)_2$ and 36ml of 0.5M KOH solution using a hand held stand mixer. This was done for each of the three different ash samples. After mixing the paste as much material as possible was transferred to a plastic tube and was left to cure in an oven set to 50 °C for 24 h. The samples were removed from the oven and transferred to pre-weighed aluminium crucibles and was then again put in an oven this time at 110 °C for at least 24 h but in this case they were left over the weekend for around 48 h. When the water that is not chemically bound had been dried from the samples they were weighed and then directly put into a furnace at 400 °C for 2 h. After 2h the samples were again weighed. The weight of the aluminium crucible, the samples weight before and after the furnace heating were later used to calculate the amount of bound water in each of the WtE ash samples.

3.4 Test on mechanical properties

3.4.1 Production of test cubes

To be able to perform strength tests concrete cubes with various amounts of SCM substituent were casted following the SS-EN 196 standard. Three cubes were created for each different recipe that was used. This was to enable strength testing with 7, 28 and 56 days of curing time. It was decided to only do one test for each since it would have taken too much time to make more cubes mainly due to the long time it took to mill the SCM. In total 27 cubes were made with 9 different recipes. For the three sample ashes two different recipes were used on with 15% substitution and one with 30% substitution. This was also done using slaked lime and lastly one recipe used 100% bascement for reference. These cubes were made as references to be able to compare with the cubes with SCM in them. The SCM containing cubes can be compared with the 100 % bascement but also with cubes containing equal amounts of inert material. This makes it possible to see if the SCM has had any effect on the strength of the concrete.

Table 3.1 Total amount of different cubes made with different degrees of substitution.

SCM	Degree of substitution
Fly ash	15 %
Fly ash	30 %
Slag	15 %
Slag	30 %
MIBA	15 %
MIBA	30 %
Limestone	15 %

Limestone	30 %
Reference	0 % (100 % bascement)

The recipe used sand which was sieved so that the particle size was smaller than 1mm, deionized water, bascement and SCM. Bascement was chosen as it is a commonly used type of cement. This type of cement already contains 15 % coal fly ash. The exact recipe used can be seen in the table below.

Table 3.2 Recipes used when casting cubes with 15 and 30 % substitution.

Ingredient	**15 % SCM**	**30 % SCM**
SCM	33.75 g	67.5 g
Bascement	191.25 g	157.5 g
Sand	675 g	675 g
Water	112.5 g	112.5 g

The molds used for casting the cubes had the dimensions 40x40x40 mm. These were prepared by oiling the sides of the mold to later make it easier to demold the cubes. Mixing the concrete followed a pre-determined procedure that was the same for each mixture prepared. Mixing was carried out using a gyratory stand mixer at two different speeds. Low speed had 125 ± 5 rot/min and a high speed 285 rot/min. Water, cement and SCM was all added to the mixing bowl and within 10 sec of adding the water the mixer was started at low speed for 30 sec. During the next 30 sec the sand was added continuously until all sand was added. After that the mixer was switched to high speed for 30 sec. The gyratory mixer was then switched off and mix that had stuck to the side of the bowl was scraped down using a spatula. The gyratory mixer was turned off for a total of 90 sec before it was again turned on for another 60 sec at the high speed. At this point the mortar was put into the molds with the help of a trowel [23]. The mortar was added in two layers and d jolting table was used to pack the mortar and remove bubbles. Using the trowel excessive concrete was removed by scraping it from the top of the mold. This process was repeated for each of the nine different mortar mixture that was prepared. After approximately 24 h the cubes where all removed from the molds and was left to cure in de-ionized water until testing their strength. The samples are cured in water for standardisation giving constant hydration and temperature.

3.4.2 Compressive strength test

As previously mentioned, the strength of the concrete cubes was tested after 7, 28 and 56 days of curing time. When 7 days of curing time had passed one concrete cube of each different mixture were picked out if the water storage container. Using paper towels the cubes where dried before continuing with the test. The cubes were put into a mount and the area that was not covered by the walls of the mold was avoided to be put against the metal plates in the mount. The sample was compressed in a Matest compression machine until mechanical failure. At this point the maximum load and maximum strength was noted down. The cracked sample was removed and put in a zip-lock plastic bag intended to

later be used in leaching tests. This process was used repeated on the following cubes tested after 7 days as well as the cubes tested at the two later curing times.

3.5 Leaching tests

There are different ways to carry out leaching test for concrete materials and they can be performed at different phases of the concrete [18]. Since the aim of this thesis was to investigate if WtE ash could be used as filler or cement replacement in concrete. A monolithic leaching test method was chosen. Which means that the leaching of elements is tested on concrete specimens that has been casted with a certain amount of WtE ash additive [19]. The reason for testing on casted concrete and not using other methods that test crushed concrete or dry cement mixture is that the intended use of the concrete is in the form of casted concrete in for example buildings. It is therefore more interesting to evaluate how much elements that leach out in casted concrete since this is how it will be used.

A one stage batch test could have been performed following the standard EA NEN 7375. The leaching agent (milliQ-water) is put with the sample in a container and samples are collected after certain times have passed. The eluate is filtered using syringe filters and with the help of different methods including Inductively coupled plasma-atomic emission spectroscopy (ICP-MS) to get metal concentration.

The tank leaching test use a known eluent volume of milliQ-water put monolithic samples in containers for a certain amount of time. Samples are extracted using and filtered to eliminate particles in the eluent. The samples are later analyzed using ICP-MS [18]. This was the leaching test that was chosen to perform in the project.

As mentioned in the introduction there was a choice made to carry out leaching tests on monolithic material which in this case was the molded 40x40x40 mm cubes that had been saved after the strength compression test. This was done since the interest in this thesis is to investigate leaching from monolithic samples [19]. However, the samples are not truly monolithic since they have been cracked in the compression strength tests. Theses cubes had to be used due to time constraints. The test follows a modified version of the standard EA NEN 7375 which is also called the tank test. In summary the test is carried out by putting the cracked molded cubes into containers with an eluate and taking out samples after set number of times [20]. These would later be analyzed using ICP-MS.

First the volume of the samples was calculated by calculating the volume of a cube with a side length of 40 mm which is equal to 64 cm^3. One problem however was that the samples had been slightly cracked during the compressive strength test, but it was eventually decided that the same volume calculations would be used for all cubes. This was because it would be too time consuming to prepare more WtE ash material and make new cubes. The small differences in volumes

were also deemed to be acceptable since the aim of this experiment was to do initial testing to see if there is any leaching from the molded samples. For further calculations the sample volume of 64 cm^3 was used. The eluate volume is according to the standard supposed to be between two to five times larger than the sample volume. In the end an eluate volume of the maximum 320 ml was chosen due to the size of the containers used and that the sample needed to be fully submerged in the eluate. The containers used was white polypropylene buckets with corresponding lids. In total 14 buckets were prepared with cubes from the first (7 days) and second (28 days) strength test.

