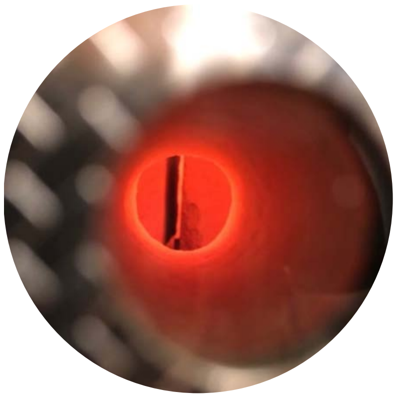
ECO CONCRETE IN FIRE



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ECO CONCRETE IN FIRE

BY

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BUILDING MATERIALS

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Table of Contents

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This project has already gathered many people at the same table over many discussions, exercises and helping each other with struggles which has been very uplifting and inspirational over this period.

I would like to express my thankfulness to prof. Andrzej Cwirzen, who is my main supervisor, Hans Hedlund and John J Provis, my co-supervisors, for their expertise, guidness and good word.

Sincere thanks to all my colleagues and administration personnel at Luleå University of Technology for their support, comprehensiveness, and kindness. Having a supportive and welcoming environment can make all the difference in achieving success and personal growth.

Marcin Sundin,
2023.

“There is a crack in everything, that’s how the light gets in.”

Leonard Cohen

SUMMARY

The recent report from the Intergovernmental Panel on Climate Change (IPCC) has emphasised the urgent need to take immediate steps to reduce carbon dioxide and other greenhouse gas emissions. One of the UN Sustainable Development Goals (SDGs) aims to ensure sustainable consumption and production of raw materials, promoting the transition of the industry towards more eco-friendly and sustainable practices. This transition will not only benefit the quality of human life but also the entire world ecosystem.

Concrete is one of the most widely used materials, second only to water. However, the traditional method of producing and using concrete involves the use of cement, which is responsible for significant CO_2 emissions, accounting for 8-10% of human-caused CO_2 emissions. Therefore, there is a growing demand for more detailed studies, research, and new technology applications to reduce the carbon footprint of concrete. One promising approach is to use industrial waste and by-products, such as steel slag, fly ash, mine tailings, rice husk ash, to replace Portland cement in concrete and at the same time to improve its performance under certain conditions.

One of the least explored areas regarding eco-friendly concretes are their behaviour under harsh conditions, such as fire and elevated temperatures. The present studies aim to investigate eco concretes exposed to 400°C and 800°C applied for 1 hour.

After conducting mechanical tests and microstructure investigations, all data were gathered to try to understand the occurred mechanisms and to highlight the possibilities and limitations of all studied binder combinations. The obtained results will be used to continue the project, which will provide valuable information to further improve the safety and performance of eco concrete structures.

The transition to more sustainable and eco-friendly concrete is a critical step towards achieving global sustainability goals. The knowledge gained from these studies will not only contribute to improve the safety and performance of eco-concrete structures but also to promote the development of more sustainable construction practices. By using more sustainable materials and exploring their behaviour under different conditions, we can reduce the carbon footprint of concrete, promote sustainable development, and create a better future for all.

LIST OF FIGURES

LIST OF TABELS

CHAPTER 1

1. INTRUDUCTION:

In recent years, there has been growing concern over the environmental impact of construction activities, particularly in terms of carbon emissions. One of the major contributors to these emissions is the production of Portland cement, which is an essential ingredient in traditional concrete. As a result, researchers and engineers have been exploring ways to develop eco-friendly concrete alternatives with a lower carbon footprint.

One common approach to developing eco-friendly concrete is to partially replace Portland cement with alternative cementitious materials (SCMs), such as blast furnace slag, fly ash, and metakaolin. These materials can be sourced from industrial waste products or naturally occurring substances, and their use can significantly reduce the carbon footprint of the resulting concrete.

In addition to these commonly used SCMs, there are other alternative materials that are being considered for use in eco-friendly concrete. Natural pozzolans, for example, are volcanic rocks that can be used to partially replace Portland cement. Mining wastes, such as tailings from copper or gold mines, are also being investigated as potential SCMs sources.

Another approach to eco-friendly concrete is the use of alkali-activated binders, which are commonly referred to as geopolymers. These binders can be made from a variety of materials, including industrial waste products, and offer several advantages over traditional concrete. Calcium Sulphoaluminate Cements (CSA) are another alternative to Portland cement and can be used in combination with Portland cement to create eco-friendly concrete. These cements have faster setting times than Portland cement, which can be useful in certain construction applications.

While the development of eco-friendly concrete is an important step towards reducing carbon emissions in the construction industry, it is also important to ensure that these materials are safe to use in all conditions. Traditional concrete based on Portland cement has been extensively studied in terms of its performance at high temperatures and in fire exposure, and the current knowledge suggests that it can be used safely with the addition of certain additives, such as polypropylene fibres (PP). However, the fire resistance of eco-friendly concrete is less understood.

Recycled concrete aggregates (RCA) are another factor that can affect the fire resistance of eco-friendly concrete. While the use of RCA can reduce the carbon footprint of concrete, it can also lower the fire resistance of the resulting material. This is due to the presence of residual binders and other impurities in the recycled material, which could lead in some circumstances to catastrophic consequences in the event of a fire. As a result, it is important to carefully consider the use of RCA in eco-friendly concrete and to take appropriate safety measures to mitigate any potential risks.

1.1 AIM AND OBJECTIVES

The aim of the PhD project is to support the Swedish construction industry in its safe and economical transition to a new reality with extensive usage of low CO_2 footprint concretes.

The PhD project contribution is to build up the necessary scientific knowledge and to enable contractors safe utilisation of eco concretes in structures exposed to high temperatures and to fire.

The transition towards eco-friendly concrete with low carbon footprints is becoming increasingly important in the construction industry as environmental concerns are gaining traction. The Swedish construction industry is looking for ways to reduce their carbon emissions while ensuring the safety and reliability of the structures they build. The project aims to address these concerns by gaining knowledge that will enable contractors to safely use eco concretes in high-temperature and fire exposure scenarios.

This output of this research will provide building scientific and practical knowledge related to eco concrete. It will support contractors to make right decisions about the use of eco-friendly concrete in their projects. This knowledge is particularly useful in the selection of the appropriate materials, the determination of mixing proportions, and the development of strategies for ensuring the structural integrity of concrete in the face of high temperatures and fire exposure.

This midterm report is based on data obtained from performed first part of the laboratory small-scale testing.

1.2 RESEARCH QUESTIONS

The following research questions/objectives were formulated for this part of the study:

1. How do regular OPC based concretes perform when exposed to very high temperature in comparison with Eco concretes?
2. What are the effects of exposure to high temperature/fire on binder matrix of eco concretes, i.e., phase composition, microstructure?
3. What are the effects of exposure to high temperature/fire on spalling, mechanical properties and microcracking of eco concretes?
4. How chemical admixtures influence the performance of eco concretes when exposed to high temperatures?

1.3 LIMITATIONS

The primary objective of this research is to assess the fire performance of environmentally friendly concrete. To achieve this goal, collaboration with local industries and response to current market demands have led to an investigation of supplementary cementing materials incorporated into concrete mix design under exposure to elevated temperatures.

The composition and properties of ground granulated blast furnace slag, a commonly used supplementary cementing material, can vary significantly between suppliers. As a result, each batch and type of slag must be carefully examined and evaluated to ensure consistency in the results obtained from the testing process. In this study, only one type of slag that is commercially available in Sweden has been tested.

Furnace used in this part of the study was only capable of closely following the standardised fire curves up to 400°C. Above this threshold, the speed of the heat rate was slower. Only two temperatures were used in this part of the study, i.e., 400 and 800 °C. The maximum achievable temperature of that furnace is 1300 °C.

1.4 CHAPTER OVERVIEW

This document is structured into six chapters, with additional research papers included as appendices.

Chapter 1 introduces the work, including its objectives and limitations, and formulates the research questions.

Chapter 2 outlines the theoretical background and current state of the art in the field.

Chapter 3 presents the materials and methods used in this work.

Chapter 4 provides an analysis of the results obtained from the tests conducted, as well as a discussion of the data.

Chapters 5, 6, and 7 are dedicated to the conclusions drawn from the study and future research directions.

1.5 LIST OF APPENDED PUBLICATIONS

The following publications are included in this mid-term report:

Paper A:	Sundin, M., Hedlund H., Provis L J., Cwirzen, A. (2022) Effects of high and very temperatures on blast furnace slag concrete. Extended conference abstract. ICCRRR 2023 Cape Town, South Africa
Contributions:	Conceptualization, Methodology, Investigation, Testing setup, Experimental performance, Draft writing M.Sundin, Draft reviewing A.Cwirzen, J. L. Provis, H. Hedlund.

Main finding: Enhanced mechanical properties of eco concrete with partial replacement of cement by GGBFS in elevated temperature. Due to higher density and quartz filler there is none spalling of extensive cracking.

Paper B: Sundin, M., Hedlund H., Provis L J., Cwirzen, A. (2023)

Eco friendly concrete in fire and elevated temperatures- review. (submitted)

Contributions: Conceptualization, Methodology, Investigation, Testing setup, Experimental performance, Draft writing M. Sundin, Draft reviewing A. Cwirzen, J. L. Provis, H. Hedlund.

Main finding: Review of current state of art an available data regarding performance of eco concrete in elevated temperatures and fire conditions. It shows that the most papers are focused on load free condition and small-scale tastings. No clear indications of spalling source and influence of concrete admixtures.

CHAPTER 2

2. FIRE RESISTANCE OF CONCRETE

Fire has undoubtedly played a significant role in the development of human civilization. From the Stone Age, when our ancestors first discovered the use of fire, it has been an essential tool for cooking, heating, and light. Over time, humans have learned to harness fire to advance our technological capabilities, from the creation of tools and weapons to the development of complex machinery and advanced technologies such as space travel.

