

Utilization of alkali activated concrete in concrete revetment products

Tom van Rijswijk



Utilization of alkali activated concrete in concrete revetment products

by

T. van Rijswijk

To obtain the degree of Master of Science
at the Delft University of Technology
to be defended publicly on Wednesday November 16, 2022 at 15:30h

Student number:	4345878
Project duration:	November 9, 2021 - November 16, 2022
Thesis committee:	Dr. G. Ye, TU Delft (chair)
	Dr. H. Dong, TU Delft (daily supervisor)
	Prof. Dr. Ir. H.M. Jonkers, TU Delft
	Dr. Ing. M.Z. Voorendt, TU Delft
	Ir. R. Jacobs, industry
	Ir. B. van Zwicht, industry

An electronic version of this thesis is available at <http://repository.tudelft.nl/>

Picture on cover page by Tom van Rijswijk

“Take the best that exists and make it better”
— Henry Royce

Preface

This thesis represents my research for utilization of alkali activated concrete in concrete revetment products. It is written in the frame of finishing my master Structural Engineering with track Hydraulic Structures at the TU Delft, Netherlands. From November 9th, 2021 until November 16th, 2022 I have been working on research and writing this report.

This research is carried out in collaboration with Holcim, for which I am grateful. I started with an internship, where I researched alternative concrete types that seem suitable for concrete revetment products whilst also lowering the environmental footprint of the products. After concluding this internship, alkaline activated concrete was determined as potential alternative, suitable for further research. The research is successful and I am proud to present all results.

I am fortunate to have received support from many people and want to share my gratitude. First of all, a big thank you towards Dr. Guang Ye and Dr. Hua Dong for their great support, discussions and critique. I also want to thank Jan Wagner and Bart van Zwicht from Holcim for making this research possible and for providing support, logistical operations and for provision of resources. Without them, this thesis would not have been possible.

From the committee, I would also like to thank Henk Jonkers and Mark Voorendt for their support. They helped with the report structure and provided great advices.

Outside of the committee there are also other mentions that I want to share. First of all, the technical staff from TU Delft and industry. Sjaak Vermeij (Holcim), Pascal Meerenburg (Holcim), Maiko van Leeuwen (TU Delft), Ton Blom (TU Delft), Arjan Thijssen (TU Delft), John van den Berg (TU Delft) and Ron Penners (TU Delft) all helped me with logistical operations, providing experimental equipment and explained the operation of equipment. A special thank you towards Teun Noordam (Holcim) as he was the person that made the internship and thus the thesis at Holcim possible. He has supported me throughout the research and helped with the practical approach for the concrete phase.

Also a big thank you to all students that have been with me during all the years at TU Delft. There are so many names which I can mention, but to each and every one of them, I want to share my gratitude. The ride to become a civil engineer is a rollercoaster, but all of you helped me become a better engineer. Thank you.

My family is also very important to me. Not only during this thesis, but throughout my entire TU Delft career. Thank you mom and dad, for providing support, love and advices. You both helped me through the most difficult part of my study and thesis and I honestly am proud to have such great parents. Also thank you Annika, my girlfriend, for all support and motivating words during the research. Your love and kindness helps me to improve myself every time and I am very proud to have you as my girlfriend. Also a big thank you to Karl for reading and analyzing the report. Last but not least the rest of my family and friends, who are always kind and supportive, whom I can always count on...

T. van Rijswijk
Alphen aan den Rijn, November 2022

Summary

Due to climate change, the concrete industry is searching for different options to lower the environmental footprint. Alkali activated concrete is seen as a possible contender. The goal of this research is to evaluate the extend to which ground granulated blast furnace slag and calcined clay-based alkali activated concrete can be utilized in concrete revetment applications with equal performance in freeze-thaw and sulfate attack resistance and lower environmental impact compared to blast-furnace slag cement concrete.

This research uses a combination of ground granulated blast furnace slag and calcined clay as precursor blend which is combined with sodium silicate and sodium hydroxide activator solution.

An upscaling methodology is used such that differences in material behavior between each upscaling step (paste, mortar, concrete) is explained such that the concrete is more efficiently optimized. This research also uses the Taguchi method for mix design development, which reduces the number of mix designs whilst obtaining the required information and detail to determine performance trends. These connect the performance of the specimens to the influence each mix design parameter and its values has on this performance.

The alkali activated concrete mix designs manage to develop good compressive strength, where both mix designs reach over 12 MPa after 24 hours of ambient curing and over 37 MPa after 28 days of ambient curing. It fulfills the requirement for revetment products in terms of compressive strength and also outperforms cement concrete.

The water absorption of the concrete specimens is on the high side. It does not fulfill the requirement, but the high value is attributed to non-optimal compaction as the concrete phase uses a special vibration and pressure compaction machine. Even though it is the best method for compaction of earth moist concrete, it does not represent the compaction enabled in a factory process as the reference concrete, provided by the industry, also fails to meet this requirement.

Freeze-thaw performance of the alkali activated mortar is sufficient as 5 out of 9 mortar mix designs meet the requirement provided by the used CDF test. Inclusion of calcined clay in the precursor blend and the effect of the modulus clearly results in worse freeze-thaw performance due to more difficult compaction, higher absorption characteristics and more interconnected pores resulting in more scaling as the de-icing solution can penetrate further in the specimens. Freeze-thaw of the alkali activated concrete is insufficient with a large amount of scaling at the end of the test. However, this is also true for the reference concrete, where part of the insufficient performance is again attributed to compaction of the specimens. However, the retarding scaling behavior of alkali activated concrete indicates very good performance on the long term.

Sulfate attack performance is only tested at mortar level and during the short testing period of 14 days, where specimens are exposed to 10 % MgSO_4 solution, no degradation is visible in terms of scaling and compressive strength. Slight formation of ettringite is observed, meaning that it is recommended to extend the testing period in future research on this topic.

Life cycle analysis is performed on product level and does not consider service life of the product and recyclability. The alkali activated concrete mix designs show the potential to reduce the milieukostenindicator or MKI with 48 % to € 5,72 and reduce the carbon dioxide emissions with 63 % towards 48 kg per m^3 produced concrete. These are significant reductions, but for full validation of environmental footprint performance of alkali activated concrete, service life and recyclability should also be involved in the calculations.

The research proves that alkali activated concrete can be a very interesting alternative for cement concrete in concrete revetment products where the saturated Dutch ground granulated blast furnace slag market can be alleviated partially by substitution of calcined clay. However, to apply the alkali activated concrete in revetment products with confidence, more research should be carried out, especially with regards to durability.

The service life should be proven by testing more durability parameters and changing the testing method, pre-treatment and duration for both freeze-thaw and sulfate attack. Recyclability

should also be considered to prove the reduction in environmental footprint. Lastly, a development program to further enhance the maturity of the material should be produced where optimal conditions for alkali activated concrete are considered for each durability test as alkali activated concrete clearly has different degradation mechanisms compared to cement concrete.

Contents

Preface	v
Summary.....	vii
List of Abbreviations	xi
1. Introduction	1
1.1. Motivation for this research	2
1.2. Problem analysis and research question	3
1.3. Delimitations and Scope	5
1.4. Approach and reading guide	7
2. Literature review: the relationship between materials and properties	11
2.1. A brief alkali-activated concrete history	12
2.2. Alkali activated concrete components	12
2.3. Alkali activated reaction kinetics and microstructure formation	19
2.4. Characteristics of alkali activated freeze-thaw resistance	22
2.5. Characteristics of alkali activated sulfate attack resistance	28
2.6. Summary and conclusion	31
3. Research methodologies	33
3.1. Research methodology: Upscaling.....	34
3.2. Mix design methodology: The Taguchi method	36
3.3. Conclusion.....	40
4. Experimental setup and materials.....	41
4.1. Experimental setup.....	42
4.1.1. Aggregate shape	42
4.1.2. Aggregate particle size distribution.....	42
4.1.3. Aggregate water absorption and specific gravity.....	42
4.1.4. Precursor and activator composition	42
4.1.5. Workability	43
4.1.6. Flexural strength.....	43
4.1.7. Compressive strength.....	43
4.1.8. Water absorption	44
4.1.9. Surface water absorption.....	44
4.1.10. Freeze-thaw resistance	44
4.1.11. Sulfate attack resistance.....	46
4.2. Materials	48
4.2.1. Aggregate shape	48
4.2.2. Aggregate particle size distribution.....	49
4.2.3. Aggregate water absorption	50
4.2.4. Precursor and activator composition	51
4.3. Conclusion.....	52
5. Mix design	53
5.1. Paste phase mix design	54
5.2. Mortar phase mix designs	56
5.3. Concrete phase mix designs	57
5.4. Conclusion.....	57
6. Experimental results and analysis	59
6.1. Paste phase	60

6.1.1. Workability	60
6.1.2. Flexural strength.....	61
6.1.3. Compressive strength.....	64
6.1.4. Paste phase conclusion.....	67
6.2. Mortar phase	67
6.2.1. Workability.....	67
6.2.2. Flexural strength.....	69
6.2.3. Compressive strength.....	72
6.2.4. Water absorption	74
6.2.5. Freeze-thaw.....	76
6.2.6. Sulfate attack resistance	82
6.2.7. Mortar phase conclusion	84
6.3. Concrete phase	86
6.3.1. Workability.....	86
6.3.2. Compressive strength.....	87
6.3.3. Water absorption	90
6.3.4. Freeze-thaw resistance	90
6.3.5. Concrete phase conclusion	94
6.4. Conclusion.....	94
7. Life cycle analysis	97
7.1. LCA reference concrete.....	99
7.2. LCA alkali activated low MKI concrete	103
7.3. LCA alkali activated performance concrete	107
7.4. LCA comparison and conclusion.....	111
8. Discussion, conclusions and recommendations.....	113
8.1. Discussion	114
8.1.1. Added value of methodology	114
8.1.2. Relation of research results and literature	115
8.1.3. Limitations of this research	115
8.2. Conclusions	117
8.3. Recommendations	120
A. Requirements for concrete revetment application	123
B. Alkali activated paste trials	127
C. Alkali activated paste mix designs	139
D. Alkali activated mortar mix designs	145
E. Alkali activated concrete phase trials	151
F. Alkali activated concrete mix designs	157
G. Reference mix design data (confidential).....	159
H. Full experimental setup	161
I. Additional LCA data.....	181
References.....	189

List of Abbreviations

Materials

SS	Sodium silicate
SH	Sodium hydroxide
BFS	Blast Furnace Slag
GGBFS	Ground Granulated Blast Furnace Slag
CC	Calcined Clay
OPC	Ordinary Portland Cement
FA	Fly Ash
MK	MetaKaolin

Alkali activated property terms

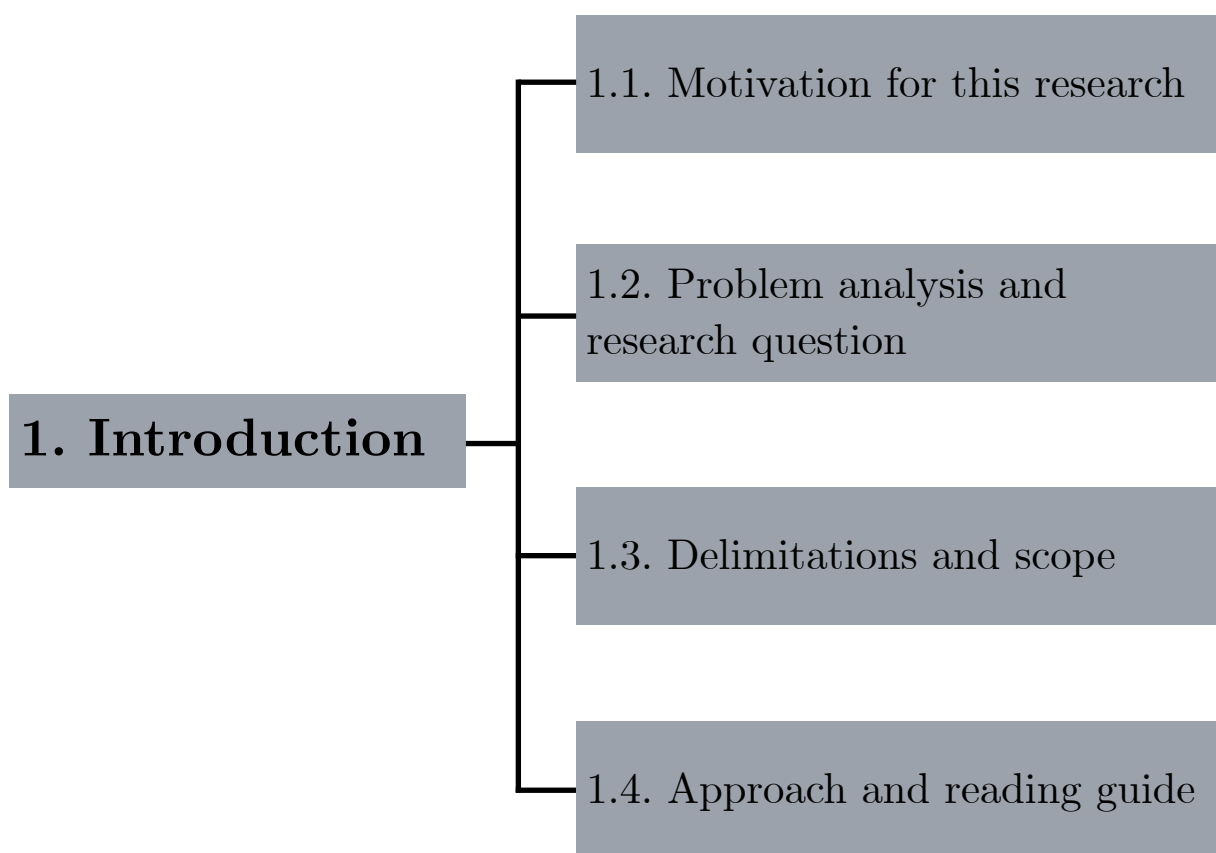
W/B	Water-to-Binder ratio
SS/SH	Sodium silicate to Sodium hydroxide ratio
Ms	Modulus
Al/Bi	Alkaline liquid to Binder ratio
M NaOH	Molarity of Sodium hydroxide

Experimental terms

FT	Freeze-Thaw
SA	Sulfate Attack
SEM	Scanning Electron Microscope
XRD	X-Ray Diffraction
XRF	X-Ray Fluorescence
PSD	Particle Size Distribution
WA	Water Absorption
MIP	Mercury Intrusion Porosimetry

Other terms

EPD	Environmental Product Declaration
LCA	Life Cycle Analysis
MKI	Milieu Kosten Indicator (EN: Environmental Cost Indicator)



This research aims to alleviate environmental stress by application of alkali activated concrete in concrete revetment products. This chapter provides the motivation for the research in section 1.1. Section 1.2 continues with the problem analysis and research question, followed by the delimitations and scope in section 1.3. The used approach and a reading guide can be found in section 1.4.

1.1. Motivation for this research

Climate change

Climate change is currently a very hot topic, but its history is relatively short. Svante Arrhenius (1896) is seen as one of the first people to notice climate change in a time where industrialization was at its peak. He concluded that a lowering of carbon dioxide or CO₂ results in temperature drops resulting in massive cooling (Barral, 2019). This drop is a necessity, as greenhouse gases provoke melting of ice sheets causing sea-water level increase. A global increase of temperature also causes decline of coral reefs and decrease in biodiversity (WWF, s.d.). Thus, climate change regimes started to form awareness of the worldwide problem that kept and keeps increasing.

The Brundtland Commission report, *Our common future*, is considered to be the start of sustainable development and temperature reduction in political, public and industrial sectors (Duurzame ontwikkeling, 2021). The Netherlands developed the “Klimaatakkoord” or climate agreement, which contains measures to reduce environmental impact by 49 % in 2030 relative to 1990, and to reduce heating of the planet to 1.5 - 2 °C by 2050 as also described in the Paris agreement (Rijksoverheid, 2019), (Klimaatakkoord, 2019).