The experiment was started by measuring and adding 320 ml of milliQ water to each of the 14 containers which had previously been rinsed. The concrete sample cubes where all placed into the corresponding labelled container and the lid of the container was closed. When samples were taken from the containers the eluate was filtered to ensure that small dust particles would not be present in the sample but only the leached chemicals. This was done using syringe filters with a size of 0.45 µm. With the syringes 10 ml of each sample was removed, filtered and stored in labelled containers. The samples were also acidified by adding 100 µl of 65% nitric acid to preserve the samples in storing. Every time 10 ml of sample were removed from the container 10 ml of milliQ water was added back. This was to ensure that the entire sample was always submerged in eluate. This would also maximize the leaching process when the eluate was diluted as

3.6 Analytical methods

3.6.1 Powder X-ray diffraction

PXRD or powder X-ray diffraction is a technique that uses X-rays that are shot at in this case the ash sample to find crystalline phases in the sample. This gives a diffractogram which is plotted using the angle theta and the intensity. By using a known database, the peaks in the diffractogram can be characterized and different phases can be found [17]. In this project PXRD was used on the three ash samples and on the corresponding cured paste. This made it possible to find out information about the mineralogy of the samples and if phases important to cement based materials had been formed in the pastes.. The ash raw material could be directly out into the circular molds that would be put in a tray and later into the XRD-machine but the R3 paste required more preparation. They had to be ground up using a mortar and pestle before filling up the tray molds. The trays were put into the XRD machine and ran.

The acquire diffractograms were later analyzed using the program DIFFRAC.EVA where the search by name function was used to search for phases that was expected to be found in in each sample [25,26]. Theses where phases that had been found in previous research on the same type or similar type materials [24]. The peaks where matched and the raw material was compared to the R3 test that was made with the same raw material to see if there had been any changes.

opposed to being concentrated if no new water was added. This dilution could also later be corrected with calculations after the ICP-MS results are acquired.

3.6.2 ICP-MS

Inductively coupled plasma – mass spectrometry or ICP-MS is a detection technique which uses plasma to ionize samples into charged ions [21]. The samples are injected into an oven and ionized at high temperature. These ions can be separated by their mass to charge ration or m/z using an electric field called a quadrupole. The setup scans for a known weight letting only ions with this weight pass to the detector while the other weights are lead away in the quadrupole. Using a detector the ions are detected and counted meaning that the concentration of elements in the original sample can be calculated. In this project this technique was used to analyse the eluate from the leaching test to see which metals are present and how much of them there are [22].

The samples collected from the tank leaching test were diluted 45 times with milliQ-water and acidified with nitric acid. In total 84 samples were collected and tested six for each different cube. The samples were collected after 1, 3, 10, 18, 25 and 32 days. When the all samples had been diluted they where put in to tray and loaded into the ICP-MS setup for analysis.

4 Results and Discussion

4.1 Mechanical properties

4.1.1 Size distribution of ash raw material

In the figure 4.2 below the distribution of particle of the slag and MIBA samples can be seen. As mentioned in the method section particle sizes larger than 1 mm could not be used for the project due to the limitations of the rotating ball-mill. This means that a large part of the materials from the slag and MIBA samples had to be discarded before milling. For slag on average between the two runs 73 % of the material was discarded. For MIBA this number was similar at around 68 %. In a full scale process it might be possible to mill more of the material with stronger milling equipment. For the fly ash sample 100 % of the material was smaller than 1 mm which meant that all material could be milled. The removal of particle size larger than 1 mm from the slag and MIBA samples. The amount of material that was discarded was larger than expected and is also the reason why there had to be a second batch of material prepared because there was not enough material to be used in the experiments from the first run. The material from both slag 1 and slag 2 where later mixed before it was used in any experiments. The same was done with MIBA 1 and 2. As mentioned earlier in the materials section it has been shown that the smaller particles in the slag ash can contain a larger concentration portlandite but also a higher metal concentration [13]. As there is a possibility that the there is a difference in composition between the larger and smaller particles in the ash samples. Meaning that the results might have been different if the larger particles could also have been milled and used in the experiments. This mean that by removing the larger particles the composition of the ash was probably affected. It could be that the strength of the concrete was benefited by this but the increase in metal concentration could also increase leaching.

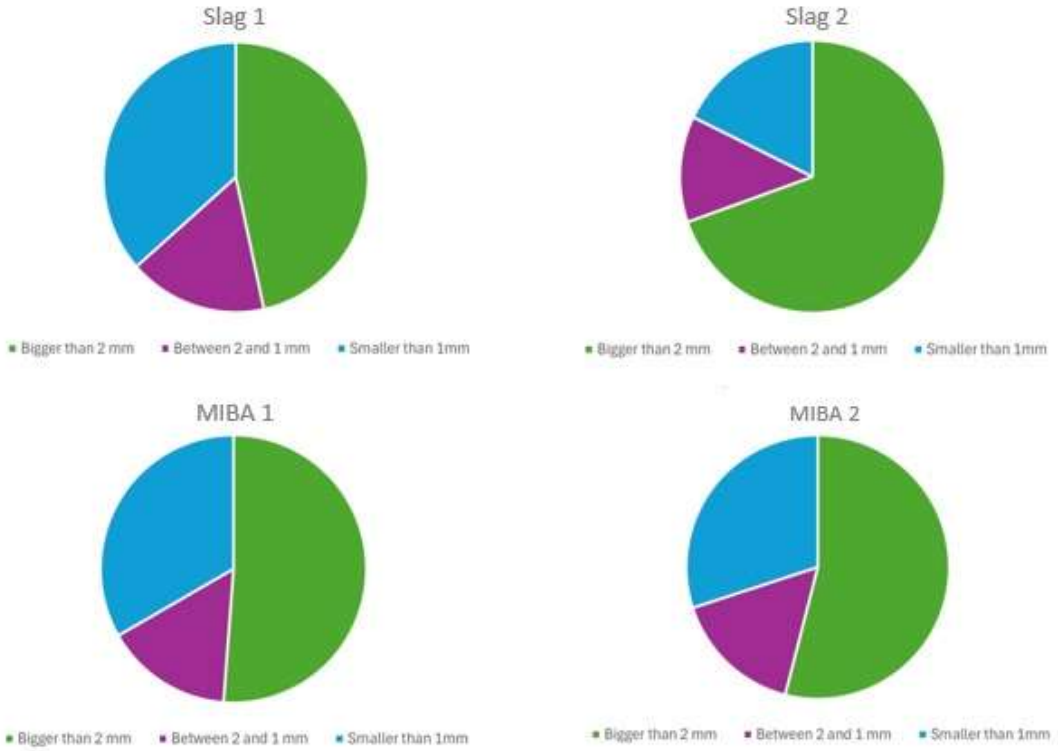

Figure 4.2 Graphs showing the size distribution of particles in the slag and MIBA samples.