However, while fire has enabled us to achieve incredible feats, it can also be a destructive and deadly force if left uncontrolled. The construction industry is particularly sensitive to the risks posed by fire, and buildings must be designed and constructed to minimize the potential for fire hazards. In many cases, such as in tunnels, underground garages, and multi-storey buildings, thousands of people can be exposed to life-threatening situations if a fire breaks out. The survival chances of individuals caught in a fire depend on numerous factors, including the architectural and structural design of the building, the types of materials used in construction, and the availability of emergency systems and protocols.

The fire resistance and integrity of a structure are largely influenced by the properties of the materials used in construction [1]. Concrete is a widely used material in the construction industry due to its durability in fire exposure compared to other materials such as steel or timber [2]. Concrete can even be used as a protective shield for other materials. However, exposure of concrete to fire initiates various processes that can affect its integrity and load-bearing capacity [3].

These processes include the evaporation and migration of capillary water, dehydration of phases like CSH and CH, which are essential to the strength of the hydrated cement matrix [1]. These processes typically occur at temperatures just below 1000°C, which can be easily reached during a fire. As a result, the binder matrix is destroyed and loses its mechanical strength. In addition, there is an incompatibility between the binder matrix and aggregates, with the binder matrix undergoing expansion until 150°C followed by thermal shrinkage. At the same time, aggregates expand with increasing temperature, producing thermal-related strain and cracking. Therefore, the mix design of concrete, including the type of cement and aggregate used, plays a critical role in determining the ultimate degradation of its mechanical properties. Moreover, larger aggregate sizes have been found to increase the rate of deterioration [1]. Summary of the most influential factors related to the fire resistance of concrete are given in Table 1.

Table 1 Factor influencing fire resistance of eco concrete

Category	Parameters
Concrete Mix design	Gradation, shape, and amount of coarse aggregate
	Water to binder ratio
	Amount and type of additives

	Binder type and amount
Fresh concrete properties	Concrete density
	Curing regime
Elevated temperature exposure	Scale of tests (small or large scale)
	Heating rate
	Cooling rate and type
	Fire flame vs temperature rise
	Geometry and size of element
	Spalling and cracks
	Surrounding conditions
	Applied loads
	Additional events

Therefore, it is critical to continually improve our understanding of the behaviour of fire in different settings and identify ways to minimize its impact and prevent catastrophic events.

Designing materials that can withstand high temperatures is a challenging task that requires careful consideration of the material's behaviour under different temperature regimes. Various factors, such as the type of cement and aggregates used, the mix design, and the temperature exposure, can significantly affect the material's performance. It is crucial to understand these factors and develop appropriate testing methods to evaluate the material's properties under different conditions accurately.

2.1 DETORIARATION MECHANISM OF CONCRETE EXPOSED TO HIGH TEMPERATURE

2.1.1 CHEMICAL DECOMPOSITION OF THE BINDER MATRIX

All materials, including also concrete, subjected to high temperature or fire will sustain changes in chemical and mineralogical compositions. In the case of concretes these include binder matrix, aggregates, fibers, and reinforcement, all of which will affect that ultimate performance of concrete structures. Chemical changes of the binder matrix depend on the type of cement used, but also on the types and amount of SCM present in the mix. This section will briefly describe the most important chemical changes to binders composed of OPC, OPC+SF, OPC+BFS and CSA.

The information provided by Khoury [4] highlights the complex behaviour of materials exposed to high temperatures. As temperature rises, different chemical and physical reactions take place within the material's structure, leading to a decrease in its mechanical properties and a potential loss of structural integrity.

For example in the case of hydrated Portland cement, at around 300°C-400°C, $Ca(Oh)_2$ begins to separate, and creep deformation occurs. At 600°C, serious damage to the matrix can be observed. At 700 °C, $CaCO_3$ with a ceramic structure dissociates, which further weakens the material. Beyond 800°C, the absolute water is lost, and the melting point is typically reached at 1200 °C -1350 °C, Table 2.

Slag concrete can undergo decomposition when exposed to fire, as shown in Table 3. The high temperatures cause a loss of strength and durability, and changes in the microstructure of the concrete. At temperatures between 200°C and 400°C, a new gel is formed which increases the density and strength of the concrete. However, at higher temperatures, the calcium silicate hydrates (C-S-H) and calcium hydroxide (Ca(OH)2) present in the concrete dehydrate. The slag particles themselves can also undergo changes and transform into crystalline phases.

Fly ash-based concrete can experience chemical changes when exposed to high temperatures, as shown in Table 4. At temperatures between 200°C and 400°C, a new gel is formed, which increases density and strength. However, at higher temperatures, the concrete loses strength and durability. The calcium silicate hydrates (C-S-H) and calcium hydroxide $Ca(OH)_2$ present in the concrete dehydrate, leading to the formation of new phases such as gehlenite and anhydrite. Furthermore, the fly ash particles themselves undergo transformations and become crystalline phases like mullite and hematite.

When exposed to high temperatures, the calcium sulfoaluminate (CSA) phases present in CSA-based concrete can undergo decomposition, Table 5. At higher temperatures, the decomposition of CSA phases can result in the loss of strength and overall durability. Between 200°C-230°C alumina trihydrate dehydroxylates, and above it, monosulfite dehydrates. The decomposition reactions can result in the formation of new phases, such as gehlenite and anhydrite, and the dehydration of calcium sulfoaluminate hydrates and calcium sulfate hemihydrate.

All these changes can lead to microstructural alterations and reduce the overall performance of the concrete under high temperature conditions.

Table 2 Ordinary Portland Cement (OPC) phase decomposition [4–6]

Temperature	Phase decomposition
>200°C	Partially ettringite dehydration and beginning calcium–silicate hydrates, C-S-H dehydration.
>300°C	Dehydration end of ettringite and dehydration follow-up of calcium–silicate hydrates C-S-H.
>500°C	Follow-up dehydration of calcium–silicate hydrates C-S-H and dihydroxylation of portlandite $Ca(OH)_2$.
>800°C	Follow-up dehydration of calcium–silicate hydrates C-S-H and partially carbonate decomposition.
>1200°C	Melting point for aggregates.

Table 3 Slag concrete phase decomposition [8]

Temperature	Phase decomposition
<200°C	Evaporation of free, mass loss, better mechanical properties by temperature induced gel formation
200-400°C	Strength increase, formation of gel, decreasing porosity [5]
400-800°C	Strength loss due to Gel crystallization, loss of strength performance
>800°C	decomposition of C-S-H, formation of ß-C2S and C3S, akermanite, merwinite and gehlenite in the sodium sulfate activated slags [6]

Table 4 Fly ash (FA)concrete phase decomposition [81]

Temperature	Phase decomposition
<200°C	Free water evaporation, mass loss, enhanced mechanical properties by temperature induced gel formation, less unreacted particles, increase density due to filled up pores by newly formed gel
200-400°C	High contraction which leads to removal of chemically bound water, continuation of gel formation and increasing the density, peak of strength value [5]

400-600°C	Slow shrinkage caused by dihydroxylation (formation of hematite and anhydrite) [7], strength loss due to crystallization of gel
600-800°C	Further shrinkage, oxidation of presence iron oxides, decrease of porosity, formation of micropores and microcracks [8]
800-1000°C	Final part of particles coalescence, deterioration of matrix, critical damage of element.

Table 5 Calcium sulfoaluminate (CSA) phase decomposition [55]

Temperature	Phase decomposition
<90°C	Ettringite dehydration and decomposition to monosulfite and calcium sulfate
>150°C	Partially monosulfite dehydration
200-230°C	Alumina trihydrate dihydroxylation
>450°C	Monosulfite dehydration

2.1.2 CRACKING, SPALLING AND DISSCOLORATION

Crazing and microcracking of the binder matrix are primarily caused by thermal incompatibility between the components of the matrix. Spalling occurs when the outer layer of concrete gradually peels or scales off when exposed to high temperatures. The exact mechanism is not yet fully understood, but it is believed that the build-up of vapor pressure from entrapped water at elevated temperatures is a key factor [11]. The pressure combined with thermal stress can lead to explosive spalling, which can cause significant damage to the affected structure [12]. The presence of interconnected pores allows gas or water to migrate from the inside to the outside of the concrete element, but the built-up pressure can exceed the tensile strength of the concrete matrix, leading to spalling. Spalling has been responsible for several building catastrophes and can be influenced by mechanical loads such as own-weight, permanent and variable loads, snow, wind, earthquakes, and explosions. Previous studies have investigated spalling under load-free conditions or with applied mechanical loads, and the temperature range of 320-360°C has been reported to result in explosive spalling. Horizontal elements exposed to high temperatures and a load of 15 MPa have also been shown to be significantly prone to spalling [13].