The role of concrete in climate change

The industrial sector, where Civil Engineering practices are based in, is responsible for approximately 20 % of the total global greenhouse gas emissions (EPA, s.d.). Zooming in specifically on the concrete and cement industry, it is stated that it accounts for 5 - 8 % of the global anthropogenic (human caused) CO₂ emissions, but is also considered to have the largest energy saving potential (28 - 33 %) out of all other industries (Kajaste, & Hurme, s.d.).

In coastal engineering applications and especially dike revetments, concrete is often used for protection due to its consistency and space efficiency when compared to loose rock (Mangor, 2020). As concrete is the most used material for revetment products, a reduction of its environmental impact directly alleviates environmental stress. For the Dutch industry, it is estimated that 6.5 % of all produced concrete is utilized in water-related structures under which revetments, thus transitioning to a lower-environmental-impact concrete really makes a difference (Kramer, 2020). This research aims to reduce the environmental impact by completely removing cement from the concrete mixture and substituting it by alkali activated concrete, seen as environmental-friendly concrete, on product level (Assi, Carter, Deaver, Anay, & Ziehl, 2018).

The Dutch market utilizes approximately 5200 kiloton of cement annually, of which 55 to 60 % consists of blast furnace cement (Kramer, 2020). As the market is saturated in terms of the use of ground granulated blast furnace slag (all produced ground granulated blast furnace slag is used in the industry) development of alkali activated concrete consisting of only ground granulated blast furnace slag does not seem logical. However, the alkali activated concrete developed in this research strives to be a good replacement to blast furnace slag cement, resulting in no significant change in available ground granulated blast furnace slag for alkali activated concrete production as calcined clay is substituted in the precursor blend to reduce the ground granulated blast furnace slag content (SGS Intron, 2020).

Relevance of this research

Alkali activated concrete is currently being researched and seen as environmental-friendly concrete as it incorporates readily found products, like ground granulated blast furnace slag (GGBFS), and combines them in a composition suitable for specific applications. However, there is no traceable research performed for the suitability of alkali activated concrete in a composition suitable for dry-cast concrete revetment products. For revetment products, especially a good resistance to freeze-thaw and sulfate attack is important due to the harsh environment the products are situated in. Good resistance to these durability factors also contributes to longer lifespan of the material. Combined with the goal of lowering the environmental impact by making use of industrial by-

products as main binder components in alkali activated concrete and with dry-cast production methodology, this research expands application possibilities for utilization of alkali activated concrete.

1.2. Problem analysis and research question

Problem analysis

Alkali activated concrete is currently not applied often in the industry, but the numbers are increasing. It is used as high strength concrete, lightweight concrete, self compacting concrete and even more. Alkali activated concrete replaces the binder of cement-based concrete by introduction of precursors and activators. The precursors are aluminosilicate or calcium rich and chemically react in an alkaline environment, forming a hardened binder (Provis, 2018). The difference with cement is that precursors are also often used as SCMs (Supplementary Cementitious Materials) like blast furnace slag (BFS) and fly ash (FA). These precursors react with activators, like sodium hydroxide and sodium silicate, which form the alkaline environment necessary for dissolution of the precursor, which results in formation of a hardened binder.

However, alkaline activated concrete is a construction material in development and some challenges still exist which determine what materials the concrete should be produced with, what durability factors are especially important for revetments and what considerations are necessary for the production of the final product.

The first challenge comprises local availability of materials. Materials should be locally available to prevent a large environmental emission footprint, as transportation of raw materials increases its environmental footprint. For alkaline activated concrete, especially fly-ash (FA), metakaolin (MK), ground granulated blast furnace slag (GGBFS) and calcined clay (CC) are popular precursors due to wide availability and suitable properties in terms of performance and durability. With the increase of closures of coal power plants, fly ash, which is a popular precursor for alkali activated concrete, is not considered for this application. Metakaolin is also not considered, as its origin is mostly in China, which is very far away, resulting in large transportation distances which significantly increases environmental impact. From other candidates, ground granulated blast furnace slag, occupied mostly in blast furnace cement, and the possibility of calcined clay, still abundantly available, are the most interesting for application of alkali activated concrete in the Netherlands and form the precursor blend in this report.

The second challenge comprises durability and degradation of the concrete. Dike revetments in the Netherlands and large parts of Europe are situated in or around the sea and rivers. The salt water combined with possible cold climates require high durability and low degradation. Therefore, freeze-thaw resistance is a very important durability parameter for this specific application. Finding the mechanisms that influence freezing and thawing performance is important to expand the lifespan of alkaline activated concrete in harsh environments and is also one of the main topics in this research. Another important durability factor is sulfate attack resistance as the concrete dike revetments are put on top of soil which may introduce salt crystallization or chemical sulfate. These chemical constituents are also present in the seawater and groundwaters (Detwiler, 2021). Both of these durability factors influence the lifespan of the concrete which also directly influences environmental impact, therefore the concrete should be sufficiently resistant to sulfate attack.

The third challenge consists of production considerations. A lot of smaller dike revetment products are produced with dry-cast concrete. Dry-cast concrete production allows for immediate demolding and very fast production speeds. This requires the concrete to be earth-moist in terms of workability. A lot of research on alkali activated concrete is focussed on regular or self compacting concrete, while earth moist concrete researches can only be found for the production of bricks. As

the revetment products have different requirements than bricks, it is a challenge to adapt both research fields towards a good performing alkali activated concrete suitable for dry-cast production.

Besides these challenges, the alkali activated concrete should also comply with a list of requirements. These requirements are formed from three perspectives:

1. Requirements from Dutch concrete revetment norms (NEN).
2. Requirements from the production process.
3. Requirements with regards to (local) availability of the materials.

Table 1.1 below contains the material selection and Table 1.2 summarizes the production and performance requirements. For a more in detail description of the formation of these requirements, refer to appendix A.

Table 1.1 - Overview of material selection

Category	Selection type	Value
Materials	Maximum aggregate size	10 mm
	Precursor types	GGBFS & CC
	Activator types	Sodium hydroxide & sodium silicate

Table 1.2 - Overview of production and performance requirements

Category	Requirement	Value
Production	Curing temperature	20 - 30 °C
	Curing duration	24h, then outside
	Curing humidity	80 - 95 %
	Consistency class (EN 12350-4)	C1 (earth moist concrete)
	Environmental class	XF4
	Density (For stability of products on dike)	> 2300 kg/m ³
	Compressive strength after 24h (Packaging of products after curing)	≥ 12 MPa
Performance	Compressive strength after 28d (NEN-EN 12390-3:2019(E))	≥ 37 MPa
	Freeze-thaw resistance (NVN-CEN/TS 12390-9:2016, CDF test)	Mass loss ≤ 1.5 kg/m ² after 28 FT cycles.
	Water absorption (NEN-EN1338:2003, appendix E)	Total water absorption ≤ 6 % by mass
	Environmental cost indicator	< € 11,-/m ³ and < 128kg CO ₂ /m ³

Research question

The combination of the motivation and problem analysis results in the following research question central in this research:

“To what extend can dry-cast slag and calcined clay-based alkali activated concrete be utilized in concrete revetment applications with equal performance in freeze-thaw and sulfate attack resistance and lower environmental impact compared to blast-furnace slag cement concrete?”

1.3. Delimitations and Scope

This research aims to alleviate environmental stress of concrete revetment products by using alkali activated concrete instead of blast furnace cement concrete. However, since this is a research with a finite duration of 12 months, it is requisite to provide the framework in which this research is carried out. To do this, a compact state of the art is provided after which the delimitations and scope of this research are elaborated.

Overview

Very useful information has been obtained from the scientometric review from Zakka, Abdul Shukor Lim, & Chau Khun (2021) and from analyzing data from Scopus. In Figure 1.1 below, a significant increase in documents researching alkali activated concrete is visible. It is interesting that most research is being carried out in India, Australia and China. Table 1.3 below provides a brief overview of the keywords of alkali activated concrete researches in these three countries. Out of the 3473 results, 224 (6.4 %) keywords contain GGBFS, 103 (3 %) keywords contain carbon footprint, 65 (1.9 %) keywords contain ambient curing and 327 (9.4 %) of the keywords contain durability. Almost no keywords with regards to industrial application can be found.

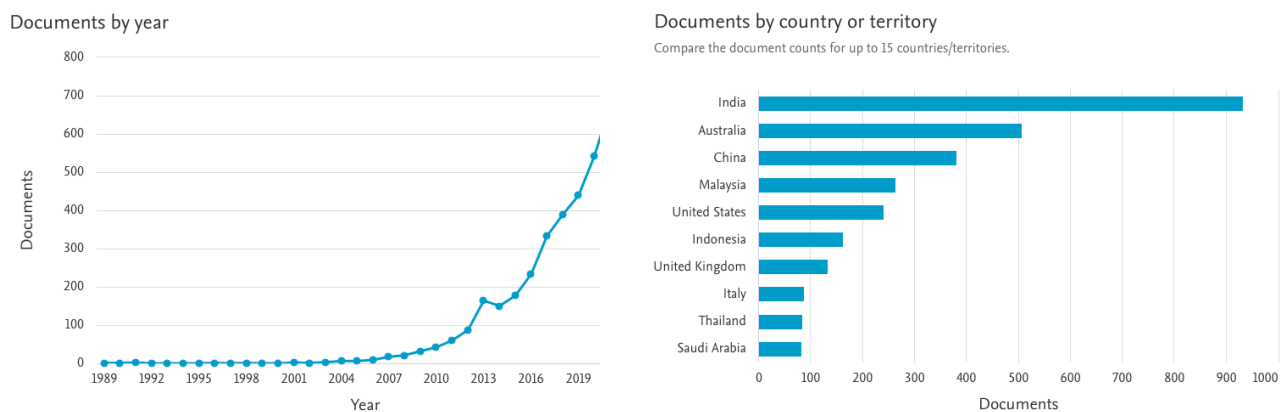


Figure 1.1 - Alkali activated concrete research documents per year and per country

Table 1.3 - Most used keywords in top three alkali activated concrete research countries

Keyword and rank	India	Australia	China
#1	Fly ash (464)	Fly ash (265)	Compressive strength (177)
#2	Compressive strength (358)	Compressive strength (227)	Fly Ash (150)
#3	Slags (174)	Slags (120)	Slags (100)

Delimitations - precursors and activators

This research uses a combination of ground granulated blast furnace slag and calcined clay as precursors. Ground granulated blast furnace slag is often used in the Dutch cement industry in the form of CEM III cement. Nearly all available ground granulated blast furnace slag is occupied in the Dutch industry. This implies that a shift towards alkali activated concrete with ground granulated blast furnace slag is only possible when its availability increases. One of the possibilities to increase this availability is to replace CEM III with alkali activated concrete.

Slag-based alkali activated concrete enables ambient temperature curing and has fast strength development when combined with sodium hydroxide and sodium silicate as activators. These components together form the precursor and activators in this research. However, substitution of calcined clay as precursor and the effects on strength development and durability of alkali activated concrete is investigated as well, as calcined clay is still abundantly available and can alleviate the saturated ground granulated blast furnace slag market if it can be applied in alkali activated concrete.

The research does not consider other precursor or activator types with the following reasoning:

- Fly ash: Fly ash-based alkali activated concrete has comparable properties of Portland Cement-based concrete, but the concrete strength development is slow due to the low amount of calcium oxide within the fly ash. Fly ash alkali activated concrete also needs high temperature curing as curing temperature has large influence on strength development. Often fly ash based alkali activated concrete is blended with other precursors like metakaolin, slag, volcanic ash, rice husk ash, oil palm ash etc. to address the issues of fly ash only alkali activated concrete. Besides the high required curing temperature and slow strength development, closure of coal power plants yields less available fly ash for production of concrete and therefore it is not considered as suitable precursor in this research (Betonakkoord, s.d.).
- Sodium carbonate and sodium aluminate are not considered as activator type due to its low reactivity and low strength development rate (Bakharev et. al, 1998).

Delimitations - Durability

Within the coastal environment, especially freeze-thaw resistance and sulfate attack are very important. Most researches that investigated both of these parameters conclude that freeze-thaw resistance can be sufficient as well as sulfate attack resistance. Freeze-thaw resistance is tested at mortar and concrete scale and sulfate attack is tested only at mortar scale and only with MgSO_4 solution.

Delimitations - Dry-cast concrete

Revetment products are produced at very fast rate with immediate demolding after compaction. Researches that focus on alkali activated concrete use wet-cast concrete, but this research only focuses on dry-cast concrete due to the specific production procedure of revetment products.

Scope

To summarize the above, this research focuses on the below stated components which combined form the scope:

- Alkali activated concrete with sodium hydroxide and sodium silicate as activator.
- Alkali activated concrete with ground granulated blast furnace slag and substitution of calcined clay as precursor.
- Dry cast production method, implying the concrete should be designed as “earth moist”.
- Ambient curing (20 - 30 °C and 80 - 100 % humidity).
- Compressive strength, freeze-thaw resistance and sulfate attack resistance as durability parameters for coastal environments.
- Environmental impact comparison is only performed on product scale and does not include the entire lifecycle.

The above stated scope, combined with the experimental results from this research, is considered to be sufficient to provide good understanding of the suitability of alkali activated concrete as replacement of slag based cement concrete in the Dutch industry for concrete revetment products.

1.4. Approach and reading guide

The full approach in the form of execution steps can be found below.

Step 1. Deriving requirements

Using the NEN, or Stichting Koninklijk Nederlands Normalisatie Instituut, a list of performance requirements for concrete revetment products is formed. Along with industry-related requirements and requirements based on local availability of materials, the total list of requirements is formed.

Step 2. Determination of the relationship between materials and properties (literature review)

With the requirements and research question known, the relationship between the materials and mechanical/durability properties are researched via literature review. First of all, a basic sense of alkali activated concrete is provided by describing a brief alkali activated concrete history and the components involved in alkali activated concrete. Secondly, the reaction kinetics and microstructure formation (how can an alkali activated concrete develop strength and what does this development depend on) are explained. Lastly, a state-of-the-art literature review is provided which aims to find the characteristics of alkali activated concrete on freeze-thaw and sulfate attack performance. With all of the above in mind, the to be designed mix designs can already be aimed at possible preferences of the material with regards to its developed properties, resulting in possibly immediately well performing mix designs, such that there is no need for iteration of mix design composition.

Step 3. The used methodology

Before the mix designs are developed, the methodology that is used in this research should be known. In this research, an upscaling method is used as experimental methodology. To find a suitable concrete mix design with low environmental impact the upscaling is a strong method. Starting with paste, one can determine basic mechanical properties of the paste without using too many materials. If these properties are sufficient, sand can be added to turn the paste into a mortar. During the mortar phase, the effects of upscaling on mechanical properties can be determined to see if it is still satisfactory. From the results of the paste and mortar phase, it is clear which mix designs are most promising which can then directly be re-calculated into a concrete mix design. By using the same mix designs over and over, possible changes in properties are more easily explained such that the concrete can be more easily optimized for the concrete revetment requirements.

The Taguchi method is used as mix design methodology in both the paste- and mortar phase to determine the influence of mix design parameters on mechanical performance with the development of trends. These trends describe the relation between mechanical performance and mix design parameters. By determining what factors influence a durability property of alkali activated paste/mortar, Taguchi can be used to efficiently determine the optimal composition of a concrete mixture by researching many mix design parameters with minimal amount of mix designs, resulting in a reduction of time, amount of materials and allowing more experiments to be conducted.