4.1.2 Compressive strength tests

In the figure 4.3 below all the tested cubes compressive strength is plotted. Here it can be seen that the reference sample had the highest compressive strength. This is to be expected as this sample contained 100 % Portland cement. In the following figure 4.4 and 4.5 the samples are separated into degrees of substitution for increased readability. Interestingly the fly ash at 15 % substitution displays the highest compressive strength of the SCMs but at 30 % substitution it has the lowest. This suggests that the degree of substitution of fly ash has a large impact on the mechanical properties of the casted cubes. At 15 % substitution both the fly ash and the MIBA display compressive strengths larger than the limestone reference suggesting that the SCMs has had a positive effect on the strength of the concrete. The slag however performed at similar level to the "inert" limestone reference and seems to not have an effect on the compressive strength of the cubes. At 30 % substitution the MIBA sample had the highest compressive strength. Both slag and fly ash got results like the strength of the limestone reference.

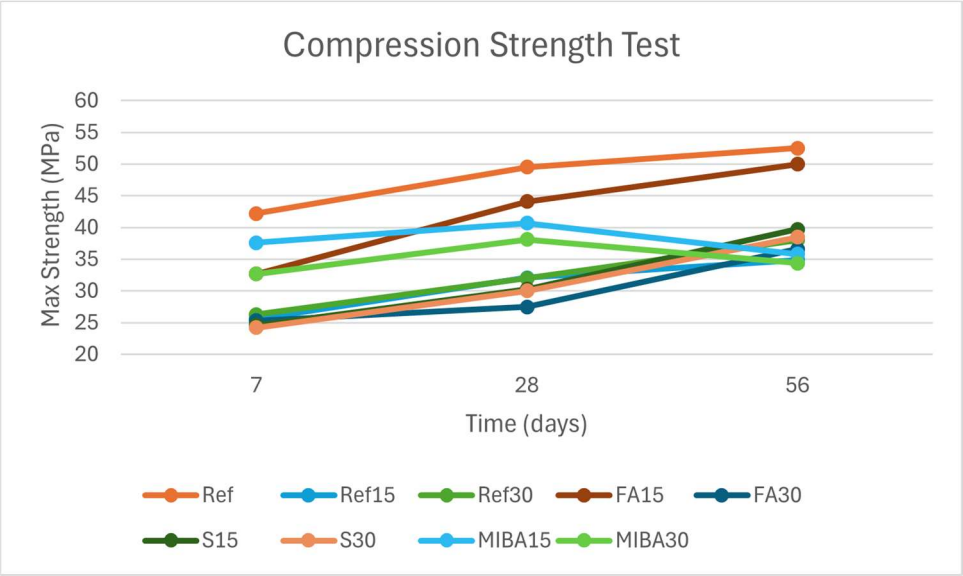

Figure 4.3 *Results from compressive strength tests carried out at 7, 28 and 56 days measured in MPa.*

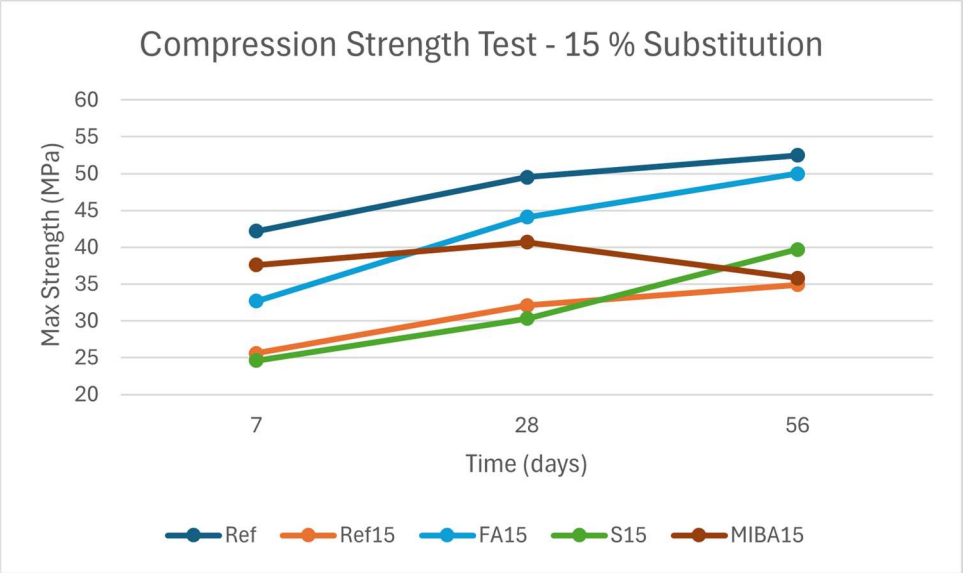

Figure 4.4 *Compressive strength test results from samples containing 15 % substitution of SCM.*

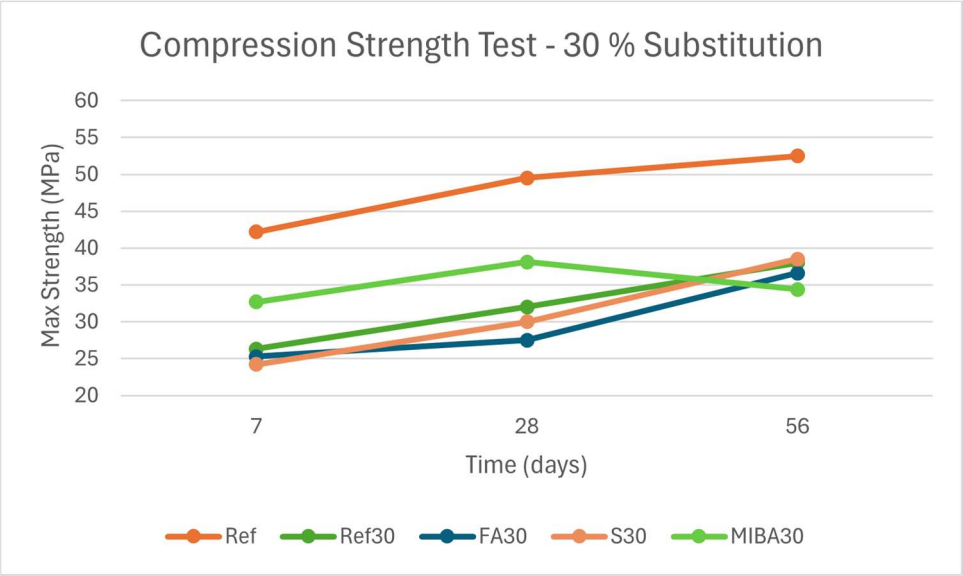

Figure 4.5 Compressive strength test results from samples containing 30 % substitution of SCM.