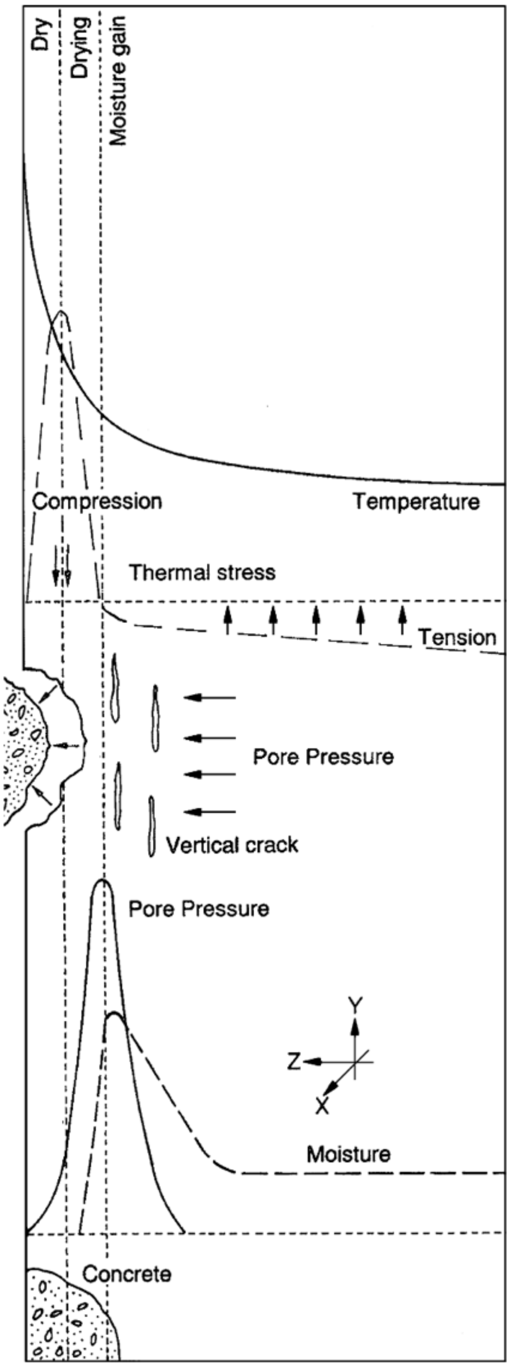


Figure 1 Combined stress and pore pressure in triggering spalling [14]

There are two commonly used methods for analysing colour changes in concrete: (i) observing the external surface and (ii) analysing a cut-out section of the concrete element to view the aggregates within [1]. The reason for colour change is due to the dehydration of the binder matrix and changes in the structure of aggregates [15]. When siliceous aggregates are present and exposed to temperatures between 300°C and 600°C, the concrete turns red. At 600°C-900°C, the colour becomes more white-grey, and between 900°C-1000°C, it turns pale yellowish.

The most notable colour change, which is red, occurs when iron is present in the slag, binder, or aggregates [16] or when siliceous riverbed aggregates are used [17]. However, if calcium carbonate aggregates are used, the colour turns paler, appearing whiteish due to the $CaCO_3$ calcination reaction [18].

Accurate and comparable data can be obtained by measuring the hue, saturation, and intensity, which are based on the wavelength. With the help of appropriate software, analysing and recognizing the data can be relatively simple.

2.2 ECO CONCRETES IN FIRE

Despite the availability of numerous test results, the fire durability of concretes incorporating supplementary cementitious materials (SCMs), geopolymers or calcium sulphoaluminate (CSA) cements remains unclear and, in several cases, highly questionable. Geopolymers made from blast furnace slag and activated by alkalis were found to perform better at very high temperatures in contrast to Portland cement-based concretes [19].

Fire resistance of concretes formulated with alternative binders, such as Portland cements blended with SCMs (like blast furnace slag, fly ash or calcinated clay), geopolymers or CSA cements, is challenging to evaluate. For instance, Eco cement that contains blast furnace slag, which is becoming increasingly popular in Sweden, tends to have higher strength loss with increasing temperature. On the other hand, some tests on geopolymers, showed promising results. Therefore, it is imperative to investigate all potential combinations of cementitious binders to assess their performance at high temperatures and in the event of a fire.

Mendes et.al [20]conducted a study to determine how different cooling methods affected the fire resistance of OPC slag concretes when exposed to temperatures of 400°C and 800°C. The results showed that the reference OPC concrete specimens lost 14% of residual compressive strength, while the slag blends concretes only lost 4-5% when loaded at 800°C. The use of slag and waste glass in concrete mixes has been shown to improve workability and fire resistance. Due to the low thermal conductivity of concrete, the internal temperature during heating is reduced, and samples containing slag and waste glass deform less and experience less spalling due to their dense structure and the use of melted glass pieces as inner locks against cracks, according to previous research [21]

Mussa et al. [22]conducted a study on high volume fly ash concrete with nano silica, which showed better results compared to OPC. The data collected from dynamic compressive strength tests, toughness, maximum strain, and destruction after exposure to high temperatures of 400°C and 700°C indicates the superiority of this material. Another research by Razak et al. [23] evaluated the fire performance of geopolymer concrete based on fly ash at temperatures of 500°C and 1200°C for 120 minutes. The alkali-activated mixture showed significantly better results than the OPC reference samples. The geopolymer concrete exhibited visible improvement in residual compressive strength at 500°C (145% of initial strength) due to gepolymerization, while the OPC samples lost almost 50% of their strength. The increased density of geopolymer concrete also prevented spalling and cracking events.

Current types of calcium sulfoaluminate (CSA) cement show a better performance in terms of compressive strength, shrinkage behaviour, and early strength gain when compared to ordinary Portland cement (OPC) under ambient conditions [24], the actual data shows that their performance deteriorates rapidly at temperatures above 150°C due to their high permeability [25, 26]. While CSA cement may be a good choice for certain applications, its suitability for use in high-temperature environments needs to be thoroughly evaluated.

To improve plastic waste management and promote sustainability, there is a variety of recycled synthetic fibres available for use in concrete production. Polyethylene (PE), polyethylene terephthalate (PET), PP, and PVA are among the most promising fibres due to their high production volume and ease of processing [27]. PET is popular in various industries and has hydrophobic properties. However, the use of synthetic fibres may decrease thermal resistance and elastic modulus in concrete, potentially leading to dangerous behaviour during a fire event. Nonetheless, as the recycling industry continues to evolve, synthetic fibres may play an increasingly important role in concrete production in the future.

Cellulose fibres such as jute, hemp, kenaf bagasse, and sisal are another sustainable option for reinforcing concrete [28]. These fibres offer benefits such as low density, reduced thermal conductivity, and improved mechanical performance. However, they also have disadvantages, such as short longevity, poor workability, and low compatibility with geopolymer matrices [29]. Volatilizable fibres decomposable at low temperatures are not recommended as an admixture for enhancing high-temperature resistance due to their tendency to shrink and create pores under load. Despite these challenges, the use of recycled synthetic and cellulose fibres in concrete production represents an important step towards sustainable construction practices.

The incorporation of fibres into geopolymer concrete matrix can bring numerous benefits to the development of more sustainable and ecological materials. Not only can it enhance the mechanical and thermal properties of the material, but it can also contribute to the reduction of waste and promote smarter waste management practices. By utilizing recycled fibres in concrete production, waste can be diverted from landfills, reducing the volume of waste that requires storage and allowing for the creation of extended green zones. This can have positive impacts on both the environment and society, such as improving air and water quality, creating new spaces for recreation and leisure, and reducing the negative impacts associated with traditional waste disposal methods. Additionally, the incorporation of fibres into geopolymer concrete can also provide economic benefits, such as reducing material costs, improving the longevity and durability of concrete structures, and promoting sustainable practices in the construction industry.

3. MATERIAL AND METHODS

The following section describes materials and methods used in this study.

3.1 MATERIALS

3.1.1 ORDINARY PORTLAND CEMENT

Three types of Portland cements delivered by Cementa AB and CEMEX POLAND were used in this study. This included classes EN 197-1: CEM 32.5 (CEM II/B-V 32,5 R – HSR), CEM I 42.5N (Anläggningscement), and CEM I 52.5R (Snabbcement skövde). Their physical and chemical properties as provided by the producer are shown in Table 6, Table 7, Table 8.

Table 6 Physical and chemical properties of CEM II 32,5 B-V 32,5 R – HSR

Blaine fineness (m^2/kg)	**Setting time (min)**	**Bulk density (kg/m^3)**	**LOI (%)**	**Insoluble (%)**	**Chloride (%)**	**Alkali Na2O (%)**	**SO_3 (%)**
352	298	1100	n/a	0-0.2	0.062	1.30	2.64

Table 7 5 Physical and chemical properties of the CEM I 42,5N

Blaine fineness (m^2/kg)	**Setting time (min)**	**Bulk density (kg/m^3)**	**LOI (%)**	**Insoluble (%)**	**Chloride (%)**	**Alkali Na2O (%)**	**Equiv.**
330	170	1250	0.5-0.4	0-0.2	0.01-0.03	0.40-0.58	

Table 8 5 Physical and chemical properties of the CEM I 52.5 5N

Blaine fineness (m^2/kg)	**Setting time (min)**	**Bulk density (kg/m^3)**	**LOI (%)**	**Insoluble (%)**	**Chloride (%)**	**Alkali Na2O (%)**	**SO_3**
362	223	972	2.555	0-0.2	0.01	0,62	<4.0

3.1.2 GRANULATED BLAST FURNANCE SLAG (GGBFS)

The used in this research GGBFS type "Merit" was delivered by Swecem. The physical properties and chemical composition as provided by producer are shown in Table 9, Table 10, respectively. The XRD spectra is shown in Figure 2

Table 9 Physical properties of the used GGBFS type Merit.

D_{50}	Blaine fineness (m^2/kg)	Density	Bulk density	Glass content %
30-34	30-35	2.8-3.0	0.9-1.1	97-99

Table 10 Chemical composition of GGBFS Merit.

CaO	SiO_2	Al_2O_3	MgO	TiO_2	Mn_2O_3	Cl
30-34	30-35	10-13	12-15	1.5-2.5	0.3-0.6	<0.1

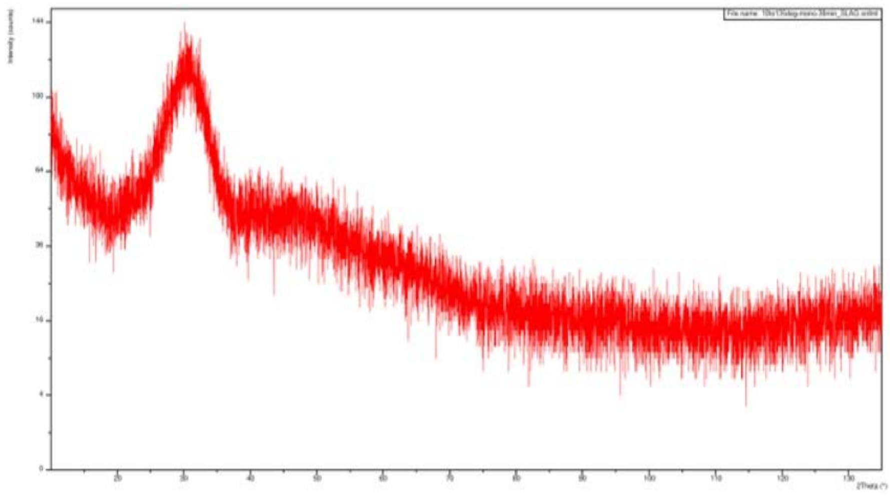


Figure 2 MERIT XRD spectra

3.1.3 Calcium sulphoaluminate cement (CSA)

Calcium sulphoaluminate cement (CSA) type BELI used in this research was delivered by Caltra's. CSA BELI cement is a type of hydraulic cement that is produced by combining calcium sulfoaluminate clinker with a small amount of gypsum. It is known for its rapid-setting and high early strength properties, making it a popular choice in applications where a quick turnaround time is needed. Its low carbon footprint and durability also make it a sustainable option for eco-conscious builders. Physical properties and chemical composition are given in Table 11, Table 12, respectively.