Step 4. Experimental setup and used materials

For the final performance results of the paste/mortar/concrete, experiments are used. In order to precisely describe the execution of all of these experiments, the experimental setup is formed. For each experiment, the scope and method are provided so that all experimental work can accurately

be performed or imitated by other researchers. In the case an experimental setup contains an adaptation of the standard, the complete procedural steps and the visualization of the setup are also included.

Before the mix design composition is determined, the raw materials used in the research are characterized.

Step 5. Mix design

After the basic properties of all materials are known, the mix designs are developed. The development of the mix designs incorporates the combination of the knowledge of the literature review (reaction kinetics, microstructure formation, freeze-thaw resistance trends and sulfate attack resistance trends) and the basic material properties.

For the paste- and mortar phase mix designs, the Taguchi method is used to compose the mix designs based on several mix design parameters and variation within these parameters. The concrete mix design uses the results of the entire paste- and mortar phase to compose mixtures which are in compliance with the full set of requirements.

Step 6. Experimental results and analysis

The experimental results are presented in this step. Each research phase (paste, mortar and concrete) starts with the results from each experiment executed in that specific phase. The result, interpretation of the result and a compact conclusion for that experiment is provided. At the end of each phase, a conclusion is given. Note that observations and conclusions are made based on the range that is provided for each mix design parameter. The conclusion of both the paste- and mortar phase is used for the concrete mix design.

Step 7. Life cycle analysis

With all concrete performance results in, it is time for a life cycle analysis. In this life cycle analysis, a performance and low environmental impact alkali activated concrete mix is compared to a reference regular cement concrete mix provided by the industry. In this step the question of whether an equal performing alkali activated concrete has lower environmental impact compared to cement concrete is answered via life cycle analysis on product scale.

Step 8. Conclude the report

With all of the above, the research question can be answered and the report is concluded in the form of a discussion of results, conclusions and recommendations for further (future) research.

The reading guide, which couples the approach steps to the chapters, is presented in Figure 1.2 on the next page.

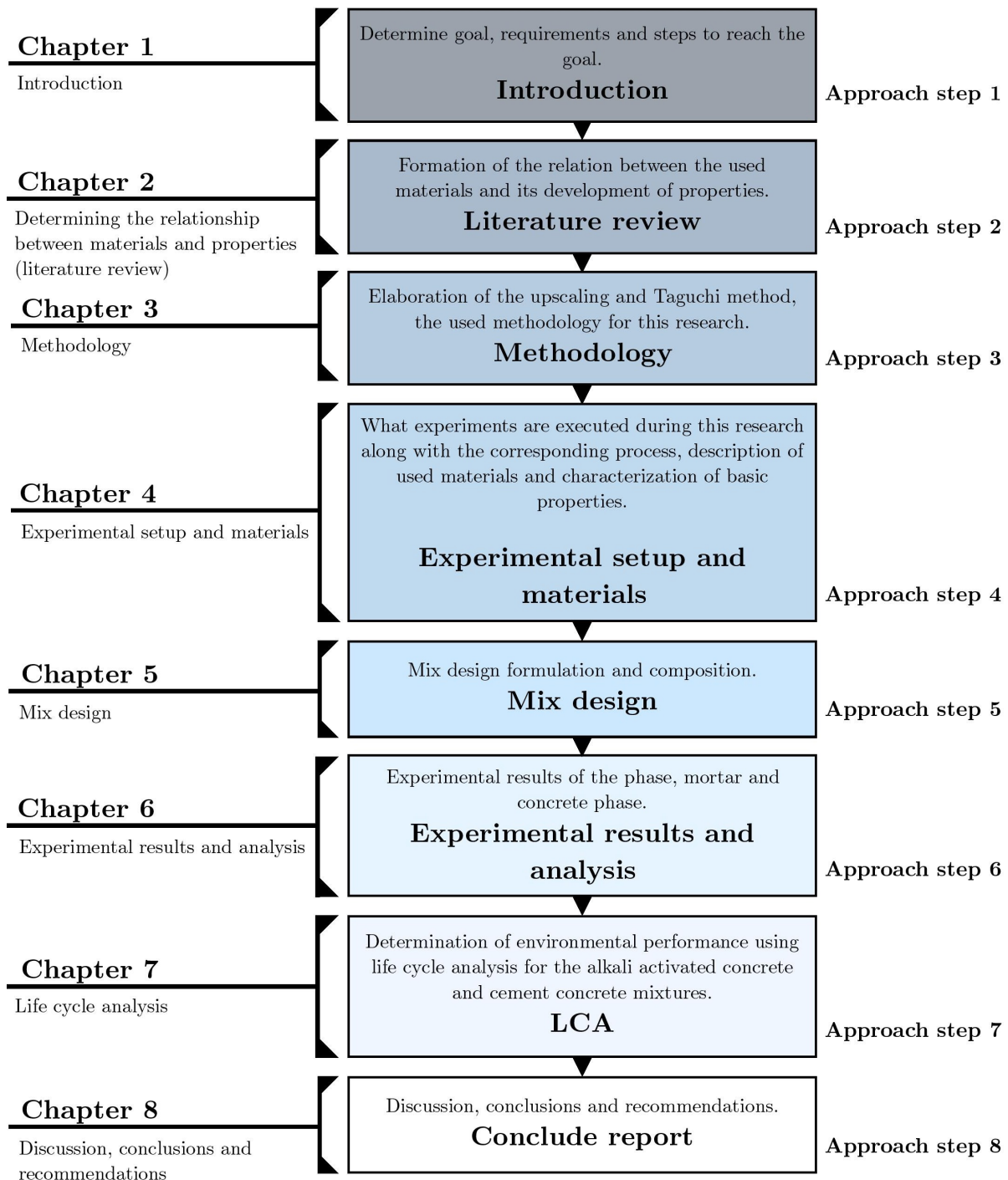
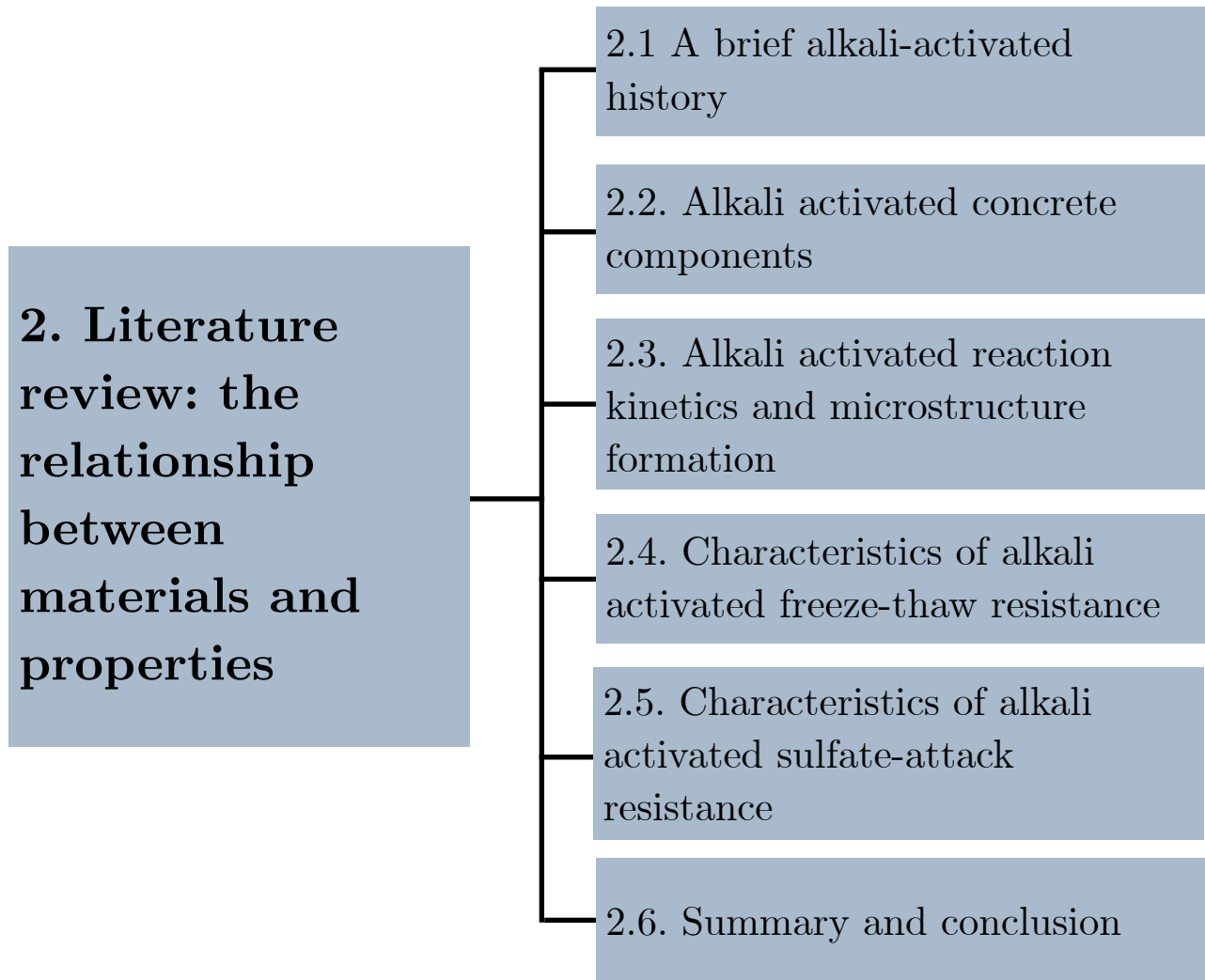


Figure 1.2 - Reading guide linked to methodology

2.

Literature review: the relationship between materials and properties



This chapter features literature in a structured manner. Section 2.1 starts with a description of the history of alkali activated concretes. This section is followed by a description of the main components of alkali activated concrete that are used in this research in section 2.2. Section 2.3 focuses on reaction kinetics and microstructure formation of alkali activated materials. Section 2.4 focuses on determination of characteristics of alkali activated concrete for freeze-thaw resistance by concluding experimental results of other researches. Section 2.5 has the same contents as section 2.4, but this time for sulfate attack resistance. Finally, section 2.6 provides a summary and concludes this chapter. Those that have experience with alkali activated concrete can start reading at section 2.3.

2.1. A brief alkali-activated concrete history

Activation of a precursor that is usually supplied as solid powder using an alkali hydroxide or alkali silicate is also known as geopolymers. Geopolymer concrete is therefore also known as alkali activated concrete. This term was coined in the 1970s by Prof. Joseph Davidovits from France.

Then, it was applied to a certain class of solid materials that were synthesized by an aluminosilicate powder with alkaline solution reaction. Initially, alkali activators were developed as fire-resistant coatings or other niche applications. However, a shift towards utilizing alkali activators in constructions has taken place, as Wastrels et al. (1993) observed that high-performance alkali activators can be made by alkaline activation of fly ash.

Alkali activated concrete is seen as development of low CO₂ construction material and as suitable alternative to Portland cement. Since there is a demand for low environmental impact construction materials, it is very interesting to investigate whether alkali activated concrete can be a replacement for Portland cement or blast furnace cement concrete (Provis & van Deventer, 2009).

2.2. Alkali activated concrete components

Alkali activated concrete contains roughly identical components compared to regular cement based concrete. However, the binder deviates from the main components. To describe the difference in components between cement concrete and alkali activated concrete, Figure 2.1 below is used and formed with knowledge from (Ye, 2020).



Figure 2.1 - Regular (left) and alkali activated (right) concrete components (self-made)

As can be observed, the components of alkali activated concrete are identical to regular cement concrete except for the binder. Where the binder of blast furnace cement concrete consists of cement and water, it consists of activator (sodium hydroxide and sodium silicate), precursors (ground granulated blast furnace slag and calcined clay) and water for alkaline activated concrete. Admixtures can be used in both types to modify the mix designs (e.g. a superplasticizer increases the workability of the mix design). The next few pages describe each component used in this research individually, starting with the activators, followed by the precursors and finally the properties of aggregates.

2.2.1. Activators

Sodium hydroxide (SH)

Sodium hydroxide is used as activator in alkali activated concrete due to its relatively low cost compared to other activators and its availability. It is a white solid compound consisting of sodium cations and hydroxide anions and can be solved in water. It is predominantly produced within the chlor-alkali process which has implications on environmental performance and emissions (Provis & van Deventer, s.d.). The material is highly corrosive, but aside from that, its most important properties are viscosity and heat of dissolution. (Provis & van Deventer, s.d.). With an increase of concentration, viscosity also increases. Until 1.0M, the activator behaves like water in terms of viscosity, but once concentration increases even further viscosity also increases at a fast rate.

As for heat dissolution, it is important to consider that very steep temperature increase takes place when a hydroxide solution is prepared. Another important property is that if the concentration of SH is too high, efflorescence takes place.

Sodium silicate (SS)

Sodium silicate is also known as water glass or Na_2SiO_3 and is often used in combination with SH in alkali activated mixtures. They are generally produced from carbonate salts and silica via calcination, followed by dissolution in water at desired ratios. The CO_2 that is emitted during these stages is much lower than that of Portland cement (Provis & van Deventer, s.d.).

2.2.2. Precursors

Ground granulated blast furnace slag (GGBFS)

Origin and production

Ground granulated blast furnace slag, or GGBFS, finds its origin in the iron and steel production industry. Iron ore, coke and sometimes limestone or dolomite are put in a blast furnace. This metallurgical furnace is used to melt these products. Once molten, the bottom of the blast furnace contains two layers: pig iron and slag. The slag can be air cooled, crushed and screened for it to be used as coarse aggregate. However, to obtain ground granulated blast furnace slag, the slag is blasted by water for rapid cooling, dewatered, crushed and then sieved. A visualized overview of the formation of GGBFS is presented in Figure 2.2 below (Nippon Slag Association, s.d.).

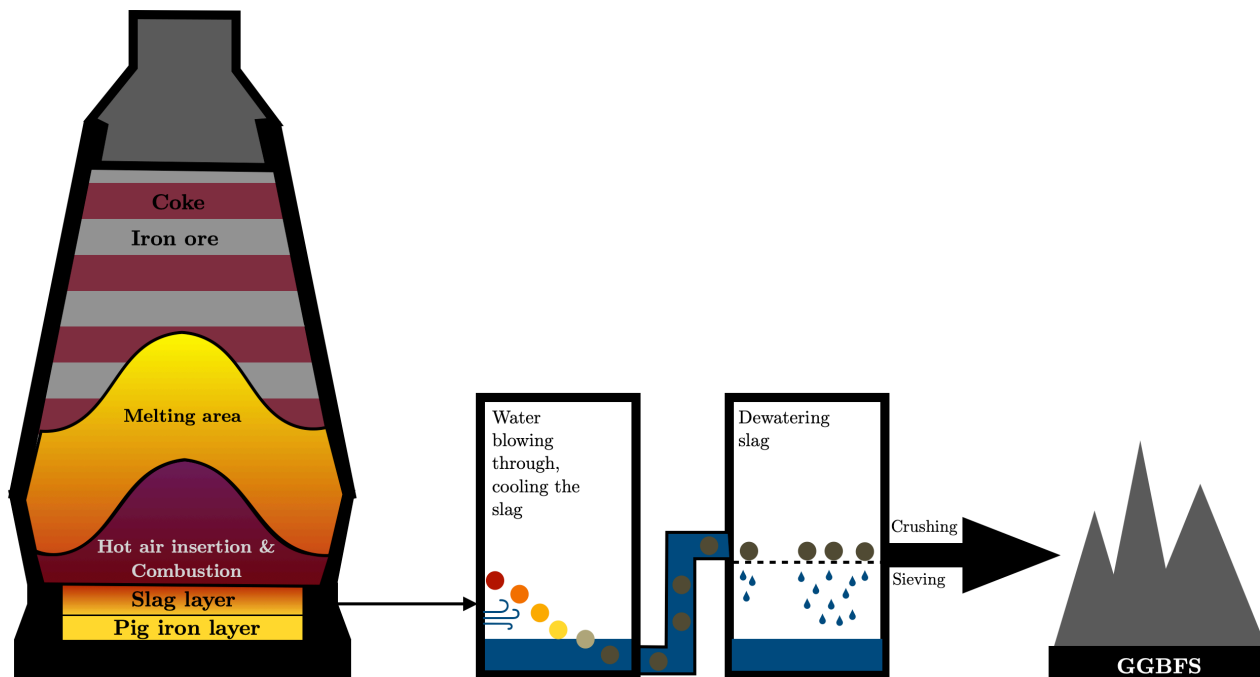


Figure 2.2 - GGBFS Production method (self-made)

The process of rapid cooling by blasting water onto the hot slag is important, such that the molten slag is solidified to a glassy state where little to no crystallization occurs. This forms sand-size fragments. Once crushed and sieved, the final ground granulated blast furnace slag product has cementitious properties (U.S. Department of Transportation, federal highway administration, 2016).