4.2 Chemical analysis

4.2.1 Pozzolanic activity

By measuring the bound water content using the modified R3 method an estimation of the three different ash samples reactivity could be done. As can be seen in figure 4.1 none of the three ash sample reach the threshold of 3.6 g/g dried material have a high chance to be classified as a reactive SCM [16]. There are however false negatives. Meaning that the materials could still benefit the mechanical strengths of concrete even if they are under the threshold [16]. Out of the three materials the MIBA sample had the most bound water in the modified R3 test followed by fly ash and lastly the slag. These results also correlate with the results from the compression strength test presented in section 4.2.2 where the most pozzolanic material MIBA generally had higher compressive strength. Slag which had the lowest pozzolanic activity seems to not improve the strength of the concrete.

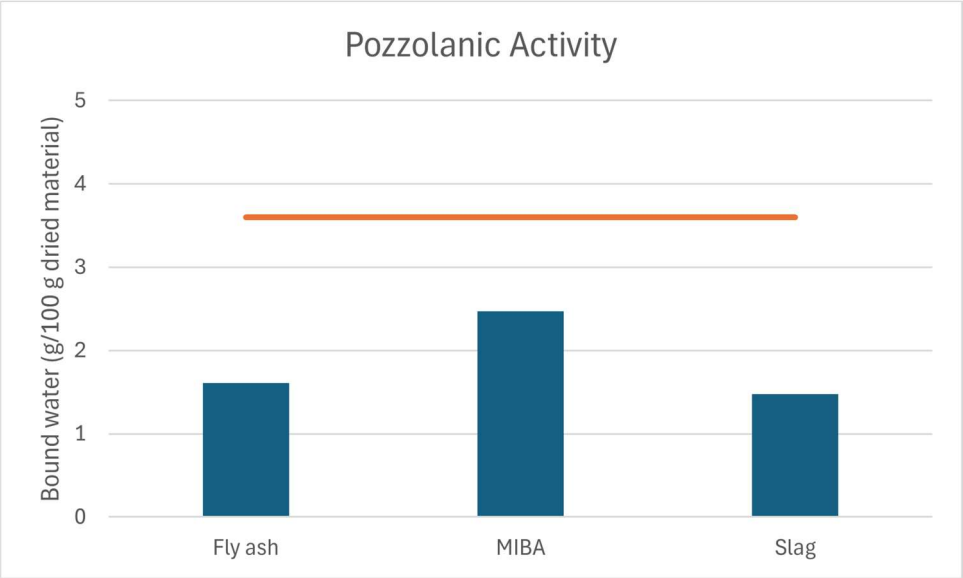

Figure 4.1 *Bound water in g/g dried material content measured in the modified R3 test. With orange line showing the threshold of where an SCM can to a 66 % certainty be considered reactive [16].*

4.2.2 Mineralogy

In the following section the diffractograms from the PXRD runs will be presented with common names in the figures 4.6-4.11 the list below details the chemical formulas of all minerals that were found.

- Quartz – SiO_2
- Anhydrite – $CaSO_4$
- Calcite – $CaCO_3$
- Ettringite – $Ca_6Al_2(SO_4)_3(OH)_{12}\cdot 26H_2O$
- Portlandite – $Ca(OH)_2$
- Sylvite – KCl
- Halite – NaCl
- AFm ("alumina, ferric oxide, monosubstituted" phase) $3CaO\cdot(Al,Fe)_2O_3\cdot CaX_y\cdot nH_2O$
- Anorthite – $CaAl_2Si_2O_8$

By comparing figure 4.6 and 4.7 it can be determined that there are differences in several peaks from the fly ash raw material and the fly ash paste that was used in the modified R3 method. This means that chemical reactions has occurred. Ettringite which is a hydrated phase that form in the early stages of concrete hydration is found in the R3 sample. Also, sylvite and portlandite is found in the R3 sample but not in the raw material. The presence of portlandite and ettringite indicates that hydration products has formed. The presence of sylvite indicates that there is chlorine (Cl) in the fly ash raw material which has most probably reacted with sodium (K) from the potassium hydroxide solution used in the mixing of the paste. Quartz (SiO_2)was found in the raw material but not in the R3

paste this is either because the quartz has reacted or because it was finely ground and was dissolved in the hydration process and become less identifiable.

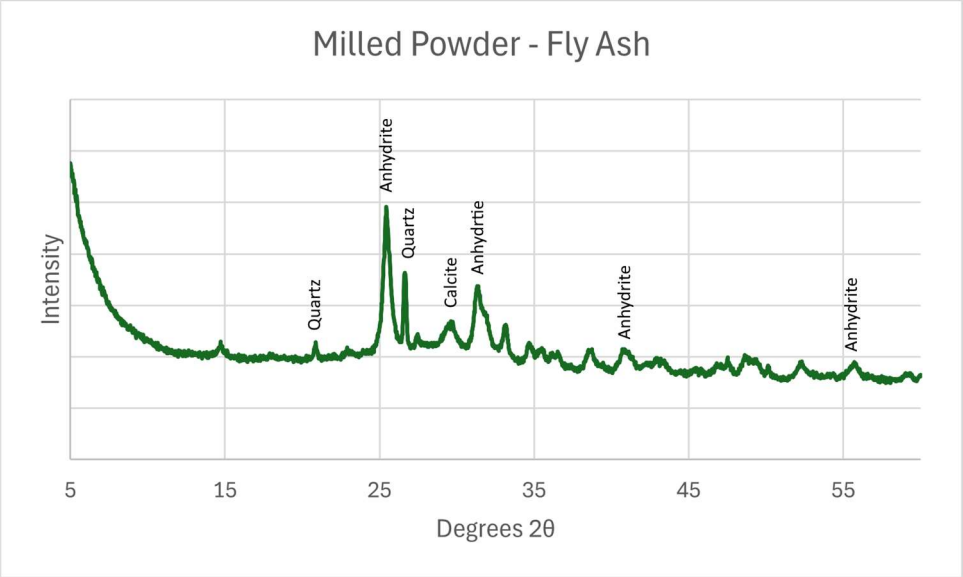

Figure 4.6 PXRD diffractogram from raw material milled powder fly ash sample labelled with minerals.