Table 11 Physical properties of CSA BELI.

Blaine fineness (m^2/kg)	**Density (kg/m^3)**	**LOI**	**Standard strength class MPa**
50	2,9	<0.2	52.5

Table 12 Chemical composition of CSA BELI.

CaO	**SiO_2**	**Al_2O_3**	**MgO**	**Fe_2O_3**	**SO_3**	**Belite**	**Calcium sulphate: wt%**	**Ye'elimite cont. wt% of CSA clinker**
40-44	6-10	>32	<1.5	<2.2	9-10	25	10	>60

3.1.4 AGREGATES

The Jehander Heidelberg cement group supplied crushed granite aggregates with a gradation of 0-4 mm and 4-8 mm, and natural fine filler was provided by Skanska AB in Luleå, Sweden and quartz filler were also utilized. These aggregates are preferred due to their superior mechanical properties such as high compressive strength, low water absorption, and high resistance to wear and tear. Its specific of gravity is 2,7 t/m^3. Sieving curves for aggregates and filler are presented on the Figure 3.

The natural fine filler from Skanska AB is also a popular choice for concrete production because it is an eco-friendly material that can be obtained from renewable sources. The quartz filler in the mix also helps to reduce the porosity of the concrete, making it more impermeable to water and other liquids. Its use in concrete mixes can help reduce carbon emissions and promote sustainability in the construction industry.

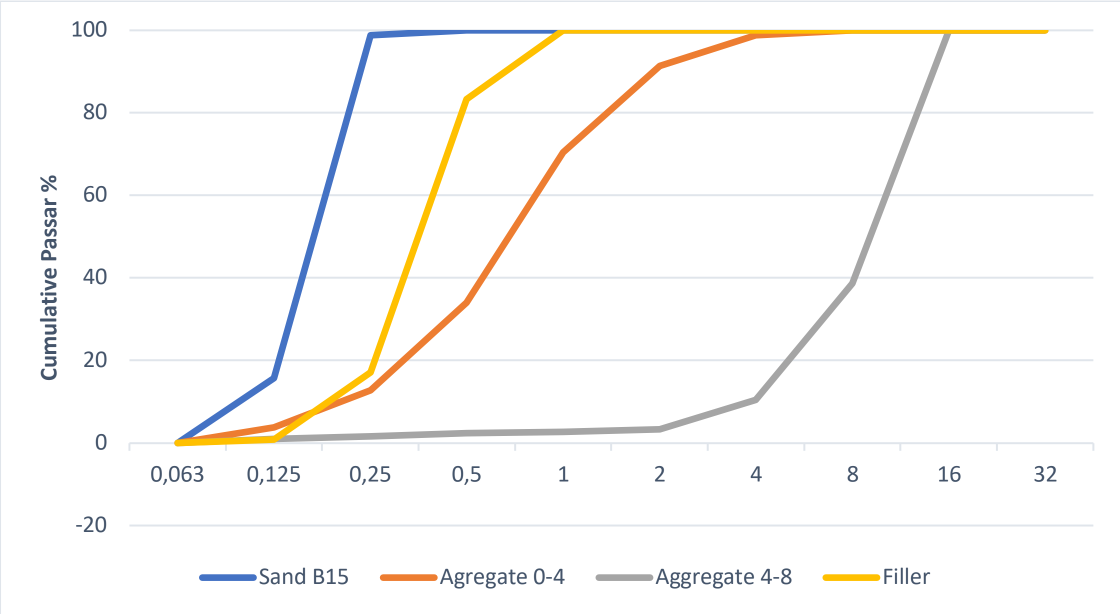


Figure 4 Sieving curves for aggregates and filler

3.1.5 ADMIXTURES

Admixtures are essential components of modern concrete technology that enhance the properties of concrete in various ways. In this case, BASF AB provided three different types of admixtures for the concrete mix: MasterX-Seed 100, MasterAir 105, and MasterEase 3300. MasterX-Seed 100 is an accelerator that is used to speed up the hydration process of cement, allowing the concrete to reach its desired strength more quickly. MasterAir 105 is an air entrainer that is used to create tiny air bubbles in the concrete mix, which can improve its durability and resistance to freeze-thaw cycles. Finally, MasterEase 3300 is a superplasticizer that improves the workability of concrete, making it easier to pour and shape while also reducing the amount of water needed.

3.1.6 MIX DESIGN

A total of 25 different mixes were prepared. Content of coarse-, fine- aggregates and filler was constant. Mixes differed only in the binder composition and presence of chemical admixtures. The total binder content was 400 kg/m^3 and the water to binder ratio was 0.4, Table 13.

Table 13 Mix composition.AE- air entraining admixture, AC- accelerator, SP-superplasticizer, SF-Skanska Filler.

Mix	W/C	OPC kg/m^3	OPC type	CSA kg/m^3	GGBFS kg/m^3	Admix wt% of binder (type)	Coarse ag. (2-8mm) kg/m^3	Fine ag. (0-4mm) kg/m^3	Quartz filler kg/m^3

Ref.1	0,5	400	32,5				522	1131	87
Ref.2	0,5	400	42,5				522	1131	87
Ref.3	0,5	400	52,5				522	1131	87
1B10	0,5	360	32,5		40		522	1131	87
1B50	0,5	200	32,5		200		522	1131	87
1B90	0,5	40	32,5		360		522	1131	87
2B10	0,5		42,5		40		522	1131	87
2B50	0,5	200	42,5		200		522	1131	87
2B90	0,5		42,5		40		522	1131	87
3B10	0,5		52,5		40		522	1131	87
3B50	0,5	200	52,5		200		522	1131	87
3B90	0,5		52,5		360		522	1131	87
Ref.C	0,5	-		400			522	1131	87
50C50B	0,5			200	200		522	1131	87
50O25C25B	0,5	200	52,5	100	100		522	1131	87
25O50C25B	0,5	100	52,5	200	100		522	1131	87
25O25C50B	0,5	100	52,5	100	200		522	1131	87
AC4	0,5	400	42,5			4 (AC)	522	1131	87
AC10	0,5	400	42,5			10 (AC)	522	1131	87
AE	0,5	400	42,5			0,1 (AE)	522	1131	87
SP	0,5	400	42,5			0,5 (SP)	522	1131	87
REF D	0,5	400	42,5				522	1131	87 (SF)
1D	0,5	200	42,5		200		522	1131	87 (SF)

REF E	0,5	400	32,5 52,5				522	1131	87
1E	0,5	200	32,5 52,5		200	0,36 (SP)	522	1131	87

3.2 METHODS:

3.2.1 SAMPLE PREPARATION

3.2.1.1 CUBES 100x100x100 mm

The plastic molds were used for casting 100x100x100 mm, concrete specimens. All concrete mixes were produced using a 20-liter "Hobart" A200N type mixer, Figure 5. The mixing procedure was standardised for all mixes and consisted of adding all the dry components to the mixer and mixing them for 5 minutes. After this, water was added, and an additional 5 minutes of mixing was performed. The total mixing time varied between 10 to 15 minutes, depending on the specific mix design.

To ensure easy demoulding of the concrete specimens without causing any damage, the molds were first sprayed with a form oil. After the specimens were sealed in the molds and left to harden for a day, they were carefully removed from the molds and subjected to various measurements, including geometrical measurements, weight, labelling, and photography. The specimens were then resealed and allowed to air cure for a total of 28 days. Once the curing period was complete, the specimens were once again measured, weighed, and photographed. The same measurements were taken after exposing the specimens to elevated temperatures. It was crucial to document any changes in the specimens' physical properties, such as cracks or deformations, to accurately assess the impact of the temperature exposure on the concrete's strength and durability.

Figure 5 Mixer used in this study, type Hobart 200N

3.2.1.2 SAMPELS AND METHODS FOR MICROSTRUCTURE analysis

The analysis of the microstructure was a critical step in evaluating the performance of the concrete specimens. The scanning microscope SEM JSMIT100 with energy-dispersive spectrometry (EDS) from Bruker type Quantax provided high-resolution images and data on the composition of the samples, Figure 6. The tests were conducted 28 days after casting. Before the analysis, the samples were stored in isopropanol for 5 days to inhibit the hydration process and prevent any further chemical reactions. The samples were then stored in a desiccator to ensure they were completely dry before impregnation with a low viscosity epoxy resin. Polishing was done using diamond sprays with particle sizes of 9, 3, and 1 μm. The magnification of 500x provided a detailed view of the microstructure of the samples. The SEM backscattered electron mode (BSE) was used. The SEM-EDS spot analysis was performed at locations assumed to be predominantly occupied by the CSH phases, based on the grey levels of BSE images. The SEM acceleration voltage was 15 kV, the electron beam current was 50 mA, and the chamber vacuum was 30Pa. The SEM-EDS analysis involved counting 50,000 counts per each analysis to obtain accurate data on the composition of the samples.