Composition

The chemical composition of ground granulated blast furnace slag may vary. Its composition is dependent on the temperature at time of water cooling and methodology of production. Of course the source of the material also has great influence on the final chemical composition. Table 2.1 below provides several chemical compositions of GGBFS as found in literature. It is clear that GGBFS contains large amounts of CaO, SiO₂ and Al₂O₃.

Table 2.1 - GGBFS Compositions as found in literature

Oxides	I.H. Aziz et al. wt. %	Hadi, Farhan & Sheikh. wt. %	Y. Fu et al. wt. %	Y. Barabanshchikov et al. wt. %	Cement concrete & aggregates Australia wt. %	Average
CaO	50.37	40.70	38.95	38.41	40	41.7
SiO ₂	30.40	32.40	33.91	37.96	35	33.9
Al ₂ O ₃	10.50	14.96	10.71	11.09	15	12.4
MgO	3.20	5.99	9.41	7.21	5	6.2
Fe ₂ O ₃	0.53	0.83	3.28	0.75	0.5	1.2
TiO ₂	0.98	0.84	3.43	-	-	1.75
ZrO ₂	0.05	-	-	-	-	0.05
MnO ₂	0.71	0.4	0.31	-	-	0.47
LOI	0.32	3.88	1.27	2.27	-	1.9

Role of GGBFS in mix design

GGBFS is used as a precursor in alkali activated mixtures. A precursor is defined as a compound participating in a chemical reaction that ultimately results in formation of another compound (Ye, 2020). GGBFS is described as a mixture of poorly crystalline phases with compositions that resemble gehlenite, akermanite and depolymerized calcium silicate glasses (Provis & Deventer, 2009). The degree of depolymerization largely controls its reactivity. As can be seen in the chemical composition in Table 2.1, there is sufficient calcium available to charge balance the aluminum contents, also known as an over-charge balanced calcium aluminosilicate framework.

The use of GGBFS can be either in a high-calcium based system or in a hybrid alkaline cement, with the first containing a calcium to aluminosilicate ratio > 1 and the latter being described as a mixed precursor consisting of a high-calcium-based and aluminosilicate-based precursor. In both systems the chemical reaction needs a relatively moderate to high alkaline condition. Curing can be performed at room temperature.

Reactions of GGBFS in mix design is dependent on particle size. Particles above 20 μm react slowly whereas particles below 2 μm react within 24 hours. The particle size distribution is therefore important and provides room for controlling strength development.

Calcined clay (CC)

Origin and production

Calcined clay is seen as one of the possible candidates to be used in alkali activated concrete as a precursor. The adoption of GGBFS, Fly ash (FA) and Metakaolin (MK) in alkali activated concrete is proven. However, due to the already high utilization rates of most of these materials, it is good to look beyond these precursors. As most information of calcined clay relates to metakaolin, metakaolin is used as source of information.

Metakaolin is the calcined form of kaolinite. Its advantages are purity and consistent composition, however a scale-up in the industry is deemed difficult due to availability of the material. Poor workability and high water demand due to plate-like morphology do not help with this problem as well. Using lower purity clay, or calcined clay, might resolve the above stated problems. (Khalifa et al., 2020). Thermal treatment is the most common way to increase reactivity of clays. By heating the clay, the octahedral layer undergoes dehydroxylation causing a reduction in the coordination number of the Al atoms making them more reactive. For this calcination process, in the industry the use of a rotary kiln or flash calcination can be used. The rotary kiln tends to increase particle size and decrease specific area, which is enhanced with higher calcination temperatures. Due to this increase of particle size, the specific area available for dissolution is decreasing, thus calcination temperature should be kept as low as possible.

The three most found and used clay materials are kaolinite, montmorillonite and illite. They are categorized as 1:1 clay minerals and 2:1 clay minerals. Figure 2.3 to the right visually elaborates the difference between the two.

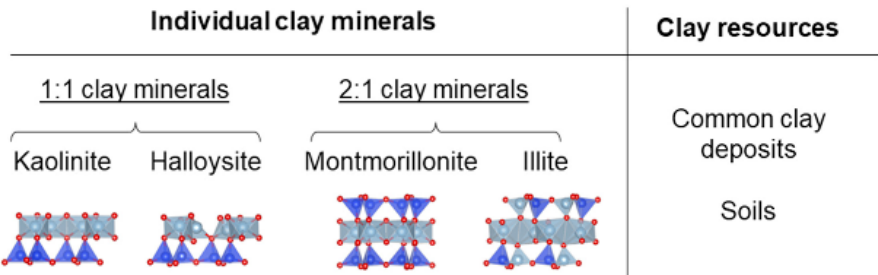


Figure 2.3 - Clay composition (a.z. khalifa et al.)

Production takes place by calcining the clay at temperatures in the range of 650 - 700 °C.

Composition

Clay minerals have wide range of chemical compositions due to the highly variable influence of formation, weathering and transportation methods. They are an abundant aluminosilicate resource, as they make up 90 % of the earth’s crust. Clay minerals are known as complex silicates of various ions as aluminum, magnesium and iron. The arrangement of these ions defines crystalline units of the clay of two types, namely 1:1 and 2:1 clays (Kumari & Mohan, 2021). 1:1 clay minerals consist of each layer that is formed by one tetrahedral sheet (silicon surrounded by four oxygen atoms to form the silica sheet) and one octahedral sheet (aluminum or magnesium consisting of aluminum surrounded by six hydroxyl units to form a gibbsite sheet or brucite sheet). In 2:1 clay, each layer has one octahedral sheet that is between two tetrahedral sheets. Figure 2.4 on the next page shows the atomic diagram and SEM image of kaolinite (1:1 clay) and halloysite (2:1 clay).

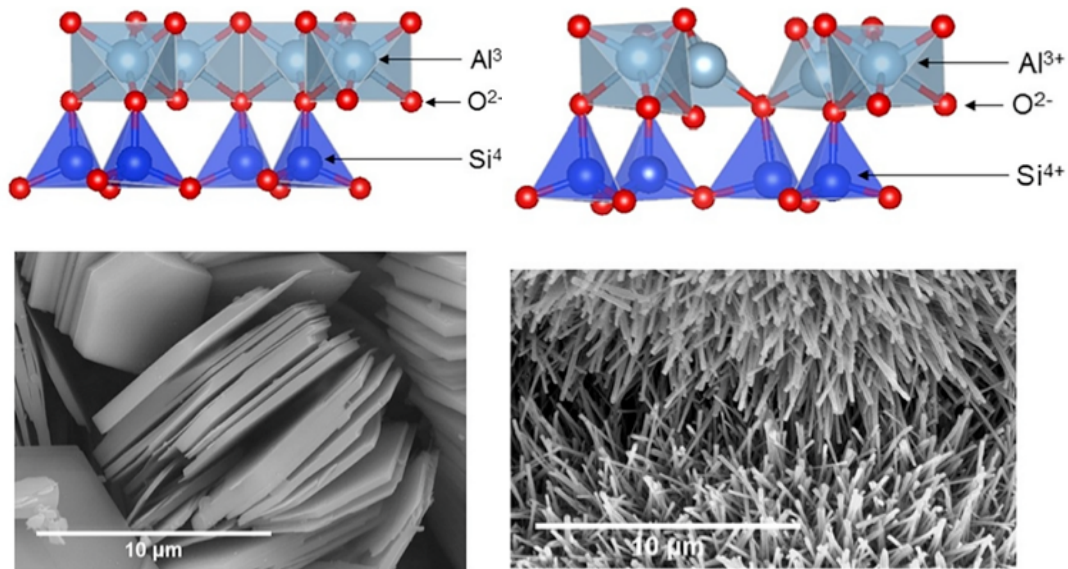


Figure 2.4 - Atomic composition and SEM image of kaolinite and halloysite (Khalifa et al.)

Kaolinite is most commonly used in the form of metakaolin (MK). Kaolinite is also known as China clay. The chemical weathering of aluminum silicate as feldspar produces a soft, white earthy mineral known as the clay. Dehydroxylation of Kaolinite, forming metakaolin, occurs at temperatures in the range of 650 - 700 °C. The coordination of Al atoms in the octahedral sheet is then reduced to a 3 to 6-fold coordination, where the 5-fold coordination is most reactive. The available surface area also decreases. Overall structure is highly variable and reactivity of MK is related to the degree of structural order (or crystallinity). Chemical activity also increases with amorphous content of MK.

2:1 clay minerals have greater variety than 1:1 clay minerals and are classified by permanent layer charge, interlayer cations and hydration ability. The most common 2:1 clay minerals are montmorillonite and illite. For montmorillonite dehydroxylation results in a reduction of Al coordination from 6 fold to 5 and 4-fold. For calcined clay, the entire formation method is relatively equal to metakaolin, but the difference is mainly its reactivity, which is lower in this case.

Role of calcined clay in mix design

Clay minerals are low-calcium systems. For these calcined clays to react there must be sufficient thermodynamic driving force and dissolution kinetics should be sufficiently fast. The formed reaction products are either crystalline zeolites or an aluminosilicate hydrate gel framework.

For clays, an optimum concentration of sodium hydroxide (SH) should be larger or equal to 5M, where an optimal range is defined as being between 8 - 12M.

Sodium carbonate can be used to activate GGBFS as it can dissolve at quite mild pH, but this is not transferrable for clay minerals because of higher pH or highly alkaline environment required for dissolution. For the overall reaction system, the Si:Al molar ratio is very important as it influences final reaction products. A range of 1 - 1.5 may result in formation of both zeolites and alkali activated geopolymers. Values above 1.5 results mainly in alkali activated geopolymers, values below 1.5 mainly results in zeolites. A higher ratio also results in longer setting time. Due to this ratio, it is common to use Na_2SiO_3 (sodium silicate, SS) solution to control this ratio. Using SS also results in reduced porosity and better mechanical strength (Khalifa et al., 2020).

Curing temperature has effect on mechanical properties of the clay as well. Highly reactive clays are able to develop good properties when cured at ambient temperatures, whereas non-calcined clay, such as kaolinite, needs curing temperatures of 60 - 100 °C. Note that CC mix designs can undergo

drying shrinkage cracking, especially when exposed to a dry environment. Rapid drying during curing is therefore not recommended. This is due to the swelling capacity of clay caused by unbalanced charge on their surface.

2.2.3. Aggregates

Aggregates are also known as gravel and sand. Generally speaking, around three quarters of concrete volume is occupied by aggregate. Using good quality aggregate is therefore important as it may limit strength of the cured concrete, durability aspects or structural performance. Often aggregates are distinguished by their size. Aggregates below 5mm are called fine aggregates and often referred to as sand and aggregates above 5mm are called coarse aggregates, also known as gravel (Neville, 1995).

Properties of aggregates

Particle shape and texture

These external characteristics are of importance. Packing of all particles in concrete is important for strength development and is dependent on the angularity of the aggregate. A good example is found in the book Properties of concrete of Neville where an example of Shergold is given where the content of round aggregate percent has been compared to voids ratio, see Figure 2.5 below.

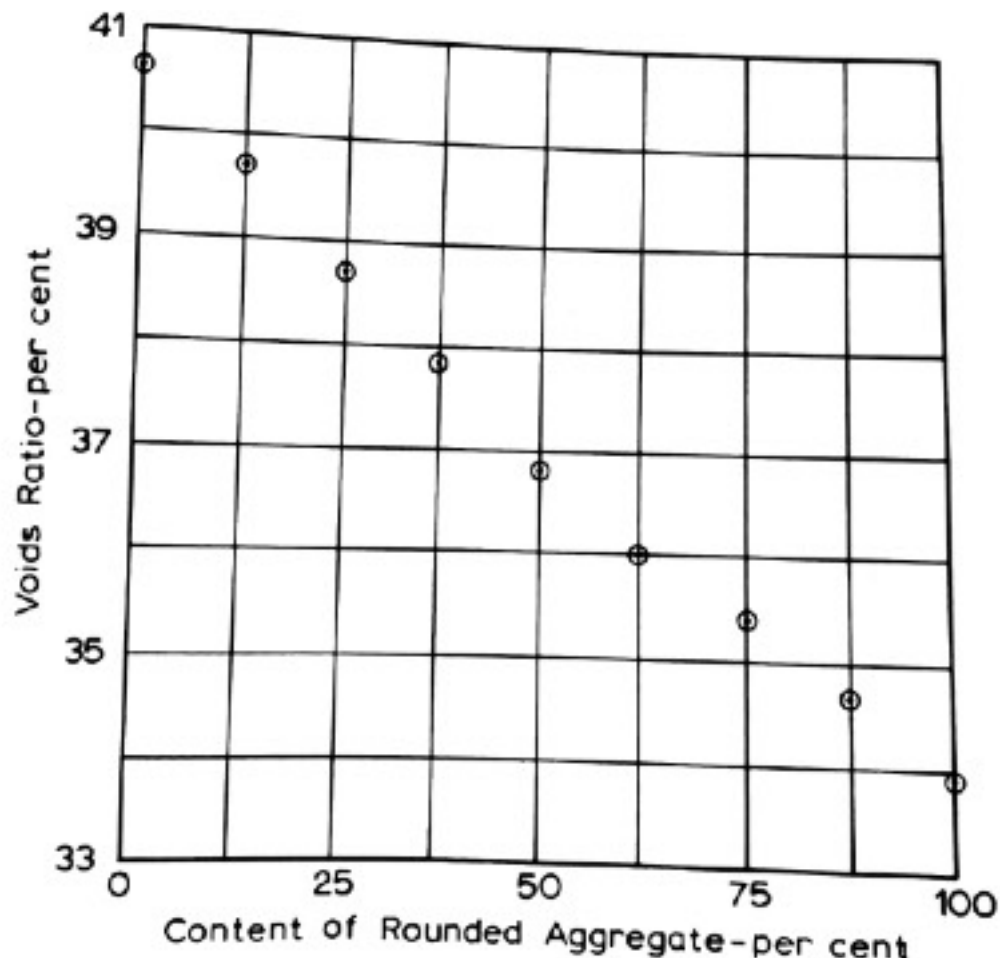


Figure 2.5 - Rounded aggregates vs voids (Neville, 1995)

Shape and the amount of pores also influence water requirement which is directly related to final strength and lastly, it has influence on workability.

Bonding

Describes the bond between aggregate and paste due to interlocking of aggregate and hydrated paste. Rougher surfaces result in more bond strength. Bonding is not really tested, but can be observed when crushing. When the aggregate is broken right through, it indicates good bond strength.

Specific gravity

Absolute specific gravity is volume of the solid material excluding pores (also excluding internal pores). Including impermeable pores but not capillary pores, then it is defined as apparent specific gravity. Specific gravity is used for defining quantity of aggregate required for a given volume of concrete.