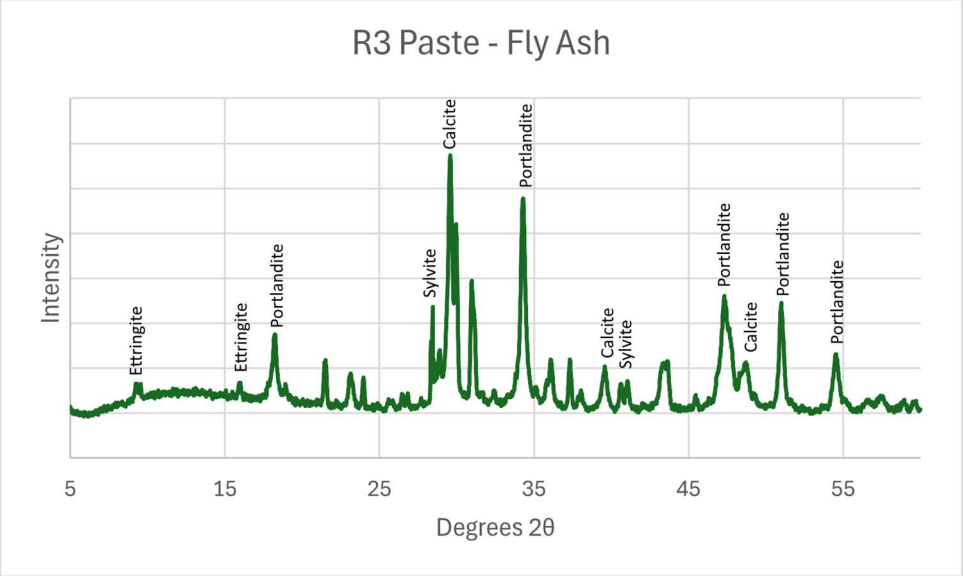

Figure 4.7 PXRD diffractogram from R3 paste fly ash sample labelled with minerals.

The slag samples in figure 4.8 and the slag R3 paste in figure 4.9 display similar minerals as the fly ash samples. But here there is no ettringite formed but instead an unidentified AFm hydration phase. Portlandite is found in the R3 paste and calcite. The halite and anhydrite are not present in the R3 paste and

has most likely reacted into different phases. Here the quartz is present in both the raw material and the paste.

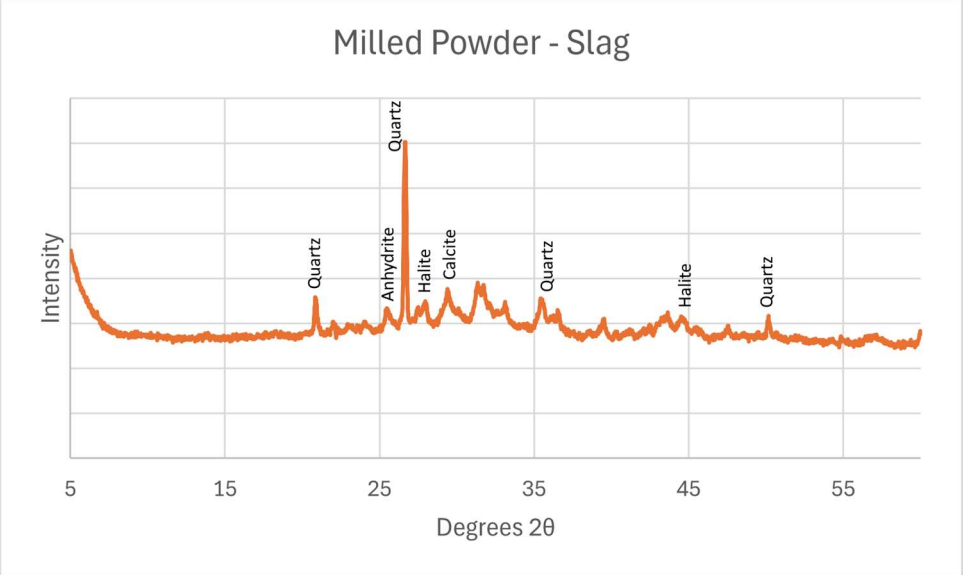

Figure 4.8 PXRD diffractogram from raw material milled powder slag sample labelled with minerals.

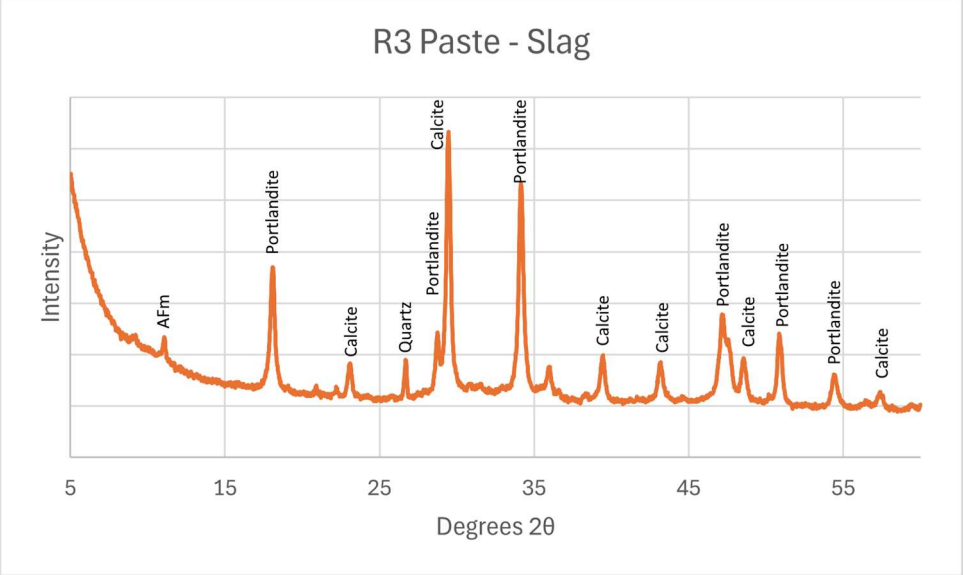

Figure 4.9 PXRD diffractogram from R3 paste slag sample labelled with minerals.

MIBA raw material from the figure 4.10 diffractogram had quartz, anorthite and calcite phases. After the R3 paste the material had AFm hydration phases but unlike the other samples it had much less portlandite. Instead, it had a large calcite peak. This is because the portlandite in the paste carbonated and reacted with CO_2 in the air to form calcite. This is a process that normally occur on the surface of concrete [3].