Figure 6 . SEM tyle Jeol JSMIT100 used in this study.

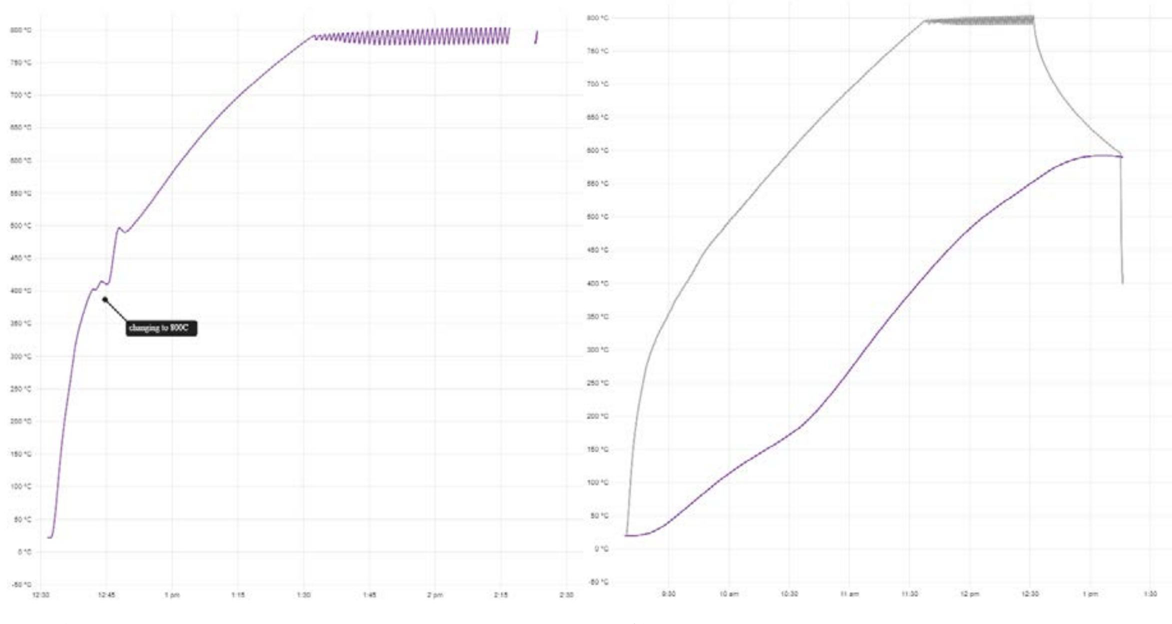
3.2.2 TEMPERATURE LOAD

The electric laboratory muffle furnace is an essential tool for this study. The LH15 furnace provided by LAC has a compact design and a high-quality heating chamber with a capacity of 15 litters, Figure 7. The furnace can reach temperatures up to 1400°C, which makes it good option for initial studies of temperature loads on different materials. The furnace is equipped with a PID temperature controller that provides accurate temperature control with a stability of ±1°C. The heating elements used in the furnace are made of high-quality materials that ensure long-lasting performance and durability. The LH15 furnace also has a safety system that includes over-temperature protection, which ensures safe operation even at high temperatures. The furnace's heating rate is adjustable, and it can reach high temperatures quickly, making it an efficient tool for time-sensitive experiments. The temperature of 400^{o}C is reached within 30 min (13,33^{o}C/min) and 800^{o}C within 175 min (4,57^{o}C/min). The technical characterization of furnace is shown in Table 14. The measured heating rates for empty and loaded furnace are shown in Figure 8.

Figure 7 Furnace used in the project LAC LH15.

Table 14 Technical characteristic of the used furnace.

FURNACE TYPE (*1)	**NOMINAL VOLTAGE U_n [V] (*2)**	**NOMINAL CURRENT I_n [A] (*3)**	**POWER INPUT P_{max} [kW] (*4)**	**MAXIMUM TEMPERATURE T_{max} [°C] (*5)**	**INTERNAL DIMENSION w x h x d [mm]**	**IP RATING (*6)**
LH15	1/N/PE 230 V AC	10, 4 (16)	2, 4	1340	250x250x250	IP 40



a) b)

Figure 8 Measured heating curves of the used furnace, a) unloaded furnace, b) loaded furnace with four concrete cubes 100x100x10mm with 10 mm space around each.(no stacking)

The specimens were loaded into the heating chamber and kept at the desired temperature for 60 minutes. Once the cycle was finished, the furnace was powered off and the ventilation system was initiated. To prevent any harm to the external components of the furnace, the temperature needed to fall below 300°C before the chamber could be opened. The specimens were kept inside the furnace overnight and extracted the following day to continue the testing.

3.2.3 MECHANICAL TESTS

Mechanical tests in this study included determination of the compressive strength. Measurements were done using Toni Technik compression machine, Figure 9. The load was applied at a constant rate of 10kN/s until the specimen failed. The test was carried in accordance with SS-EN 12390-3, which outlines the procedures for testing hardened concrete. The maximum force at failure was recorded, and the compressive strength was calculated by dividing the maximum force by the cross-sectional area of the specimen.

Figure 9 Compressive strength machine used in the project

4. RESULTS AND DISCUSSION

4.1 COMPRESIVE STRENGHT

All the results obtained from the compressive strength tests of samples aged for 28 days and subjected to three different temperature loads were collected and compiled below.

The results showed a generally good performance of concretes with GGBFS, Figure 10. An exposure to 400oC resulted in a similar or better compressive strength in comparison with the reference concrete made with 100 wt% of Portland cement. At 800oC the compressive strength was significantly lower when 90 wt.% of Portland cement was replaced with GGBFS. Concretes containing CSA cement in combination with OPC and/or BFS showed mixed results and must be further investigated.

Numerical data for all results are shown in Appendix A.

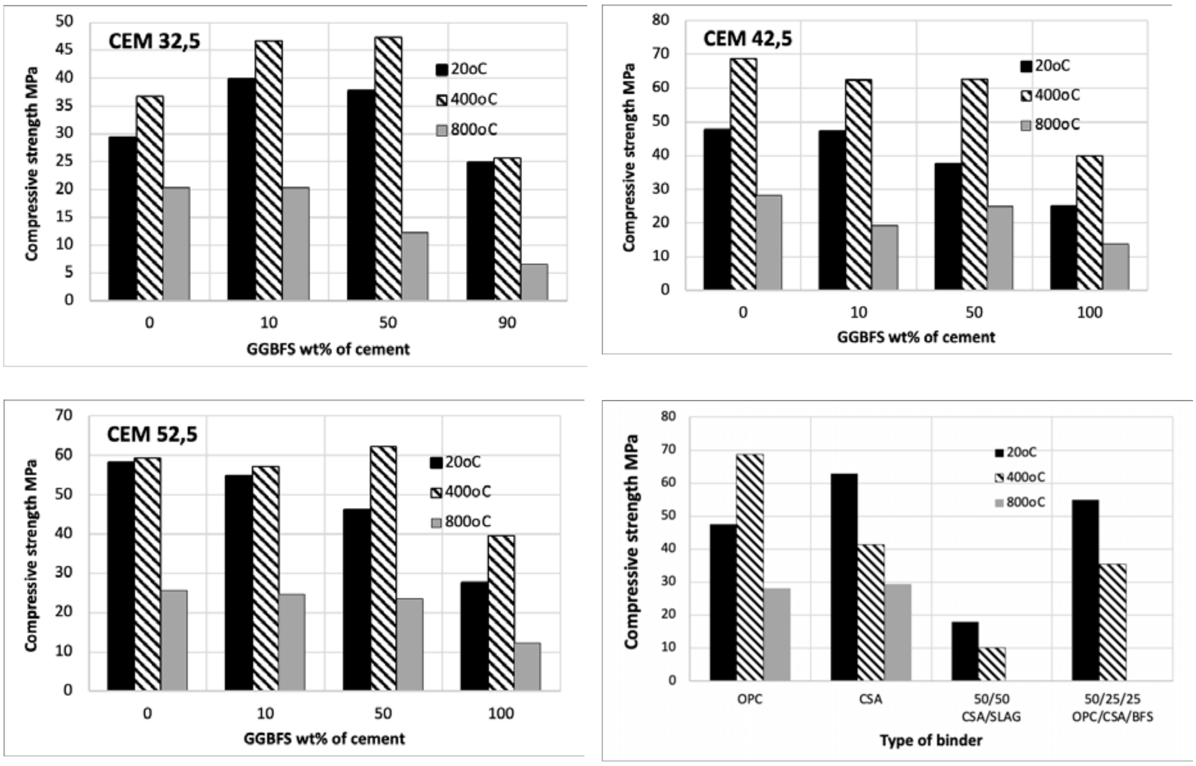


Figure 10. Effects of 400 and 800^oC on compressive strength of concretes made of three types of Portland cement and containing 10, 50 and 90 wt% of GGBFS. Effects of CSA cement on compressive strength.

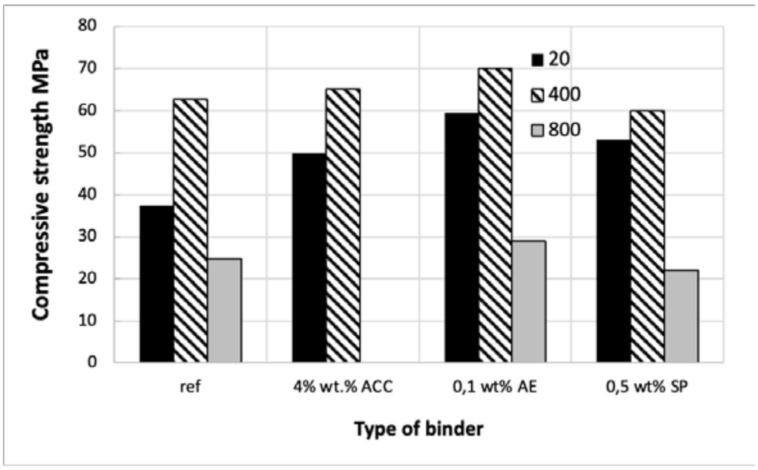


Figure 11. Effects of chemical admixtures on compressive strength of concretes containing 50 wt% of CEM 42,5 and 50 wt% of GGBFS. ACC- synthetic CSH base accelerator, AE-air entraining admixture, SP-Polycarboxylate based superplasticizer. Right image shows remaining of samples containing ACC and exposed to 800oC.