Bulk density

The transformed specific gravity into units often used in concrete, namely from kg/l to kg/m³. It is used to determine how much mass of aggregate fills a container of certain volume. Bulk density depends on the size distribution and shape of the aggregate. When, for example, packing smaller aggregates between larger ones, the bulk density will increase as more voids are filled without increase in volume.

Porosity and absorption

Porosity, absorption and permeability influence aggregate properties as the bond between it and cement paste, resistance to freeze-thaw, chemical stability and resistance to abrasion. By determining porosity and water absorption, mix designs can be adjusted as more or less water than prescribed might be needed due to more/less porosity/absorption.

Moisture content

Aggregates have a basic state of saturated surface-dry. This implies that internal voids are saturated with water combined with no water on the surface of the aggregate. Moisture content is defined as the water in excess of saturated surface-dry condition. As aggregates get exposed to rain, mix design has to account for surface water or free moisture on the aggregate, especially for fine aggregates as these can contain up to 10 % of surface moisture. The different moisture composition in aggregates can be observed in Figure 2.6 below.

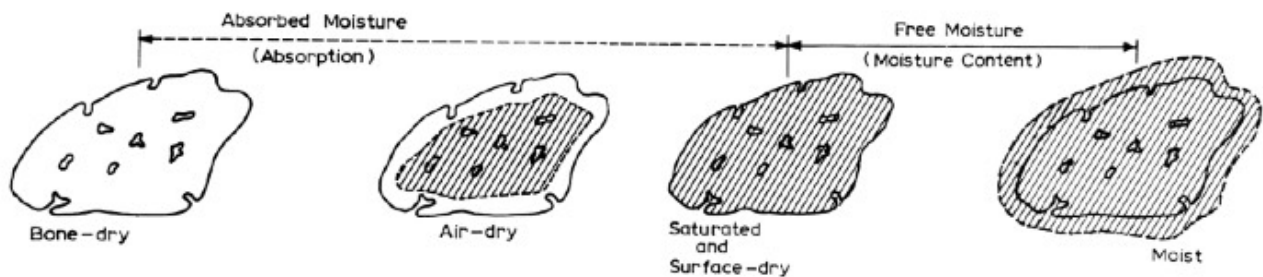


Figure 2.6 - Moisture composition in aggregates (Neville, 1995)

Role in mix design

In this research a special type of concrete production is used for creation of alkali activated concrete, namely the dry-cast production methodology. For this method, earth moist concrete (EMC) is required. According to literature, behavior of alkali activated concrete compared to regular cement concrete is similar in terms of workability and aggregate composition. Therefore, particle size distribution of regular cement EMC should be used for mix design of alkali activated concrete. Especially the packing of solids is important, as it influences both workabilities and properties of the hardened concrete.

2.3. Alkali activated reaction kinetics and microstructure formation

The aluminosilicate precursors that are used in this research are within a class of materials where Si and Al form the main framework. Ca and Mg both are elements which are able to modify this framework (Duxson & Provis, 2008). In order to understand how alkali activated concrete can develop strength, the following topics should first be understood:

1. Dissolution of alkaline activated materials
2. Pore solution chemistry of alkaline activated materials
3. Reaction kinetics of alkaline activated materials
4. Reaction products of alkaline activated materials

These topics are discussed in this section, in the order presented above, after which microstructure formation is described.

1. Dissolution of alkaline activated materials

Dissolution of alkaline activated materials describes the dissolution process of the precursor into the activator. The dissolution process of aluminosilicate precursors is described using the following four steps, also visualized in Figure 2.7 (a-d):

1. Dissolved alkali and alkali-earth metal elements originating from the surface due to metal/proton exchange reactions (Figure 2.7 (a)).
2. Hydrolysis of aluminum oxide bonds starts (Figure 2.7 (b)).
3. Silica oxide bonds start to break (Figure 2.7 (c)).
4. Release of tetrahedrally coordinated Al and Si in the framework into the solution. Framework is gradually broken down (Figure 2.7 (d)).

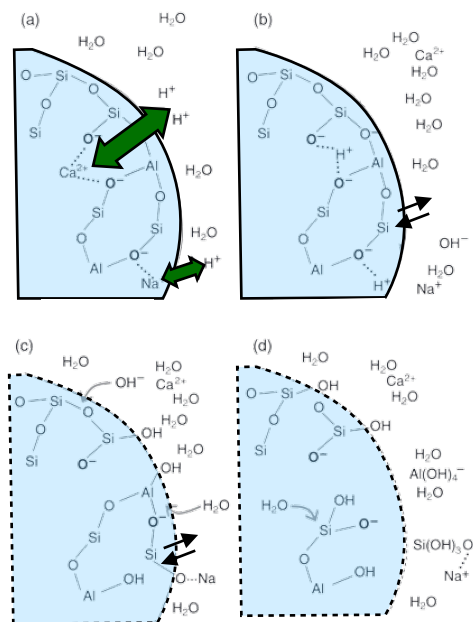


Figure 2.7 - Illustration of dissolution of aluminosilicate precursors (modified from (Duxson & Provis, 2008)). Blue shows the precursor framework, while the white area around is the solution.

During dissolution, the Al dissolves more easily than Si as the Al-O bonds break more easily than Si-O bonds. The framework of aluminosilicate glass therefore breaks in two steps: initial dissolution of a small fraction of Al, followed by the dissolution of Si that is adjacent to the initially dissolved Al (Zuo, 2019). After dissolution, the dissolved Al and Si generate silicate species through hydrolysis.

2. Pore solution chemistry of alkaline activated materials

The pore solution describes the alkaline solution which is present in the pores of the alkaline activated concrete. In this medium, the aluminosilicate precursors dissolve, reactions take place and subsequently solid reaction products are formed (Zuo, 2019). For alkali activated slag, higher alkalinity of the pore solution results in higher concentrations of Si and Al, whilst it lowers concentrations of Mg and Ca. The alkali activated slag also has pH dependent behavior, showing that a pH of > 11.5 is required to effectively activate ground granulated blast furnace slag, as a pH < 11.5 results in ground granulating blast furnace slag not reacting at all due to low equilibrium solubility of Si (Song & Jennings, 1998). The pore solution is also important for the durability of alkaline activated materials, as a higher alkalinity of the pore solution results in more stable calcium aluminosilicate hydrates and alkali-aluminosilicate gels (Zuo, 2019).

3. Reaction kinetics of alkaline activated materials

Reaction kinetics in alkaline activated materials describes the rate of chemical reactions. For slag activated materials, the activator that is used has influence of the reaction kinetics. These reaction kinetics are generally studied using isothermal calorimetry. Similar to ordinary Portland cement, alkali activated materials experience several reaction stages which are controlled by different reaction mechanisms. The total reaction process can generally be described with the following stages (Shi & Day, 1995):

- Initial dissolution period
- Acceleration/deceleration period
- Steady period

A general trend for slag-based alkali activated materials in terms of reaction kinetics is presented in Figure 2.8 below. It is shown for both using sodium hydroxide and sodium silicate as activating medium.

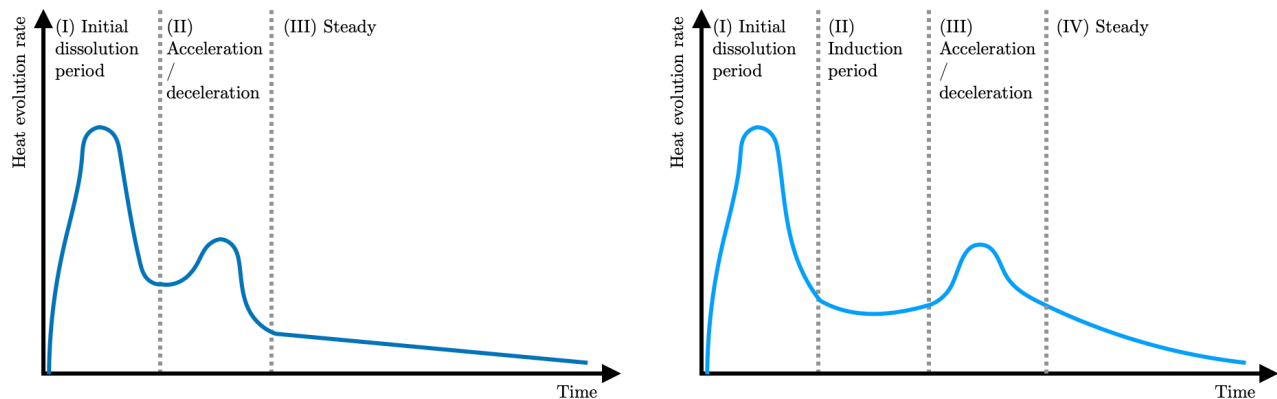


Figure 2.8 - General heat evolution rate and heat release for sodium hydroxide (left) and sodium silicate (right) activated ground granulated blast furnace slag (modified from (Zuo, 2019)).

The very high heat evolution in the first moments of time for sodium hydroxide activated slag is explained by the dissolution or wetting of the slag after mixing it with the activator. The second peak, during the acceleration/deceleration period represents the formation of many different reaction products. Afterwards, a steady period can clearly be visible with no observable peaks. Compared to the reaction process of Portland cement, no noticeable induction period is observed for sodium hydroxide activated slag.

For sodium silicate activated slag, an additional induction period is visible. The heat evolution starts with a sharp peak, again indicating the dissolution/wetting of the slag. This is followed with an induction period. Afterwards, another acceleration/deceleration peak is observed followed by a steady period.

The research from (Zuo, 2019) mentions that both the height of the heat evolution peaks and the delay of the acceleration peak can be controlled by the sodium oxide (Na_2O) content and the amount of added SiO_2 . Higher Na_2O results in higher heat evolution rates whilst also increasing the height of the acceleration peak. Introduction of additional SiO_2 results in both a delay and decrease in height of the acceleration peak.

Strength development can be coupled to reaction kinetics. As sodium hydroxide activated alkali materials have the trend of fast initial dissolution, immediately followed by an acceleration/deceleration period, this means that sodium hydroxide contributes to early strength development. Sodium silicate activated alkali materials also have a fast initial dissolution period, but the acceleration/deceleration period is delayed resulting in a more gradual strength development.

4. Reaction products of alkaline activated materials

The type of aluminosilicate precursor has influence on the type of reaction products that are formed. For activated slag, the main reaction product is calcium aluminosilicate hydrate gel, or C-(N)-A-S-H (Zuo, 2019). When blending non-calcium aluminosilicate calcined clay with calcium rich blast furnace slag, the reaction products can be complex, like the same C-(N)-A-S-H gel or aluminosilicate hydrate gel, N-A-S-H. If alkalinity is high, N-A-S-H gel is the main contributor to mechanical strength whereas if alkalinity is low, the presence of calcium silicate hydrate or C-S-H gel contributes significantly to the performance of the binder (Rakhimova & Rakhimov, 2018).

Microstructure formation

Microstructure formation of alkaline activated materials is dependent on the type of alkaline activator. A good example for activated slag is presented in Figure 2.9 below:

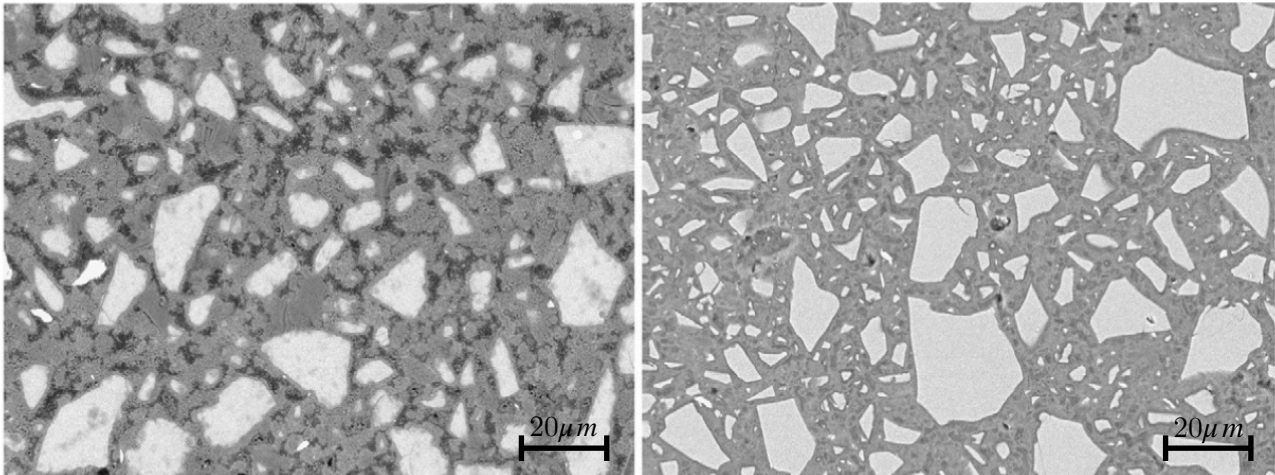


Figure 2.9 - Microstructure formation of sodium hydroxide (left) and sodium silicate (right) activated slag paste after 28 days curing (from Haha et al., 2011).

The sodium hydroxide activated slag has more pores (dark parts in the image) than the sodium silicate activated slag. The hydration products are also less homogeneously distributed and mostly around the slag particles for the sodium hydroxide activated slag, whereas more spread out hydration products can be seen in the sodium silicate activated slag (Haha et al., 2011).

Microstructure formation of alkaline activated calcined clay is presented in Figure 2.10 below:

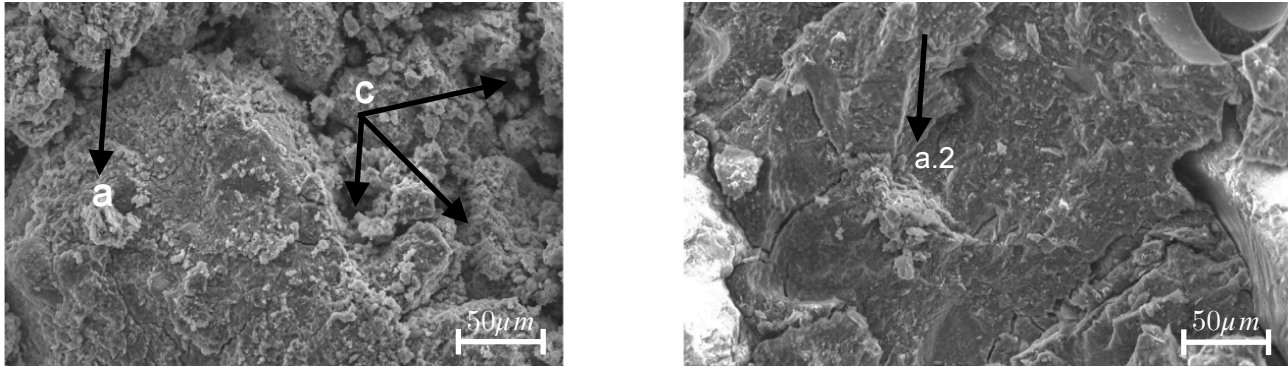


Figure 2.10 - Microstructure formation of sodium hydroxide (left) and sodium silicate (right) activated calcined clay paste after 28 days of curing (from Bature et al., 2020).

When activated with sodium hydroxide a densified and packed structure (a) can be observed as well as a large pore content (c), comparable to microstructure formation with slag as precursor. The sodium silicate specimen shows a bulk geopolymer structure with sharp edges. The geopolymer gel appears to the surface of the matrix (a.2). The microstructure of low purity calcined clay geopolymers cured at ambient temperatures is consolidated enough to provide high compressive strength (Bature et al., 2020).

While the microstructure formation of both activated slag and calcined clay have common properties, it is clear that microstructure formation is strongly influenced by the type of activator that is used. Sodium hydroxide activated precursors generally show more pores and a less homogeneously distributed geopolymer network, whereas the sodium silicate activated precursors have a more homogeneously geopolymer network combined with less pores.