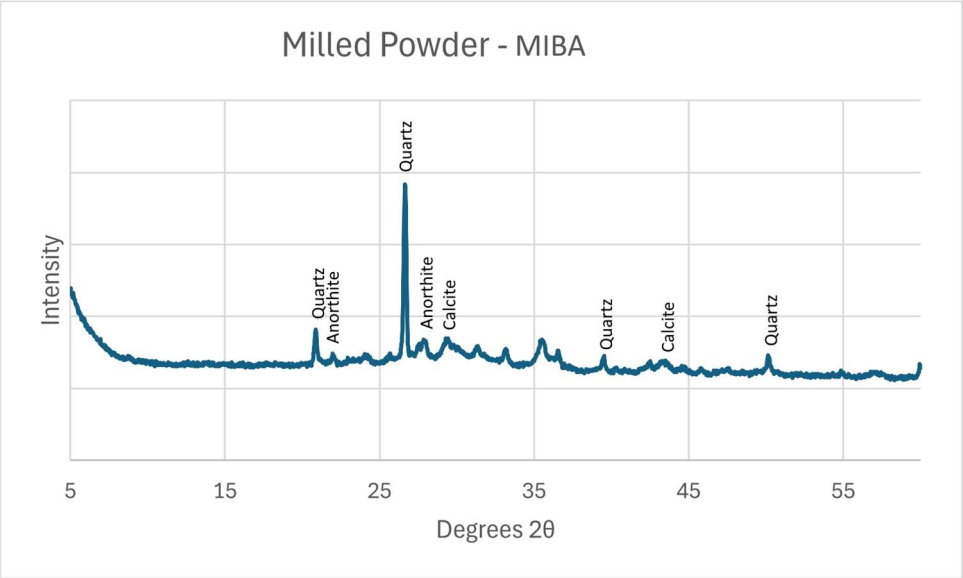

Figure 4.10 PXRD diffractogram from raw material milled powder MIBA sample labelled with minerals.

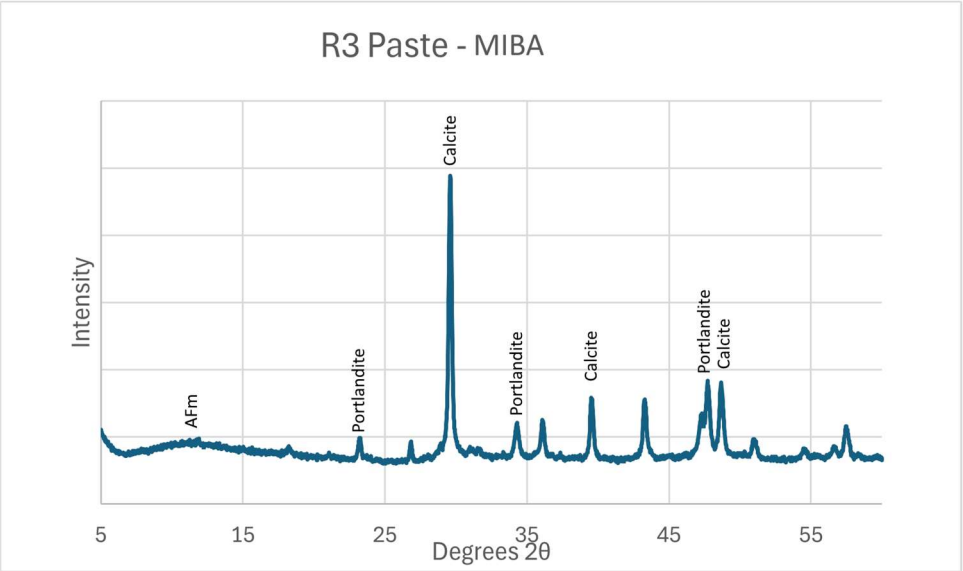

Figure 4.11 PXRD diffractogram from R3 paste MIBA sample labelled with minerals.

4.3 Leaching from monolithic concrete samples

Metal release from the monolithic concrete samples measured with ICP-MS were initially compared with thresholds set by the Swedish institute "Naturvårdsverket" [19]. If the leaching of metals is under the threshold there is less, then a minor risk of environmental impact. Another important clarification

is that these values are provided for leaching from ground up samples and not monolithic samples. As mentioned in earlier in the report there are no set values for monolithic concrete. It was in the end decided that these values could not be used as comparisons since the method and form of the concrete is highly different to what samples were used in the leaching tests. Instead values from a paper by Snellings et. Al. was used [27]. A table of the collected threshold values can be found in the paper were values from four different places that have regulations on leching from monolithic concrete materials are compiled. It is not stated in the report what method they have used to get these values which means that comparing them with the results from this thesis is still hard. But due to the lack of knowledge and regulations this is the best way to get a grip on how severe the leaching of metals could be from a monolithic material made from WtE ash.

From the ICP-MS measurement that was carried out there was values for a total of ten different metals acquired. In the beginning more was supposed to be present but due to interference in one of the modes that the instrument was ran in some metals could not be measured. Out of the ten measured five are deemed interesting due to appearing as possibly environmentally damaging in "naturvårdsverkets" datasheet [27]. These were lead (Pb), cadmium (Cd), chromium (Cr), Copper (Cu) and Zinc (Zn). Note that there was no leached value recorded that was higher than the threshold values in Snellings report [27]. This can be seen in the figures 4.12-4.15 were the values from the ICP-MS measurement is plotted against the time the sample was collected. This give an indication that the leaching of metals from concrete which would be made with WtE ash might be at an acceptable level.

As previously mentioned, the samples are not truly monolithic but have cracks which will make the samples leach at a higher rate than if there were not cracks in the samples. Because the samples cracked in different ways the different cubes can be cracked in different ways meaning that comparing between the cubes is hard, but it is more interesting to see if there are any general trends. Another possible point of error is that the samples have been diluted several times due to the ICP-MS assembly requirements of not having to high concentrations of ions. Since the sample eluate was diluted to be 45 times as concentrated small differences in volume caused by measuring errors could have a large impact on the final calculated value.

4.3.1 Leaching of lead

The leaching of lead of the different samples and sample times can be seen in figure 4.12. The highest recorded value was measured for the fly ash sample with 30 % substitution and 7 days curing time. This value was close to 0.4 mg/kg concrete which is below the thresholds of all different regions in Snellings report which are between 0.6-8.3 mg/kg depending in place of origin. [27]. The fly ash samples generally had higher amounts of leaching than the other two ash fractions which can be seen in table 3.1. This is most probably because the fly ash raw material has a higher concentration of lead than the other two ash fractions.