4.2 MASS LOSS

Due to the evaporation of water caused by exposure to elevated temperatures, the specimens have experienced weight loss. The following data presents the selected weights of a single cube after being subjected to different temperature loads, demonstrating the effect of temperature on the weight of the samples. It is important to note that the weight loss due to temperature exposure can have significant implications for the durability and strength of the concrete.

It has been noticed that mix containing 50% of OPC and 50 % of GGBFS was the denser (Table 15). It resulted in the smallest mass loss from all tested specimens and in the superior result regarding the exposure to elevated temperatures. The biggest diffrence in weight before and after exposure was reported on hybrid sampels contaning two types of cements with 50 % replecment by GGBFS.

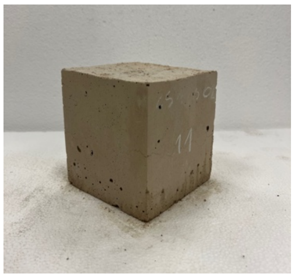
Table 16 Selected weights of the specimens after temperature exposure

MIX	AMBIENT	400^{o}C	800^{o}C
OPC GGBFS 50/50	2.32	2.13	2.13
CSA GGBFS 50/50	2.24	2.09	2.04
Wet on wet OPC GGBFS 50/50	2.17	2.05	1.99
OPC GGBFS ACC	2.27	N/A	N/A
OPC CSA GGBFS	2.27	2.13	N/A

4.3 VISUAL ASSESMENT

The change in colour, cracks formation, and spalling occurrence were observed in the specimens due to their varying chemical compositions and exposure to different temperature factors, Table 17. The colour changes were noticeable, with some specimens turning darker or lighter than their original colour. Cracks also appeared on some of the specimens, which affected their integrity and strength. Furthermore, spalling, or the flaking of the specimen's surface layer, was also observed especially in mixes containing accelerator and blend of CSA, OPC and GGBFS, which could further affect the durability and reliability of the samples. All these observations were recorded and analysed as part of the ongoing research project to better understand the behaviour of these specimens under various conditions.

Table 17. Visual assessment of external damage of samples subjected to higher temperatures.

MIX	Ambient	400°C	800°C
OPC SLAG			
CSA SLAG			
Wet on wet OPC SLAG			

OPC SLAG ACC			
OPC CSA SLAG			

4.4 MICROSTRUCTURE AND CEHMCIAL COMPOSITION

Example SEM images and EDS spectra are shown in Figure 16, Figure 17, Figure 18, Figure 19, Figure 20, Figure 21, Figure 22, Figure 23 and in Figure 24 .

All shown figures relate only to mixes containing 50 wt.% of CEM I 42.5, 50 wt.% of GGBFS, which showed the best performance so far.

Generally, no cracking of the binder matrix was observed in any of these samples exposed to 400^{o}C but some were visible in samples exposed to 800^{o}C, Figure 12.

The Si/Al ratio tends to influence microstructure and performance of geopolymer concrete. Geopolymers consisting of fly ash with high Si/Al (≥ 5) revealed improved compressive strength and better volume stability in a condition of 1000^{o}C comparing to the geopolymer with same binder but lower Si/Al ratio (<2) [30].

Specimens containing 50% GGBFS as a cement replacement in ambient conditions and after exposure to temperature of 400^{o}C shown the ratio Si/Al ratio above 5, Table 18, 19. Over further testing those samples demonstrated superior performance. Specimens subjected to a temperature of 800 ^{o}C exhibited the lowest compressive strength and showed numerous cracks during the test, Table 20.

Concrete mix containing 50 wt.% of CEM 32.5 and 50 wt.% of GGBFS, melted when subjected to a temperature of 1100°C, Figure 13 and 14. The presence of iron oxide in the matrix has increased which was related to the formation of wüstite, Figure 15. It is a mineral form of iron (II) oxides [31].

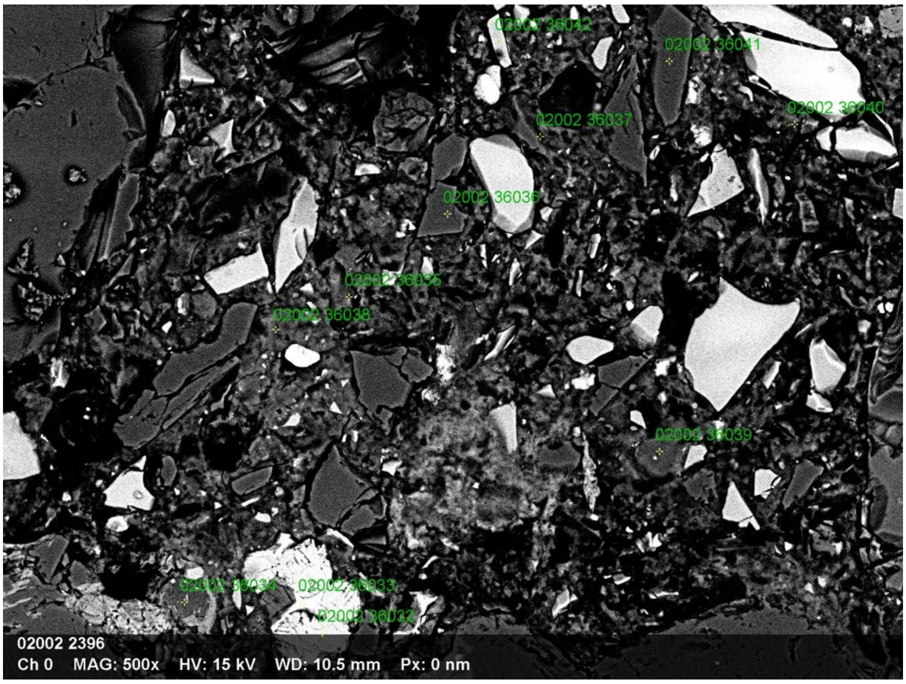


Figure 16 SEM image 50/50 CEM I 42,5 GGBFS ref. ambient conditions

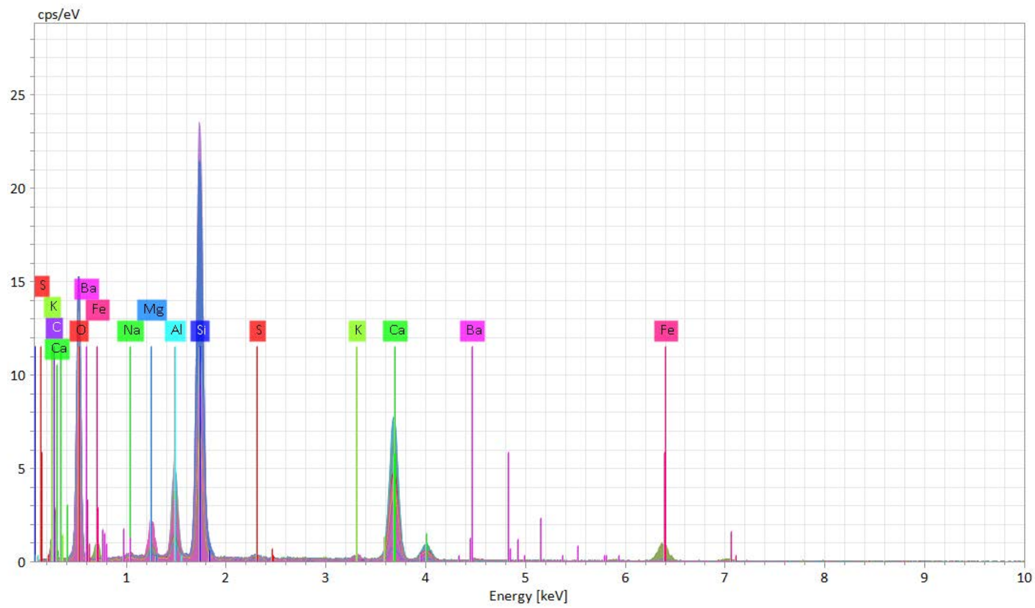


Figure 17 EDS analysis of chemical composition 50/50 CEM I 42,5 GGBFS ambient conditions