The pore formation of alkaline activated materials also depends on the dosage of alkali activator. When increasing alkali dosage, porosity tends to decrease while the strength increases. An increase of alkali dosage increases the degree of hydration, which results in formation of more reaction products that cause the pore structure to be more dense (Shi et al., 2017). With higher alkali dosage, the percentages of small pore volume are larger than that of large pores for alkali activated slag. Total porosity of alkaline activated slag decreases with both dosage and curing time (Hu et al., 2020).

2.4. Characteristics of alkali activated freeze-thaw resistance

Water expands about 9 percent when it freezes. This water, situated in pores of concrete, produces pressure when it freezes due to this expansion. If this developed pressure exceeds tensile strength of the concrete, cracks occur that ultimately result in degradation of concrete (PCA, s.d.).

Definition, requirement and relevance

Definition: A deterioration process that occurs in concrete structures in cold climate due to expansion of water causing failure due to exceeding (internal) tensile stress and scaling of material.

Requirement: For application in coastal areas, there are two requirements that should be fulfilled for freezing and thawing (FT). One requirement is described for full scale products, but the requirement used in this research is used at lab scale. The concrete in revetment products can be located in or above sea water level. Therefore it should be resistant to both the salt sea water and possible de-icing salts. This research utilizes the widely adopted CDF test, as described in NVN,CEN/TS 12390-9:2016. This method features a preconditioned specimen which is subjected to

28 freeze-thaw cycles with a surface covered in 3 % NaCl solution. Mass loss is determined, which should be $\leq 1.5\text{kg/m}^2$ after 28 FT cycles.

Relevance: A good resistance to freezing and thawing is very important, especially for coastal revetment products. If a concrete is not freeze-thaw resistant, cracking occurs resulting in reduced properties, for example a reduced compressive strength capacity. Freeze-thaw can also introduce weight loss (also known as scaling) to the concrete, which reduces stability of concrete revetment products. Ultimately, if coastal revetment products do not have sufficient FT resistance, failure of the revetment may occur due to a combination of weight loss and internal cracking. Therefore, this is a durability parameter that greatly influences the lifespan of these concrete products and resistance should be made sufficient.

Freeze-thaw resistance literature review

There are two mechanisms that influence the performance of alkaline activated concrete subjected to freezing and thawing: Internal damage and scaling. Freezing of concrete in water seldom leads to surface scaling, but more often to internal damage whilst freezing of concrete exposed to salt solution at the surface seldom leads to internal damage (Fagerlund, 1995). Both mechanisms are described separately below.

Internal damage

Is caused by freezing water that is contained in:

- Capillary pores within the paste
- Pores in saturated and porous aggregates
- Air-pores
- Cracks
- Interfacial transition zone between aggregate and paste

To elaborate internal damage caused by freezing water, Figure 2.11 is used.

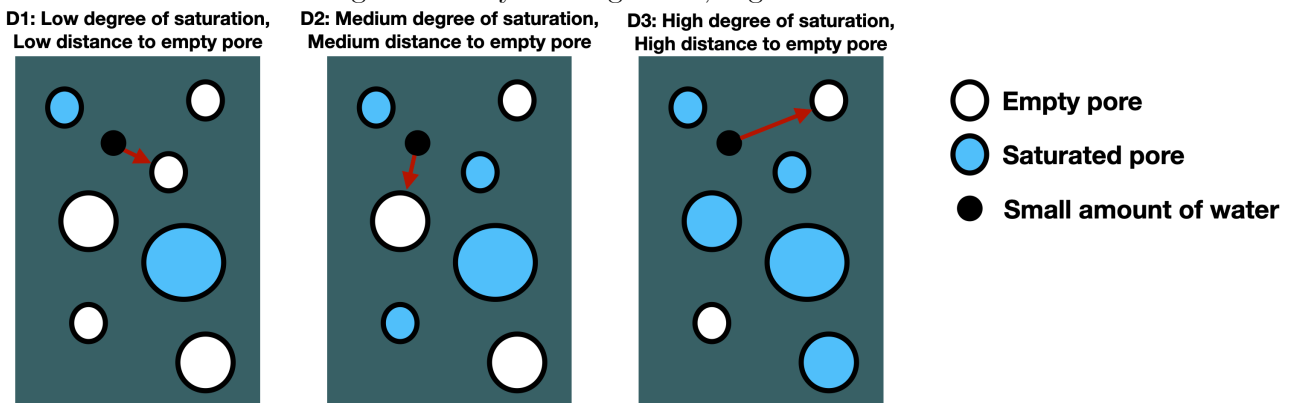


Figure 2.11 - Degree of saturation and flow distance, modified from (Li et al., 2012).

Figure 2.11 represents a concrete/mortar specimen with different pores in three different cases:

1. Low degree of saturation and low distance to empty pore (D1, left)
2. Medium degree of saturation and medium distance to empty pore (D2, middle)
3. High degree of saturation and high distance to empty pore (D3, right)

The black dot represents a small amount of water. If that water has low flow distance to an empty pore, it has room in the empty pore to expand when subjected to freezing. However, if the flow distance to the empty pore is too large (either because of a low amount of pores or a high amount of saturated pores) the crystallization pressure caused by ice formation cannot be released by the

pores. When the concrete/mortar freezes, this water expands and causes stresses in the concrete which results in cracking. This way, it deteriorates concrete/mortar (Li et al., 2012).

Freezing and thawing resistance can be alleviated in terms of internal cracking by reducing the degree of saturation. Below a degree of saturation of 85 %, the pores within the concrete are filled with gases which provide a buffer volume in which the stresses induced by ice formation can be relieved. As long as the stress caused by the freezing of water is lower than the stress the layers around can handle, no cracking occurs. Therefore, air bubbles are very important when it comes to freeze-thaw performance in terms of internal damage (Li et al., 2012).

The air bubble content (volume), size of the air bubbles and the distribution of the air bubbles within the concrete are as important. Too large amount of air bubbles cause strong reduction in mechanical performance as there is simply less strength generating paste and aggregates around these air bubbles (Li et al., 2012).

Permeability is also an important characteristic for internal damage (Abed & Balázs, 2021). With low permeability, the pores in the concrete are small, which results in strong concrete but the pore solution cannot relief its crystallization pressure if there are not sufficient empty pores available. Therefore, for internal damage characteristics, low permeability is only recommended if there is a sufficient amount of empty pores.

Internal cracking results in compressive strength loss, therefore compressive strength is one of the experiments conducted to determine freeze-thaw performance of alkaline activated mortar and concrete.

Concluding internal damage

Internal damage is caused by expansion of water due to freezing causing ice crystallization pressures that cause internal damage if there is insufficient empty pores that relief this pressure. Therefore, sufficient air voids are important for the concrete to withstand this mechanism, but too much air voids results in reduction of mechanical performance. Low permeability also results in good freeze-thaw resistance, but only if sufficient pores are available to release the internal stress caused by freezing water present in the capillary pores, air-pores, saturated aggregates, cracks and interfacial transition zone.

Surface scaling

Surface scaling is caused by freezing of the concrete surface when it is in contact with saline solutions of weak concentration (Fagerlund, 1995).

Scaling is the main parameter that is measured for expressing freezing and thawing performance. This salt scaling, due to the sodium chloride solution to which the concrete is exposed, consists of progressive removal of small flakes or chips of binder, reducing concrete cover and thereby causing reduced mechanical performance. The salt solution, when frozen, results in an ice layer that cracks when it forms a composite with cementitious slabs, which does not occur with a pure ice (without salt solution) under the same conditions. Salt scaling progression is either accelerated, linear or retarded with increasing amount of cycles. The difference is elaborated with Figure 2.12 on the next page.

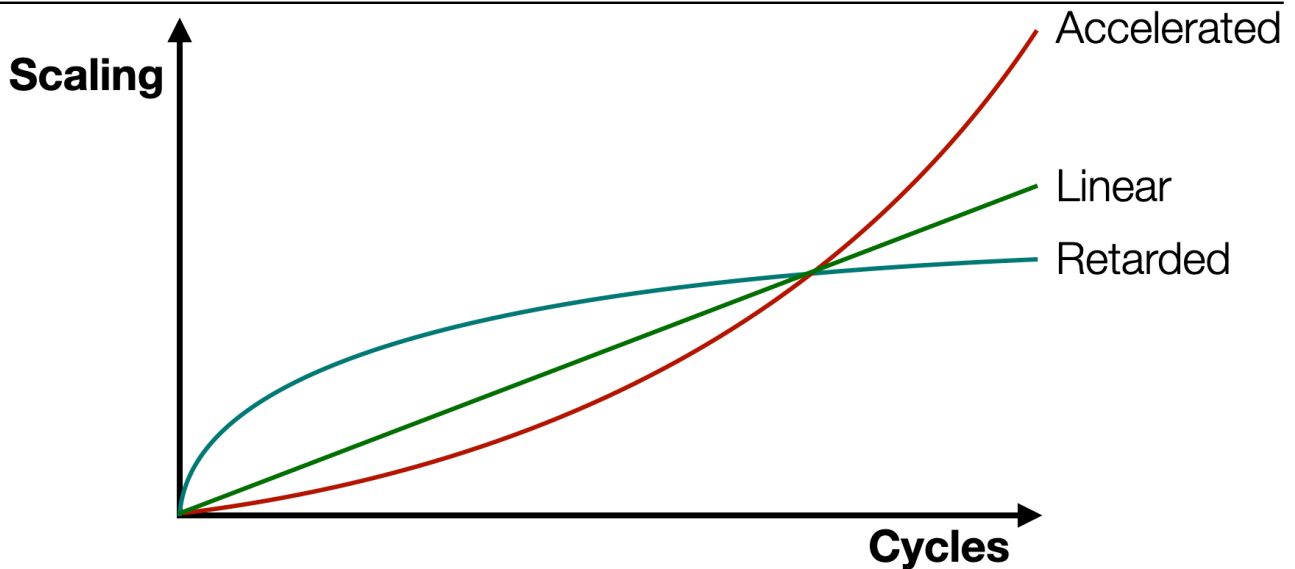


Figure 2.12 - Scaling types plotted for amount of scaling relative to amount of cycles.

Generally, when scaling is retarded or linear the concrete has high salt scaling resistance.

The amount of scaled material can be reduced by using air entrainment, as it reduces bleeding which results in a stronger surface. Air entraining agents can also impose suction in the pore fluid, compressing the porous skeleton, resulting in better performance as well, but this admixture is not considered in this specific research (Valenza & Scherer, 2006).

Another method to reduce scaling is to reduce permeability. With lower permeability, the scaling solution cannot penetrate into the specimens, reducing the damage it can do due to cracks on the surface of the concrete.

Pore connectivity and pore size distribution are also very important contributors for scaling. While larger pores provide more expansion possibilities for release of crystallization pressure caused by freezing water, they also provide more surface area to which the saline solution can bond. After freezing, this results in more scaled material. Same for pore connectivity, when there is a lot of pore connectivity the saline solution is able to penetrate more deeply into the specimen, resulting in more available surface area for the saline solution to form a layer to the concrete structure causing more scaling.

Concluding surface scaling

Surface scaling is caused by freezing of the concrete surface when it is in contact with saline solutions of weak concentration due to formation of an ice layer which weakens the concrete surface. Generally, scaling progression is described by an accelerating, linear or retarding behavior where the latter two are seen as concretes with high salt scaling resistance. Scaling can be reduced by using air entrainment to reduce bleeding resulting in a stronger surface, reduce permeability such that the solution cannot penetrate the specimen or by prevention of pore connectivity and large pores as these provide more surface area for the saline solution to form an ice layer.

Shrinkage, cracking and recycled aggregates and why they influence freezing and thawing performance

The microstructure and especially the interaction between aggregates and the hardened paste, also known as the interfacial transition zone, is also a possible degradation mechanism. This starts with internal small cracks in the microstructure of the gel material which gradually increases in size. At the same time, the aggregates can start to split, where the residual strength of the concrete depends mainly on the bonding between aggregate and gel (Chen & Wang, 2021).

Shrinkage of alkaline activated slag is reported often and can cause micro cracks in the internal structure of the paste (Collins & Sanjayan, 1998), (Puertas et al., 2003).

When exposed to drying conditions, alkali activated slag concrete can show greater shrinkage strains than regular concrete, especially when stiff coarse aggregates are used which form a restraint, leading to more microcracks (Collins & Sanjayan, 2001). Same goes for specimens exposed to freezing and thawing cycles. The de-icing solution can penetrate the microcracks and during freezing period, expand (as water expands 9 % when freezing), causing larger internal cracks which also leads to scaling.

Recycled aggregates can also induce more drying shrinkage, causing micro strain in the concrete. This effect is more pronounced for alkaline activated slag concrete than regular concrete whilst recycled aggregates also increase internal porosity, therefore also increasing the permeability, resulting in more scaling (Nanayakkara et al., 2020).

State-of-the-art literature review

Freeze-thaw resistance with GGBFS combined with SH and SS, with a modulus of SS of 3.34 is tested in this paper. The mix design varied in amounts of slag and activator solution:slag by mass. Mass loss and relative dynamic elasticity modulus were investigated. A percentage of mass loss of less than 1 % was measured for all specimens after 300 cycles, with relative dynamic elasticity modulus of 89 % for the worst performing specimen. SEM analysis showed little inner cracks and pores. With hydration products being symmetrical and close-grained. It is concluded that due to this packing, water can hardly penetrate the material thus saturated concrete is unlikely, improving FT resistance. The paper also recommends to use dynamic elasticity modulus attenuation power function models to reflect FT damage such that a forecast of FT standard life time of slag-alkali activated is possible (Fu et al., 2011).

Another research investigated FT resistance of slag-alkali activated concrete with recycled concrete aggregates (RCA). 10M NaOH solution and sodium silicate with sodium oxide to silica oxide ratio of 3 are used as activators. RCA are used with GGBFS binder of 300-400-500 kg/m³ and after 28d curing exposed to 100 FT cycles. Compressive strength, weight change, sorptivity values were used in combination with SEM and EDS.

Compressive strength losses due to FT were between 12 - 15 % and resistance increased with increasing the binder content (causing decreasing sorptivity values and pores). FT resistance decreased when substituting RCA as these aggregates have higher water absorption and weaker mechanical structure than river aggregate. Degirmenci reported a decrease of strength after FT cycles as Na₂SiO₃/NaOH increased. Weight losses after 100 FT cycles ranged from -1.9 % in the worst case (300 kg binder + 100 % recycled aggregate) upwards to -0.7 % (500 kg binder + 0 % recycled aggregate) (Ugurlu et al., 2021).

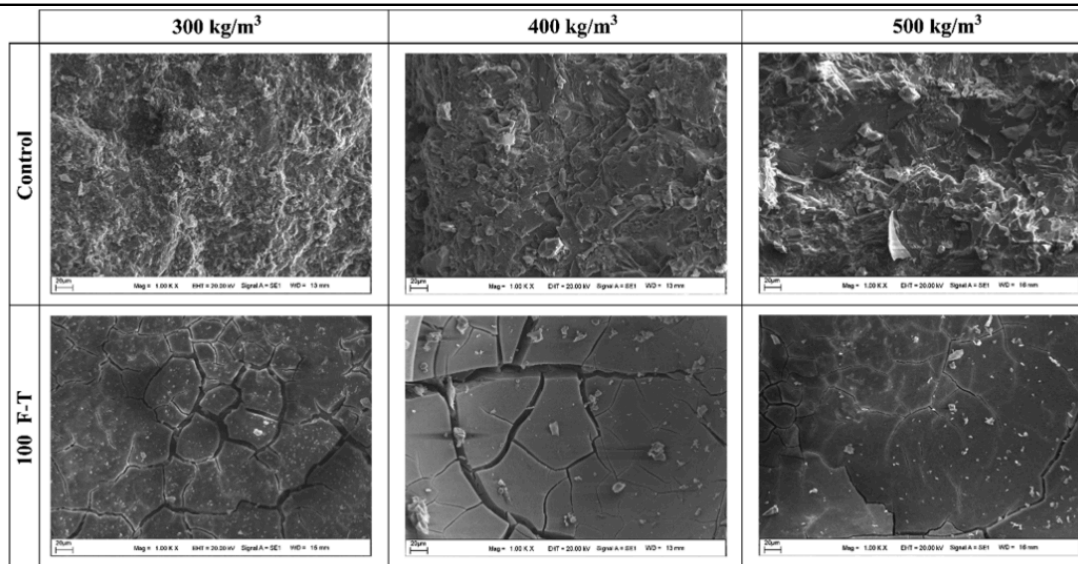


Figure 2.13 - SEM FT GGBFS vs Control mix images (Ugurly et al.)