One of the four fly ash samples also had significantly higher leaching than the other fly ash cubes. This is thought to be because of the samples has cracked differently in the compression strength test. The reference samples showed no leaching of lead suggesting that all the occurred leaching of lead comes from the addition of WtE ash.

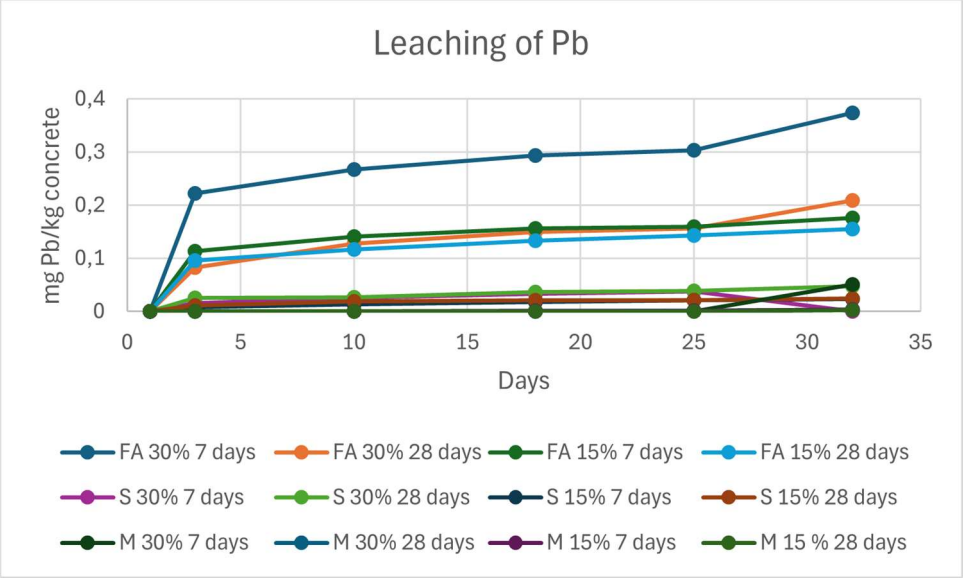

Figure 4.12 Graph of leached amount of lead from monolithic cubes from 1 – 32 days leaching time. The references are absent because they did not contain any lead.

4.3.2 Leaching of chromium

Looking at the leaching of chromium in figure 4.13 the highest value was acquired on a MIBA sample after 32 days leaching time. This value is around 0.05 mg/kg concrete which is well below all the values from the four different regions in Snellings report [27]. The values are found in a range between 0.5-7 mg/kg depending on which place the regulation is from. The reference samples for also had some leaching of chromium suggesting that not all the leaching comes from the addition of ash.

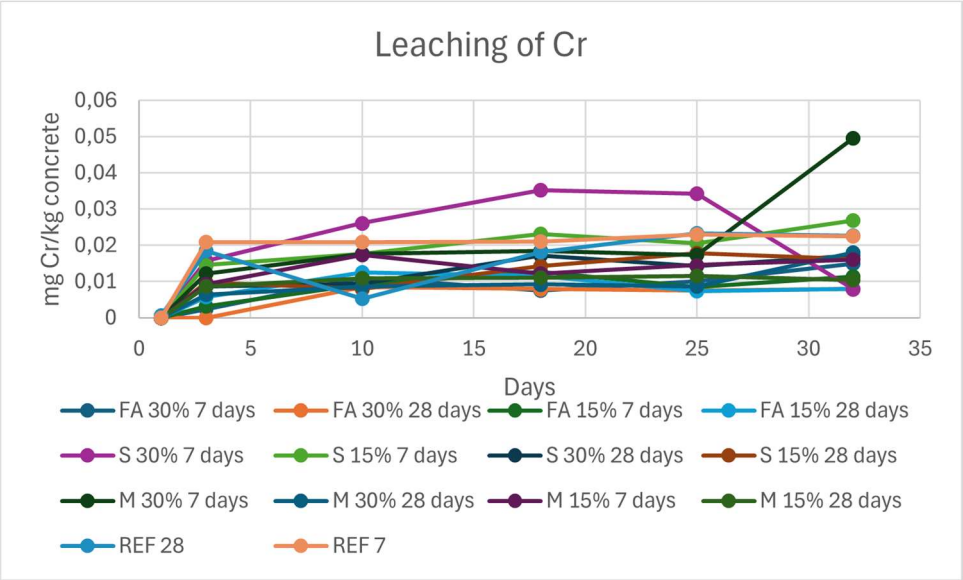

Figure 4.13 Graph of leached amount of chromium from monolithic cubes from 1 – 32 days leaching time.

4.3.3 Leaching of copper

The leaching of copper was below the limit of detection and all the samples investigated had values of 0 mg/kg concrete except one sample. This sample had copper in it, but this is thought to be due to either contamination or something that went wrong with measuring this sample. There was 9.1 mg/kg concrete in that sample which is strange since the other samples did not contain any copper at al. The following samples collected from the same container also did not contain any trace of copper, so it is thought to be something wrong with the value from this sample.

4.3.4 Leaching of zinc

The leaching of zinc from the cube samples gave values between 0.2 and 1 mg/kg concrete. This is below the values from Snellings report which are between 2.8-14 mg/kg concrete [27]. All the ash fractions as well as the references seem to exhibit similar values of leaching with some outlying values probably caused by dilution errors.

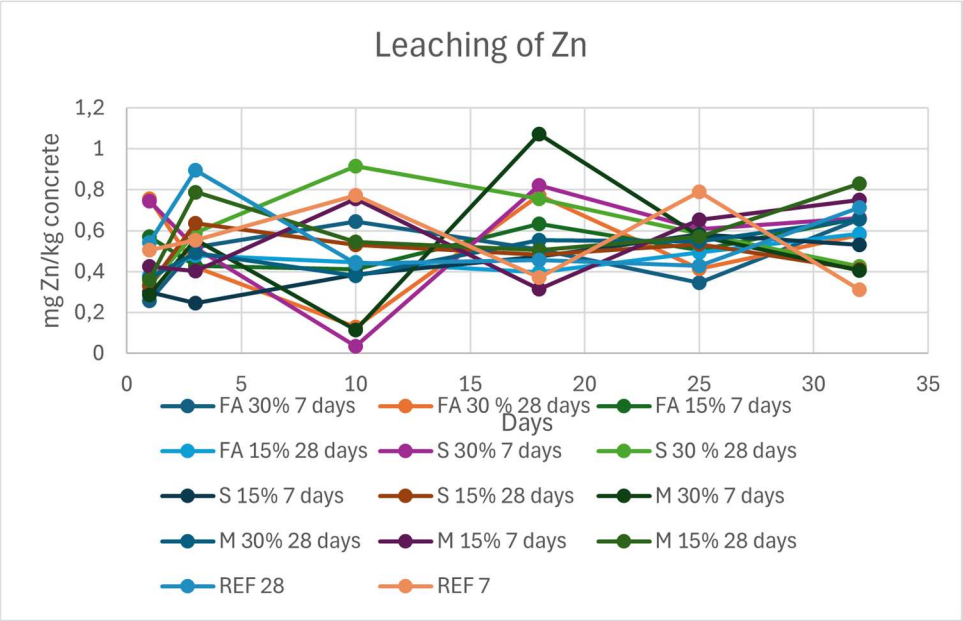

Figure 4.14 Graph of leached amount of zinc from monolithic cubes from 1 – 32 days leaching time.