Table 19 Calculated average atomic ratios

Ratio:	**Si/Al**	**Ca/(Si+Al)**	**Ca/Si**
	5.59	0.66	0.78

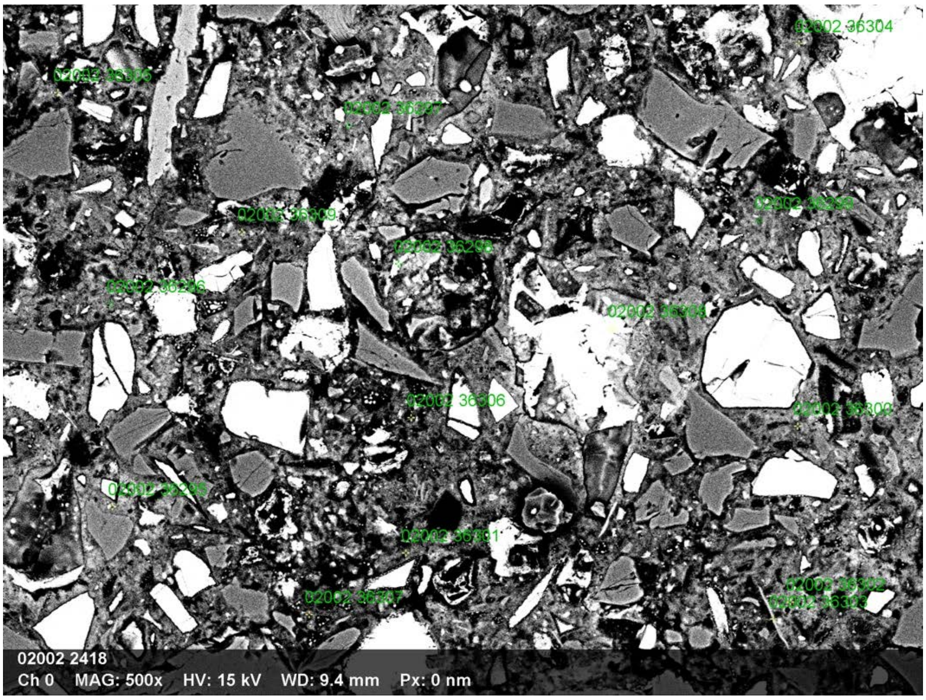


Figure 18 SEM image 50/50 CEM I 42,5 GGBFS 400ºC

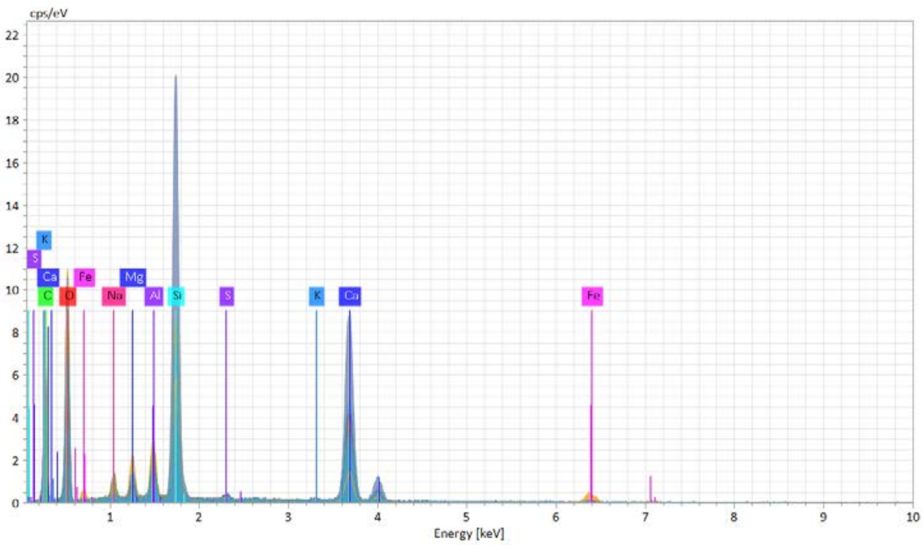


Figure 19 EDS analysis of chemical composition 50/50 CEM I 42,5 GGBFS 400ºC

Table 20 Calculated average atomic ratios

Ratio:	Si/Al	Ca/(Si+Al)	Ca/Si
	5.64	0.73	0.86

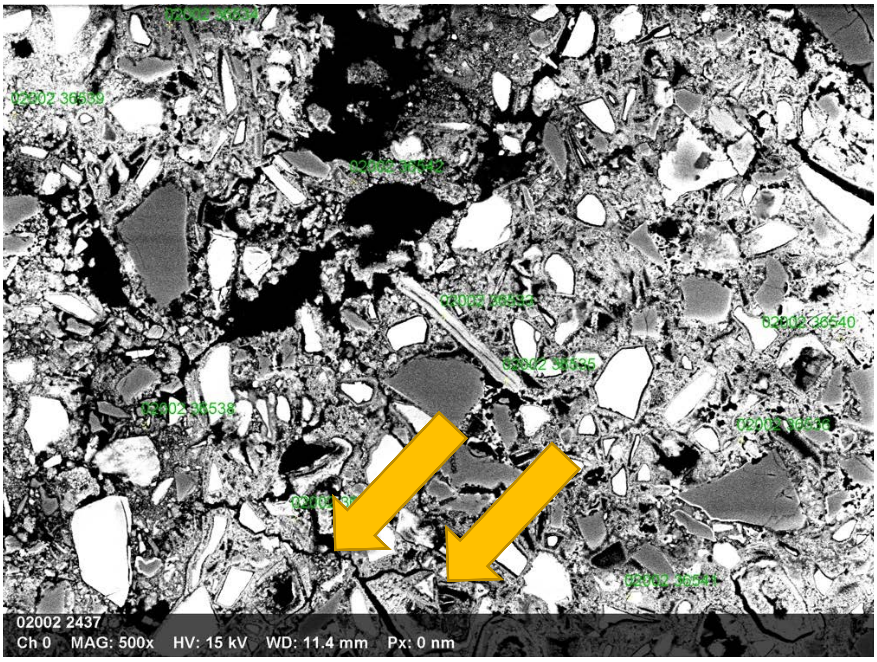


Figure 20 SEM image 50/50 CEM I 42,5 GGBFS 800^{o}C

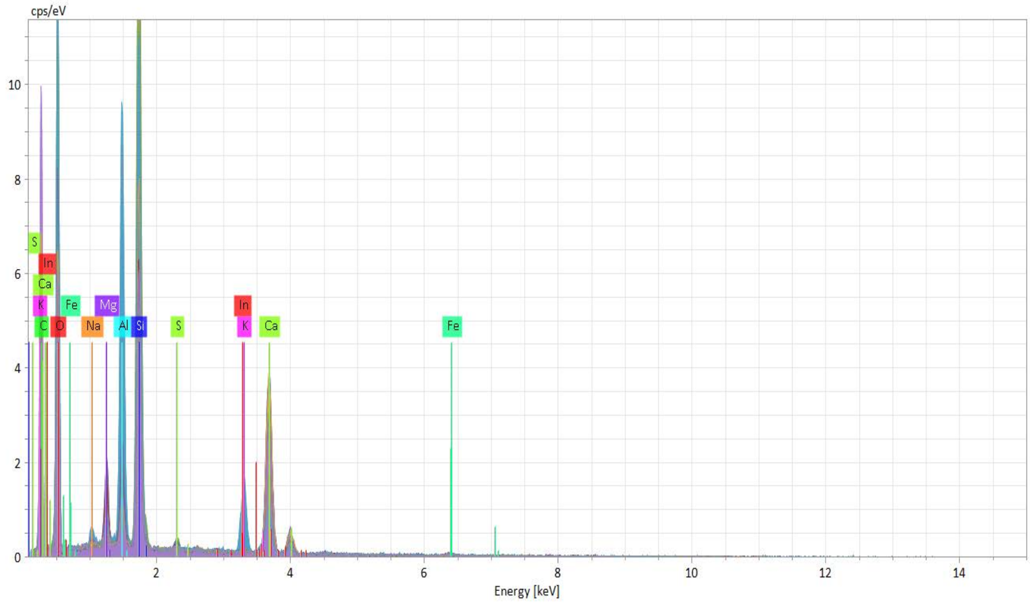


Figure 21 EDS analysis of chemical composition 50/50 CEM I 42,5 GGBFS 800^{o}C

Table 21 Calculated average atomic ratios

Ratio:	**Si/Al**	**Ca/SI+Al**	**Ca/Si**
	2,69	0,49	0,67

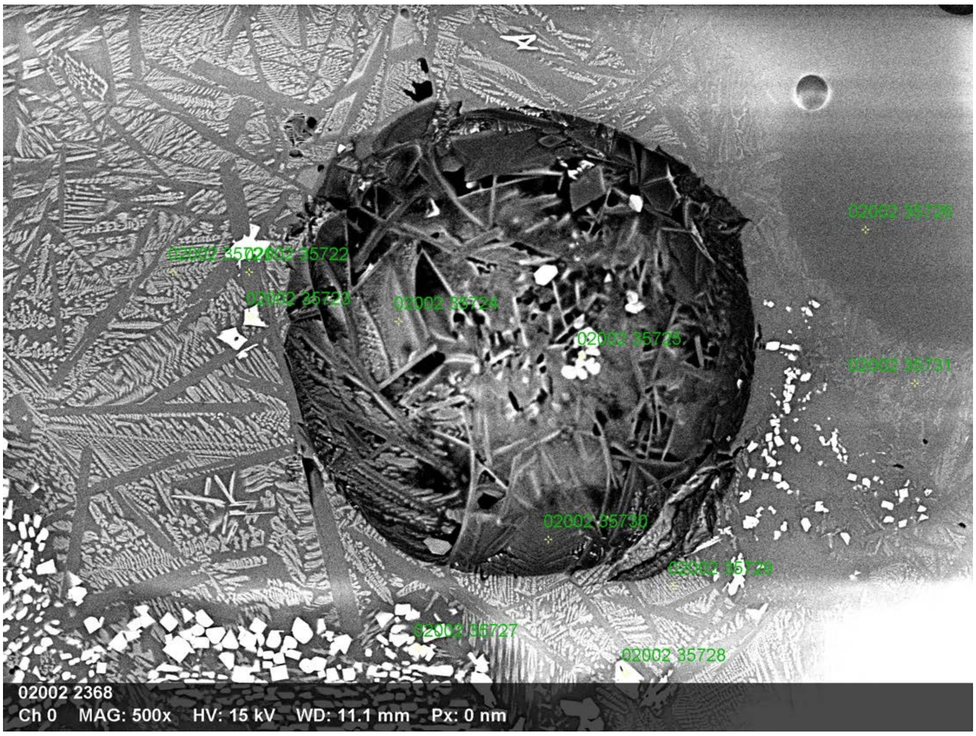


Figure 22 SEM image 50/50 CEM I 32,5 GGBFS 1100^{o}C

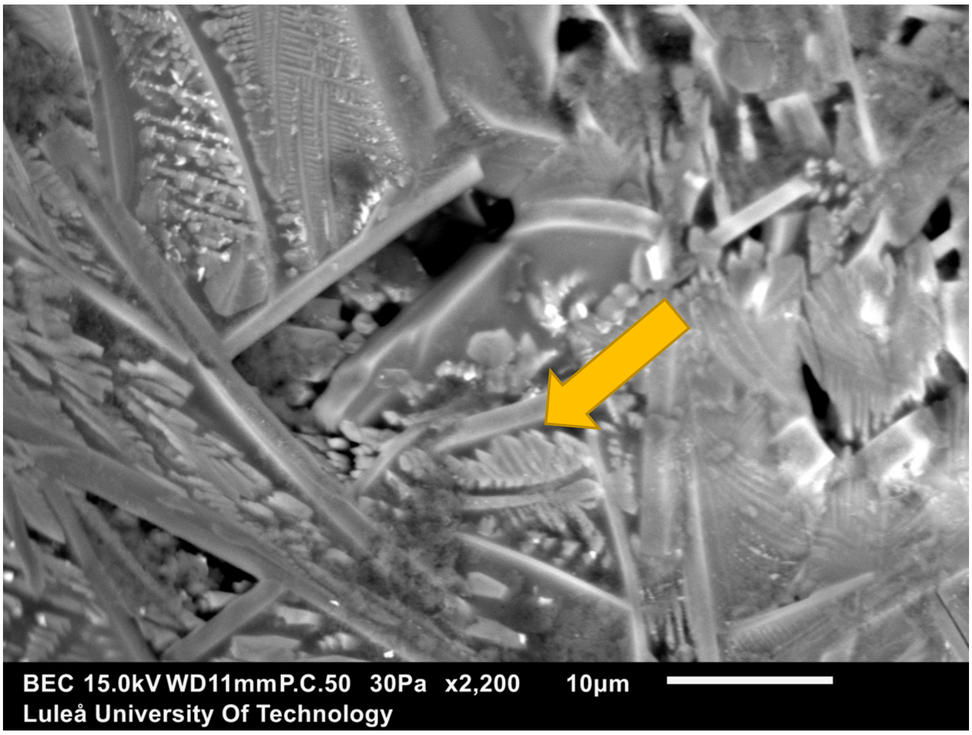


Figure 23 SEM image 50/50 CEM I 32,5 GGBFS 1100oC closure

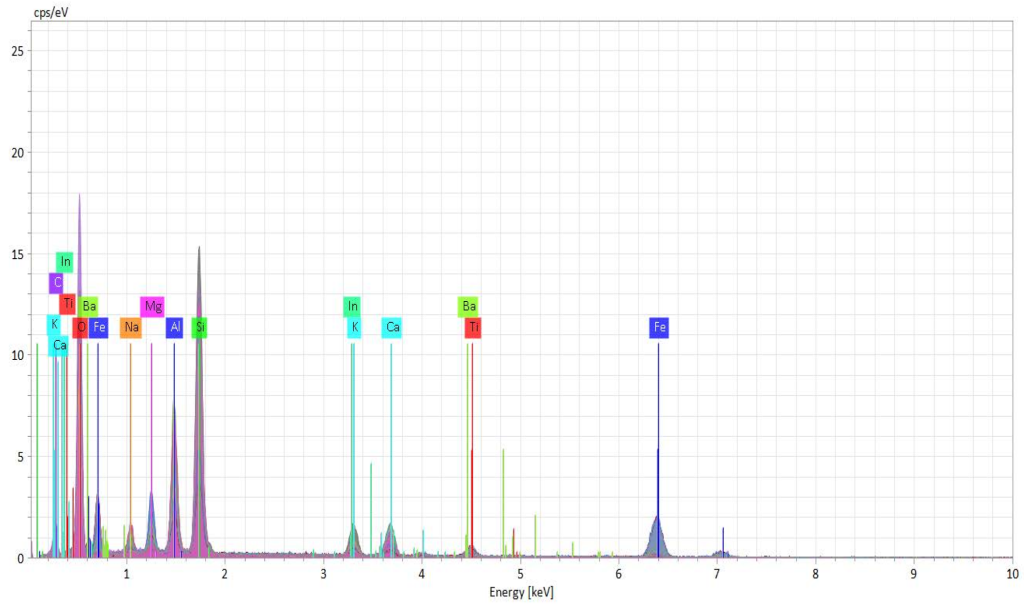


Figure 24 EDS analysis of chemical composition 50/50 CEM I 32,5 GGBFS 1100ºC

Table 22 Calculated average atomic ratios

Ratio:	**Si/Al**	**Ca/SI+Al**	**Ca/Si**
	2,42	0,12	0,17

5. CONCLUSIONS

The literature study showed that performance of concretes with full or partial replacement of Portland cement with SCMs, geopolymers, or CSA cements in exposure to fire is still questionable and required more in-depth testing.

The experimental study performed so far produced mixed results. Some of the studied binders performed very well but some showed potential problems that need further investigation. All can be summarised as follows:

Main findings

- Concretes containing up to 90 wt.% of BFS performed well in temperatures of up to 800°C.
- Exposure to 800°C resulted in chemical changes to some of the binders and caused formation of microcracks in the binder matrix.
- Larger amounts of accelerator caused explosive deterioration.
- The presence of air entraining admixture and superplasticizer added in amounts recommended by producents did not deteriorate performances of Eco concrete exposed to 800°C.
- CSA cements, when combined with OPC and BFS, showed variable results and should be studied more.
- The class strength of the used Portland cement impacted the strength loss, especially when exposed to 800°C.
- Exposure of eco concretes containing GGBFS to 1000 °C can result in formation of wüstite

Answers to research questions based on results obtained so far:

1. How do regular OPC based concretes perform when exposed to very high temperature in comparison with Eco concretes?

   OPC based specimen losses their weight and density, due to dehydration. The porosity is increased, and the loss of strength occurs. The decomposition of calcium hydroxide leads to microcracking and cracking formation and the further reduction of the OPC performance. Eco concrete remains more stable, the newly formed gel fill up pores, which make it more robust and compact. No major cracks and spalling occur.

2. What are the effects of exposure to high temperature/fire on binder matrix of eco concretes, i.e., phase composition, microstructure?