As can be observed in Figure 2.13 above, the amount of visible cracks after 100 FT cycles decreased which also explained the lower sorptivity values when using more binder.

The RILEM round robin test also considered FT resistance of alkali activated concretes. Two slag based mix designs were considered. A sodium silicate solution of modulus 2.0 was used combined with sodium hydroxide. FT methods included CDF test and ASTM testing procedures. The higher strength mix design exhibited less surface scaling resulting in better FT resistance. However, the different laboratories in which the specimens were tested showed significant deviations in behavior of the specimens. It was concluded that curing conditions highly influenced the outcome of the experiments (Winnefeld et al., 2020).

Another research mentioned that convenient characteristics that can be used to measure FT resistance is dynamic modulus of elasticity, compressive strengths and change of mass. This review also states that it is likely that frost resistance is largely dependent on physicommechanical mechanics. Alkali activated materials have shown better performance than Portland cement concretes exposed under the same circumstances as stated by Rostovskaya et al. Kukko and Mannonen and Bin and Pu have also noted good frost resistance in slag based alkali activated concrete and explained this by low total porosity and small pore radius. Gifford and Gillott found that FT of slag based alkali activated concrete was dependent on air content and air bubble distribution, where performance was at least as good as Portland concretes. Talling and Krivenko stated that use of SS improved FT performance while specimens with SH failed earlier and performed worse than OPC (Provis et al., 2009).

This research tested early strength and durability of metakaolin-based alkali activated concrete. Here, FT was performed for 30 FT cycles according to ASTM C666 where mass losses were ranging from 1.29 % to 6.86 %. The research concluded that higher water content results in less FT resistance combined with the fact that it seemed that the MK concrete is less durable than conventional concrete. Air entrained concrete is one of the recommendations to improve durability for MK concrete. What is interesting to observe is that the two mixes with lowest mass losses were the ones where $\text{SiO}_2/\text{Na}_2\text{O}$ was 1.0, whereas less performing mix designs had a ratio of 1.4. In terms of compressive strength, a ratio of 1.0 seems optimal as well. Including slag in the mix design reduced in compressive strength due to different levels of C-A-S-H formation. Slag increased workability (Alanazi et al., 2017).

This research evaluated performance of blended metakaolin/blast furnace slag mortars. Especially interesting was the observation that the introduction of metakaolin into the system led to an increase of porosity as measured via the saturation test and MIP. Due to the stickiness of the

metakaolin mixtures, compaction was more difficult which could explain the increase of porosity with increase of metakaolin content. This trend is also found by Zhang et al., but in that study it is concluded that the C-A-S-H gel in the microstructure densified the pore structure. As water absorption and porosity combined are important parameters for freeze-thaw resistance, it can be concluded that introduction of calcined clay might increase porosity and therefore weaken the overall mortar resulting in lower freeze-thaw performance (Borges et al., 2016).

Concluding

Table 2.2 below summarize the observations from literature.

Table 2.2 - FT observations from literature

Research	Observations	Experiments performed
Fu et al.	Packing of mixture is very important. Less pores is better FT resistance due to impermeability.	Dynamic elasticity Compressive strength
Ugurlu et al.	More binder yields better FT performance as it allows for less pores to be formed. Binder varied from 300-500 kg/m ³	SEM Compressive strength Weight loss
RILEM round robin test	Higher strength mixtures tend to perform better in FT resistance. Curing conditions are important.	Compressive strength Weight loss
Provis et al.	Experimentation of SS/SH ratio, also known as the modulus, is important. Low porosity of a specimen is better (impermeable).	-
Alanazi et al.	Clays are most likely to reduce workability. Higher water content yields lower FT resistance. Air entrainment improved FT performance.	Compressive strength Weight loss
Borges et al and Zhang et al.	Introduction of metakaolin increases porosity and decreases C-A-S-H densification of microstructure	Saturation test MIP

2.5. Characteristics of alkali activated sulfate attack resistance

Water and ground contain all kinds of soil which may introduce salt crystallization or chemical sulphate. This might result in slowly decaying concrete, thus the concrete should be sufficiently resistant to sulfate attack.

Definition, requirement and relevance

Definition: Sulfate attack describes physical salt attack due to salt crystallization and chemical sulfate attack by sulfates from soil, groundwater or seawater.

Requirement: There is no specific requirement, but studies focus on mass loss and compressive strength.

Relevance: Sulfate attack is a deterioration system that can lead to expansion, cracking and strength loss of concrete. It is generally attributed to the reaction of sulfate ions with calcium hydroxide and calcium aluminate hydrate which form ettringite and gypsum. With the formation of

the latter two, the material expands causing internal and external cracks, mass loss and decrease in strength. Sulfate attack can be caused due to sulfates from soil, groundwater or seawater. As concrete revetment products are situated on top of soil and exposed to seawater, increasing the concrete resistance to sulfate attack increases durability of the material resulting in a longer lifespan in which it can be utilized.

Sulfate attack literature review

Sulfate attack of alkali activated has been investigated in a number of articles. An overview and observations from these articles is provided.

Generally speaking, sulfate attack on alkaline activated materials is described as the resistance of the alkaline activated materials towards either a Na_2SO_4 solution or MgSO_4 solution. When ground granulated blast furnace slag is the main precursor, the resistance towards Na_2SO_4 solution is generally better than the resistance to MgSO_4 . The C-A-S-H inside the alkaline activated slag undergoes extensive decalcification and silicate polymerization when attacked by MgSO_4 solution. This suggests formation of magnesium-aluminosilicate-hydrate (M-A-S-H) and/or silica gels, which have little contribution towards mechanical properties. When exposed to Na_2SO_4 solution, only little changes are observed at the C-A-S-H with little precipitation of gypsum and ettringite (Ye et al., 2019).

A different research mentions that main reaction products from slag-based alkali activated paste is characterized by low calcium-oxide-to-silica-oxide ratio (CaO/SiO_2) which justifies their stronger chemical stability against sulfate attack. The study used SS and SH as activators for the slag with equal sodium oxide concentration (4 % in slag mass). It is concluded that SS activated mortars presented high stability against sulfates whereas SH mortars were the most sensitive where formation of ettringite and gypsum was observed. However, this formation was scarce resulting in a low decrease of mechanical strength (Puertas et al., 2002).

This research investigated sulfate attack resistance of slag-alkali activated concrete with recycled concrete aggregates (RCA). 10M NaOH solution and sodium silicate with Sodium oxide to Silica oxide ratio of 3 is used as activators. RCA was used with GGBFS binder of 300-400-500kg/m³. Specimens were immersed in 3 % MgSO_4 and 1.5 % MgSO_4 + 1.5 % Na_2SO_4 for 12 weeks. Compressive strength, UPV and weight change were observed along with SEM.

Increase of binder content yielded less losses and decreased permeability of the specimens which explains the better resistance against sulfate attack. Adding more RCA yielded less resistance due to more water absorption and presence of two ITZs. UPV results could also directly be linked to the above stated observations. MgSO_4 has larger influence on sulfate resistance than Na_2SO_4 (Ugurlu et al., 2021).

Another research states that the key difference between ordinary Portland cement (OPC) and alkali activated degradation under sulfate attack are related to the role of decalcification of the C-S-H type binder phases and formation of degradation products in OPC. It mentions that change of properties of slag alkali activated concrete under sulfate attack is measured using UPV, compressive strength, elastic modulus or resonance frequency analysis. According to the book of She et al. the performance of alkali activated concrete under sulfate attack depends strongly on the chemistry of the source material, type of activator and composition and concentration of sulfate solutions that are used for testing (Provis et al., 2009).

According to this paper, testing external attack of sulfate on mortar or concrete is the most realistic setup as it tests the entire system, where porosity and permeability properties are also taken into account. There is scope for outcomes from laboratory testing of pastes and mortars, but in the end the test should be on concrete (Provis & van Deventer, s.d.).

This research developed a sulfate resistance test on the basis of ASTM C1012, applied to slag alkali activated concrete. The specimens were immersed in either 50 g/L Na_2SO_4 or 50 g/L MgSO_4 solution. Compressive strength was tested after 12 months and compared to reference specimens which were stored in potable water. The results was that slag alkali activated concrete performed better when immersed in Na_2SO_4 compared to Portland cement concrete, but equal to Portland cement concrete in the MgSO_4 solution. The latter caused gypsum formation and decomposition of the C-S-H (Provis & van Deventer, s.d.), (Bakharev et al., 1998).

The research also states that magnesium leads to decalcification of calcium rich gel phases present in alkali activated systems causing degradation of the binder system and precipitation of gypsum. The water to binder ratio appears to contribute to resistance to degradation where a low W/B densifies the binder resulting in a reduction of attack onto the binder system (Ismail et al., 2012).

This research studied the influence of Si/Al ratio on sulfate durability of MK-based alkali activated paste. They used SS and SH, where the SS had $\text{SiO}_2/\text{Na}_2\text{O}$ of 3. The mix design contained different Si/Al ratio's, namely 2 and 2.25, and a water/solid ratio of 0.7 was used based on other literature studies for good mechanical strength and workability. Both in sodium sulfate solution and magnesium silicate solutions there were no significant reductions in mechanical properties, however the Si/Al had more influence in the magnesium sulfate solution as the 2.25 ratio had triple the strength increase during the exposure period compared to the 2.00 ratio specimen (Chen et al., 2020).

Concluding

Table 2.3 below and on the next page summarizes the observations from literature.

Table 2.3 - Sulfate attack observations from literature

Research	Observations	Experiments performed
Puertas et al.	SS has better sulfate attack resistance than SH. Used Na_2SO_4	Compressive strength
Ugurlu et al.	Increase of binder decreased losses and permeability. MgSO_4 has more influence on degradation	Compressive strength UPV Weight change SEM
Provis et al.	External attack of sulfate should be tested.	Useful experiments: Compressive strength UPV Elastic modulus
Bakharev et al.	Slag based alkali activated paste performs equal or better than portland cement concrete. Gypsum formation degraded strength.	Compressive strength
Ismail et al.	Alkali activated pastes are more sensitive to MgSO_4 and a lower W/B ratio improves sulfate attack resistance	XRD FTIR TGA SEM

Chen et al.	On MK mixtures, Si/Al has influence on outcome with good results with a ratio of 2.25. MgSO ₄ has more influence on degradation	Compressive strength pH XRD FTIR SEM
-------------	--------------------------------------------------------------------------------------------------------------------------------------------	--------------------------------------------------

2.6. Summary and conclusion

The literature study shows that the type of activator and the ratio between the activator types is important for development of mechanical properties and formation of microstructure.

For freezing and thawing, internal damage and scaling are separate degradation mechanisms. For preventing internal damage, the mortar/concrete should have sufficient air voids to relief build-up of internal pressure caused by crystallization during freezing. For preventing scaling, it is important to prevent large interconnected pores and to have low permeability, whilst preventing bleeding as much as possible such that the outer surface is stronger.

Alkaline activate mortar containing GGBFS is sensitive for sulfate attack when exposed to high concentration MgSO₄ solution as this can cause formation of M-A-S-H (with no mechanical strength) and formation of ettringite and gypsum which both cause expansion with possible internal cracking, especially when the mortar is activated with sodium hydroxide.

For both sulfate attack and freezing and thawing the precursor content and composition is also very important, as higher precursor content results in a less permeable mortar. However, the activator used in combination with the precursor defines the internal pore structure which is very important for good freeze-thaw resistance.

This research attempts to address multiple gaps and therefore makes important contributions.

First, this research extends the limited research on freeze-thaw deterioration mechanisms for alkali activated mortar. This research is one of the first that utilizes a combination of both GGBFS and CC in the precursor blend to identify the effect of CC on freeze-thaw resistance.

Second, the research is unique in determination of freeze-thaw performance for dry-cast alkali activated concrete and in observing the difference in performance under freezing and thawing when upscaling the mortar to a dry-cast concrete.

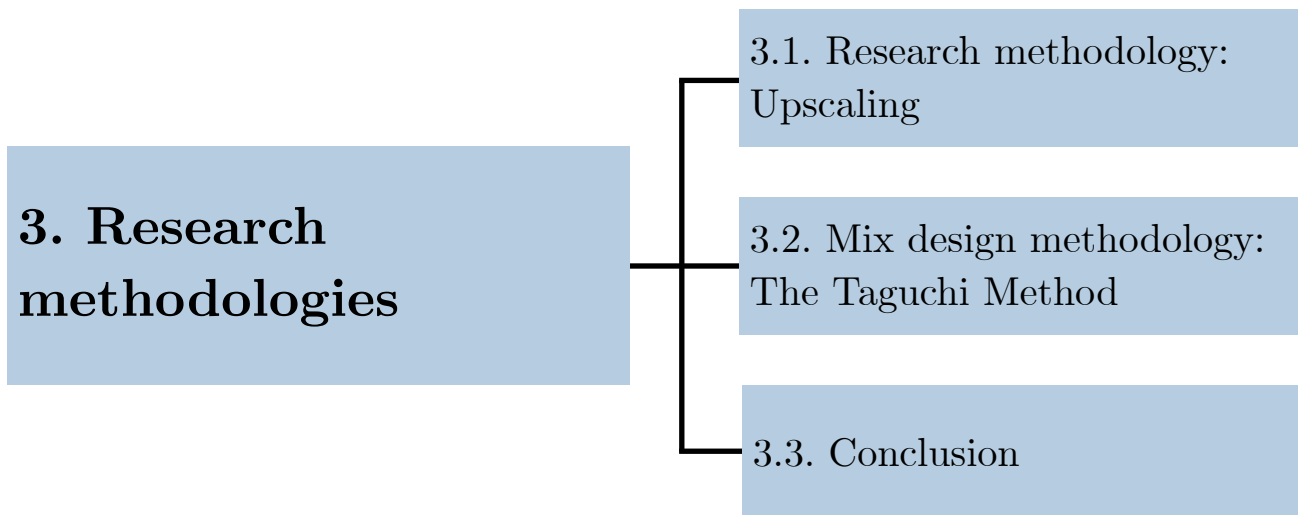
Third, the research expands the ongoing research of sulfate attack resistance of alkali activated mortar by identification of the effects of both GGBFS and CC in the precursor blend.

For the first two contributions, this research uses a combination of water absorption, compressive strength capacity, scaling and visual observation for both the alkali activated mortar and concrete to couple the two principal mechanisms to the performance. For the third contribution, this research uses a combination of visual observation and compressive strength capacity to couple the mechanisms to performance.

The above provided information results in a clear direction for mix design, such that the methodology, used materials and mix designs can be developed.

3.

Research methodologies



This chapter elaborates the research and mix design methodologies that are used in this research in sections 3.1 and 3.2 respectively. Section 3.3 concludes the chapter.

3.1. Research methodology: Upscaling

In order to find a suitable concrete mix design that has equal performance but lower environmental impact than blast furnace cement based concrete and to investigate the deterioration mechanisms of freezing and thawing in alkali activated systems, an upscaling methodology is used.