4.3.5 Leaching of cadmium

The leaching of cadmium is below 0.0005 mg/kg concrete for all samples which is much lower than the thresholds in Snellings report which are at a range between 0.03-0.06 mg/kg concrete [27]. Cadmium seems to leach in small amounts and does not seem to be a problem when using WtE ash as a cement replacement material.

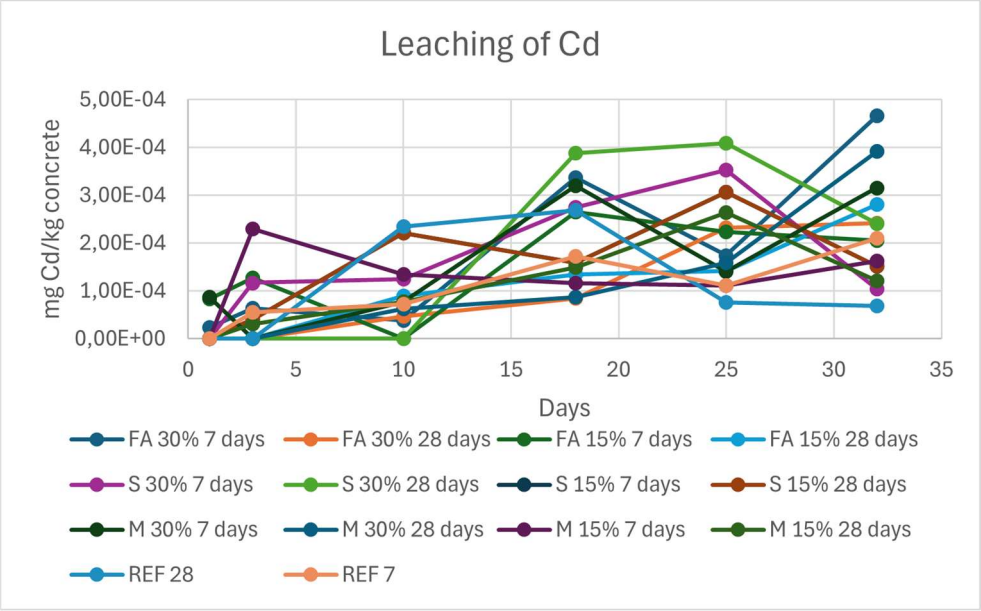

Figure 4.15 Graph of leached amount of cadmium from monolithic cubes from 1 – 32 days leaching time.

5 Conclusion

The tree ash fractions are reactive but have a low degree of pozzolanic activity. They undergo hydration reactions and hydrated phases was found in all three materials in PXRD. The strength of concrete cubes made from the ash samples depend on the degree of substitution of SCM. The results from the compressive strength tests suggests that the ash material had better effect at 15% substitution then at 30%. Two of the ash fractions MIBA and fly ash seems to have a positive effect on compression strength. The slag sample did not seem to have any effect. But due to the low scale of testing with only single tests these results are not definitive.

Leaching from monolithic cracked cube samples of concrete with SCM showed that none of the made samples exceeded values set from leaching of monolithic samples of concrete from Netherlands, Flanders, France and Austria. Due to little knowledge a few regulations the interpretation of results from leaching tests on monolithic concrete is hard. There is a need for regulations and guidelines on how much leaching that can occur from monolithic samples of concrete in Sweden.

The results from this report show that it might be possible to use ash from one or more of the three different ash fractions as a cement replacement material. But in the end more research is required to be able to tell how these materials could be used. It is also concluded that more and easier to follow regulations is required in Sweden.

6 Continued Work

There are several ways that this work could be continued one is to repeat some of the experiments with more attempts. For example, the casting of concrete cubes could be made so that there are three cubes for each mixture and curing time. Then average values could be calculated and make the results more reliable eliminating potential outliers. The curing time could also be made longer to see what happens when the concrete cubes cure for longer times than 56 days.

Another possibility is to further investigate promising results like for example the fly ash that showed promising results in the mechanical test performed. Investigations could be done on what is the optimal degree of substitution. Another possibility is to try mixed design and mix the samples. It would also be interesting to do PXRD on the cured cube sample that were made and maybe compare different curing times.

7 Acknowledgement

I want to thank Karin Karlfeldt Fedje and Arezou Baba Ahmadi for all the supervision and help I have gotten when working with this project. I would also like to thank Amir Saeid Mohammadi and Bruno Oliveira Goncalves for being very helpful in my laboratory work.

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9 Appendix

9.1 Compressive strength test values

Table 4.2 Result of compression strength test after 7 days curing time.

Samples 7 Day Test	**Max Load (MPa)**
Reference 100 % cement	68
Reference 15 % Limestone	41
Reference 30 % Limestone	42
Fly Ash 15 %	52
Fly Ash 30 %	41
Slag 15 %	39
Slag 30 %	39
MIBA 15 %	60
MIBA 30 %	52

Table 4.3 Result of compression strength test after 28 days curing time.

Samples 28 Day Test	**Max Load (MPa)**
Reference 100 % cement	79
Reference 15 % Limestone	51
Reference 30 % Limestone	51
Fly Ash 15 %	71
Fly Ash 30 %	45
Slag 15 %	49
Slag 30 %	48
MIBA 15 %	65
MIBA 30 %	61

Table 4.4 Result of compression strength test after 56 days curing time.

Samples 56 Day Test	**Max Load (MPa)**
Reference 100 % cement	84
Reference 15 % Limestone	61
Reference 30 % Limestone	56
Fly Ash 15 %	80
Fly Ash 30 %	59
Slag 15 %	57
Slag 30 %	55
MIBA 15 %	64
MIBA 30 %	62

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