   Matrix of eco concrete becomes denser and more impact. Ratio Si/Al remains stable and consequently high after exposure to a temperature of 400ºC. No major cracks and spalling were observed.

3. What are the effects of exposure to high temperature/fire on spalling, mechanical properties and microcracking of eco concretes?

   Eco concrete containing GGBFS and OPC has performed superior comparing to OPC specimens. No spalling and wide cracks were observed. Mechanical properties were improved after exposure to 400ºC.

4. How chemical admixtures influence performance of eco concretes when exposed to high temperatures?

   The preliminary tests have shown that the chemical admixtures visibly interrupt performance of eco concrete subjected to elevated temperatures. It must be investigated more profoundly to provide reliable data.

6. FUTURE STUDY

The following investigations are planned for the second part of this PhD study

- Investigation of concretes based on alkali activated binders
- Determination of thermal conductivity, thermal expansion, and compressive strength.
- Further study on the effects of chemical admixtures
- Limited tests on larger specimens exposed to fire
- Effect of recycled concrete aggregates
- Modelling

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Appendix A

Table 23 CEM 32,5 with GGBFS

CEM 32,5				
	100	**90.10**	**50.50**	**10.90**
Ambient	29.3	39.7	37.7	24.8
400°C	36.7	46.3	47.3	25.6
800 °C	20.3	20.2	12.3	6.6

Table 24 CEM 42,5 with GGBFS

CEM 42,5				
	100	**90.10**	**50.50**	**10.90**
Ambient	47.6	47.2	37.5	24.8
400°C	68.7	62.6	62.7	40.0
800°C	28.1	19.3	24.9	13.8

Table 25 CEM 52,5 with GGBFS

CEM 52,5				
	100	**90.10**	**50.50**	**10.90**
Ambient	58.1	54.7	46.1	27.5
400 °C	59.3	57.3	62.2	39.7
800 °C	25.6	24.7	23.5	12.3

Table 26 Wet on Wet O: CEM 32,5 GGBFS 50/50 I:CEM 52,5 GGBFS 50/50

50/50 O: CEM 32,5 I: CEM 52,5 SLAG P 0,36%

Ambient	48.6
400°C	42.5
800°C	14.8

Table 27 Accelerator 10%

50/50 CEM 42,5 SLAG ACC 10%	
Ambient	79.70
400°C	N/A
800°C	N/A

Table 28 Accelerator 4%

50/50 CEM 42,5SLAG ACC 4%	
Ambient	50.00
400 °C	65.20
800 °C	N/A

Table 29 Air entraining admixture 0,1%

50/50 CEM 42,5 SLAG AE 0,1%	
Ambient	59.5
400°C	70.1
800°C	29.1

Table 30 Plasticizer 0,5%

50/50 CEM 42,5 P 0,5%	
Ambient	53.00

400°C	59.95
800°C	22.00

Table 31 CSA

CSA	
Ambient	62.90
400°C	41.30
800°C	29.47

Table 32 50/50 CSA GGBFS

50/50 CSA GGBFS	
Ambient	26.5
400°C	17.93
800°C	10.1

Table 33 50/25/25 OPC CSA GGBFS

50/25/25 OPC CSA GGBFS	
Ambient	54.97
400°C	35.47
800°C	SPALLING

Table 34 25/50/25 OPC CSA GGBFS

25/50/25 OPC CSA GGBFS	
Ambient	53.63
400°C	17.23
800°C	SPALLING

Table 35 25/25/25 OPC CSA SLAG

25/25/50 OPC CSA GGBFS	
Ambient	35.07
400ºC	21.43
800ºC	SPALLING

Table 36 CEM 42,5 with SKANSKA FILLER

CEM 42,5 with Natural FILLER	
Ambient	55.00
400ºC	54.00
800ºC	25.43

Table 37 50/50 CEM 42,5 GGBFS with SKANSKA FILLER

50/50 CEM 42,5 GGBFS with Natural FILLER	
Ambient	35.07
400ºC	21.43
800ºC	SPALLING