Upscaling consists of the three methodological steps is visualized in Figure 3.1 below:

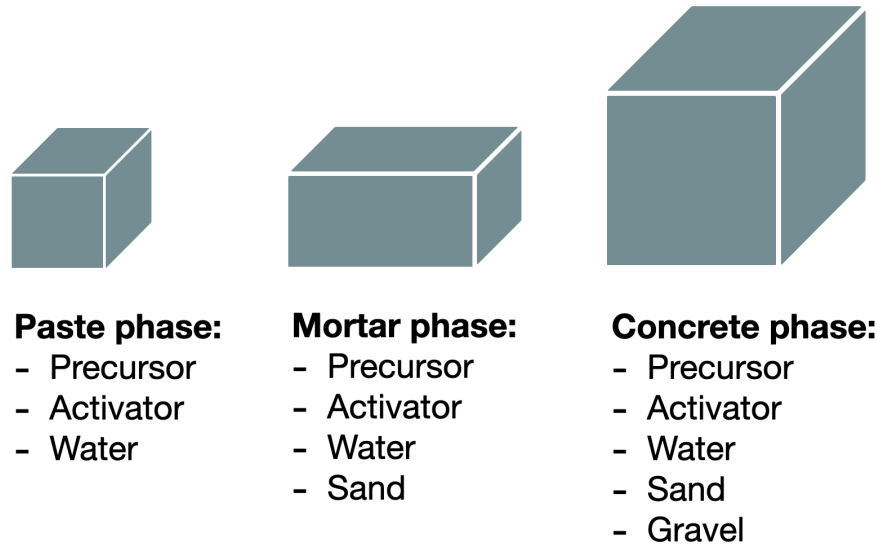


Figure 3.1 - Methodological upscaling visualized

Paste phase

Purpose

The paste phase is used to determine basic material behavior and mechanical properties.

Recipe

The paste phase contains a total of 9 mix designs, consisting of precursors, activators and potable water.

Program

The paste phase tests the following properties:

1. Workability
2. Flexural strength
3. Compressive strength

Results

The paste phase provides information about the strength of the paste and what the influences of the mix design parameters and materials are on the behavior of the paste. With this information in mind, combined with the mix design methodology, it enables to determine if the chosen mix designs are suitable for upscaling towards mortar.

Mortar phase

Purpose

The mortar phase has two goals, based on the list of requirements:

1. Find the most suitable mixtures for upscaling to concrete in terms of performance and environmental impact.
2. Determine deterioration phenomena due to freezing and thawing cycles in alkali activated systems.

Recipe

The mortar phase contains 9 mix designs, consisting of precursors, activators, potable water and sand. The binder composition of these 9 mix designs is equal to the paste phase.

Program

The mortar phase tests the following properties:

1. Workability
2. Flexural strength
3. Compressive strength
4. Water absorption
5. Surface water absorption
6. Freeze-thaw resistance
7. Sulfate attack resistance

Results

The mortar phase provides information about the strength of the mortar and what the influences of the mix design parameters and materials are on the behavior of the mortar. The effects of upscaling on mechanical properties can be determined to see if these are still satisfactory. From the results of the mortar phase, it is clear which mix designs are most promising which can then directly be recalculated into a concrete mix design.

Concrete phase*Purpose*

The two best performing mix designs at mortar scale are upscaled to concrete mix designs and tested in accordance to the norms to validate if the developed concrete meets the set requirements. These two mix designs are also directly compared to an industry-provided blast furnace slag cement mix design.

Recipe

One mix design is based on the lowest environmental impact with the expectation to reach the requirements. The other mix design is based on maximum performance, but still reasonable environmental impact compared to cement-based concrete.

Program

The concrete phase tests the following properties:

1. Workability
2. Compressive strength
3. Water absorption
4. Freeze-thaw resistance

Results

If in the end a mix design meets the set of requirements and provides lower environmental impact than the industry-provided mix design, it is considered suitable for utilization in concrete revetment products within the scope of this report.

3.2. Mix design methodology: The Taguchi method

The Taguchi method is used in this study for determination of the influence of a lot of mix design parameters, compacted to 9 mix designs which is also known as a Taguchi L9 array.

Experimental design using the Taguchi orthogonal array approach aims to solve problems by optimizing the number of experiments that need to be conducted. This method is designed by Dr. Genichi Taguchi.

The goal of experimental design is to have accurate and realistic information about the input and output variables of experiments. When including more and more mix design parameters, the number of experiments required increases and so does the amount of time, the cost and the amount of materials. Taguchi developed a method where an entire process can be studied using minimum balanced trials, also known as orthogonal arrays.

The effect of Taguchi becomes apparent when comparing it to full factorial design. In this study, 4 different factors (mix design parameters) are used which are varied at 3 levels (a low, medium and high value). Using full factorial design, a total of 3^4 or 81 mix designs need to be made to determine the effect of each individual mix design parameter and its levels. Taguchi minimizes this number to only 9 mix designs that need be conducted in order to determine the effect of the same 4 mix design parameters and its 3 levels with trends. This method saves a significant amount of time, resources and cost and is therefore used in this research project (Dar & Anuradha, 2018).

The following steps are involved in the Taguchi method (Fraley et al., 2021):

1. Define process objective.
2. Determine the design parameters that affect the process. In this case, these parameters are the parameters that are expected to greatly influence the compressive strength of alkali activated systems (water-to-binder ratio (W/B), the amount of ground granulated blast furnace slag relative to the amount of calcined clay (precursor ratio), sodium oxide percentage and the modulus (Ms)).
3. Creation of orthogonal arrays for the design parameters, including number and conditions for each experiment. The exact orthogonal array configuration depends on the number of design parameters and the level of variation (This research uses a L9 configuration, so 4 mix design parameters that are varied at 3 different levels).
4. Conduct experiments and collect data on the effect of the performance measure (e.g. compressive strength).
5. Complete data analysis to determine the effect of each design parameter on the performance measure (usually referred to as Taguchi trends).

1. Define process objective

In the case of this research the process objective is a performance measure like compressive strength. In most situations in this research, maximum compressive strength is determined to be the process objective.

2. Determine mix design parameters

This research uses 4 mix design parameters which are varied at three different levels, see Figure 3.2 on the next page.

W/B ratio	Precursor ratio	Sodium oxide %	Modulus
Low	Low	Low	Low
Medium	Medium	Medium	Medium
High	High	High	High

Figure 3.2 - The 4 different mix design parameters and three levels of variation used in Taguchi mix design

3. Creation of orthogonal arrays

These mix design parameters and levels of variation are then translated towards a so called Taguchi L9 array, which uses these exact mix design parameters and their level of variation as input. This Taguchi L9 array is shown in Figure 3.3 below.

Mix number	W/B ratio	Precursor ratio	Sodium oxide %	Modulus
Mix 1	Low	Low	Low	Low
Mix 2	Low	Medium	Medium	Medium
Mix 3	Low	High	High	High
Mix 4	Medium	Low	Medium	High
Mix 5	Medium	Medium	High	Low
Mix 6	Medium	High	Low	Medium
Mix 7	High	Low	High	Medium
Mix 8	High	Medium	Low	High
Mix 9	High	High	Medium	Low

Figure 3.3: Taguchi L9 array used in this research

In Figure 3.3 the complete Taguchi L9 configuration as used in this research can be seen. The L9 array consists of 9 different mix designs, where a total of 4 design parameters are varied on 3 different levels. Mix 4 in Figure 3.3 above further elaborates how the mix designs are composed. Mix 4 uses:

- W/B ratio = Medium value
- Precursor ratio = Low value
- Sodium oxide % = Medium value
- Modulus = High value

4. Experimental data collection

Besides the mix designs themselves, Taguchi is in this case also used to find trends in the performance of the mix designs relative to the mix design parameters.

In this example, compressive strength is measured as performance parameter. Compressive strength measurements require 3 results per experiment. The result of each experiment can be described with T_{ij} where i = mix design number and j = trial number. The Taguchi table including experimental results then looks like Table 3.1 on the next page.

Table 3.1 - Taguchi L9 array with results

Mix number	W/B ratio	Precursor ratio	Sodium oxide %	Modulus	Result 1	Result 2	Result 3
1	Low	Low	Low	Low	T _{1,1}	T _{1,2}	T _{1,3}
2	Low	Medium	Medium	Medium	T _{2,1}	T _{2,2}	T _{2,3}
3	Low	High	High	High	T _{3,1}	T _{3,2}	T _{3,3}
4	Medium	Low	Medium	High	T _{4,1}	T _{4,2}	T _{4,3}
5	Medium	Medium	High	Low	T _{5,1}	T _{5,2}	T _{5,3}
6	Medium	High	Low	Medium	T _{6,1}	T _{6,2}	T _{6,3}
7	High	Low	High	Medium	T _{7,1}	T _{7,2}	T _{7,3}
8	High	Medium	Low	High	T _{8,1}	T _{8,2}	T _{8,3}
9	High	High	Medium	Low	T _{9,1}	T _{9,2}	T _{9,3}

5a. Formation of signal-to-noise ratio from collected data

With these results known, it is time to determine the effect of each design parameter on the compressive strength. To do this, the signal-to-noise ratio (S/N) needs to be calculated. The S/N represents the quality of the results relative to uncontrollable factors. Simply said: it indicates how well a mix design performs based on the average outcome and the variance of each mix design. The higher the S/N value, the more preferred the mix design is for the parameter that is investigated.

It is important to know that in most cases in this research, the aim is to maximize the output. In this example, it is the aim to maximize compressive strength. In the equations below, $y_{i,j}$ is the output value of experiment number i, trial j.

The S/N for maximizing the output be determined using the following equation:

$$S/N_i = -10 \cdot \log\left(\frac{1}{N_i} \cdot \sum_{j=1}^n \left(\frac{1}{y_{i,j}^2}\right)\right) \quad (1)$$

Where:

i = experimental number

j = trial number

N_i = number of trials for experiment i

When determining compressive strength performance, the results are desired to be maximized.

However, the equation for maximizing the results only considers individual values of the experiment and only allows positive data. While zero results can occur the excel that is used for determination of the S/N will return a 0 value for S/N when there is no compressive strength, meaning that the specific mix composition should not be considered for good compressive strength development.

5b. From signal-to-noise ratio towards influence of each mix design parameter on performance

After calculating the S/N for each experiment, it is time to determine the average S/N for each mix design parameter and its levels. For this, Table 3.2 on the next page is used.

Table 3.2 - Taguchi L9 array with S/N factors

Mix number	W/B ratio	Precursor ratio	Sodium oxide %	Modulus	S/N
1	Low	Low	Low	Low	S/N_1
2	Low	Medium	Medium	Medium	S/N_2
3	Low	High	High	High	S/N_3
4	Medium	Low	Medium	High	S/N_4
5	Medium	Medium	High	Low	S/N_5
6	Medium	High	Low	Medium	S/N_6
7	High	Low	High	Medium	S/N_7
8	High	Medium	Low	High	S/N_8
9	High	High	Medium	Low	S/N_9

To determine the influence of each level of variation of the sodium oxide % (mix design parameter 3), the following average S/N values are determined (from top to bottom: lowest sodium oxide % value, medium value and highest value):

$$S/N_{P3,1} = \frac{S_{N1} + S_{N6} + S_{N8}}{3} \quad (2)$$

$$S/N_{P3,2} = \frac{S_{N2} + S_{N4} + S_{N9}}{3} \quad (3)$$

$$S/N_{P3,3} = \frac{S_{N3} + S_{N5} + S_{N7}}{3} \quad (4)$$

After determination of the S/N for all design parameters and each level, they can be tabulated as shown in Table 3.3 below.

Table 3.3 - Overview of S/N per parameter, Δ and rank

Level	Parameter 1 (P1)	Parameter 2 (P2)	Parameter 3 (P3)	Parameter 4 (P4)
1	$S/N_{P1,1}$	$S/N_{P2,1}$	$S/N_{P3,1}$	$S/N_{P4,1}$
2	$S/N_{P1,2}$	$S/N_{P2,2}$	$S/N_{P3,2}$	$S/N_{P4,2}$
3	$S/N_{P1,3}$	$S/N_{P2,3}$	$S/N_{P3,3}$	$S/N_{P4,3}$
Δ
Rank

With the S/N per parameter, the delta, or Δ , can be determined. The delta describes the range of the S/N for that parameter (Δ = highest S/N of mix design parameter minus lowest S/N of mix design parameter). The higher the delta is, the more influence the design parameter has on the compressive strength and the higher the expected outcome of compressive strength is when using the mix design parameter coupled to the S/N value. This is due to the fact that a change in signal causes large effect on the output variables.

Based on the delta, each parameter can be ranked. Highest rank means that the parameter has the most influence on compressive strength performance. Lowest rank means that the parameter has least influence on compressive strength performance.

5c. Formation of performance trends

The results of the experiments can be plotted per parameter to visualize the effects of each mix design parameter on the performance characteristic (e.g. compressive strength) concerned. This provides valuable information, because it allows for better elaboration why certain values of the mix design can make the paste/mortar/concrete perform good or bad. To elaborate this, four graphs are presented in Figure 3.4 below.

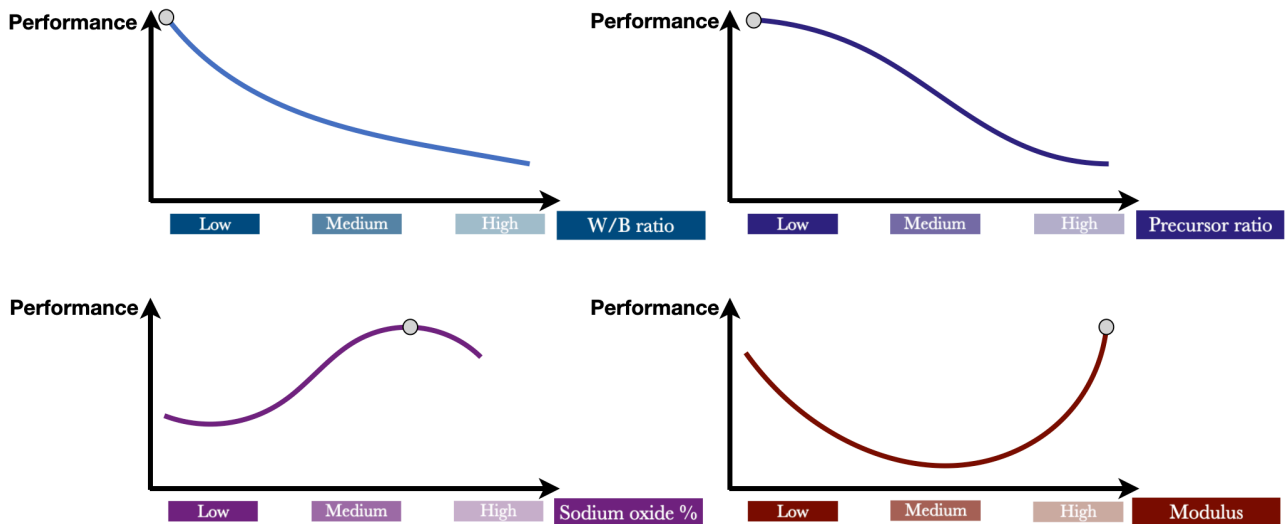


Figure 3.4 - Taguchi trends

Figure 3.4 shows the four mix design parameters used in this report along with made-up Taguchi trend lines. As can be seen, for each of the mix design parameter there is a maximum value for each trend. In this example, the maximum S/N value for the W/B ratio (top left graph) is situated at the “low” W/B ratio value. This means that if the W/B ratio is low, it is likely that the product performs well in the aspect that is evaluated (e.g. compressive strength development). For the sodium oxide % (bottom left graph), the optimum value to use for maximum development/performance is situated between the medium and high value.

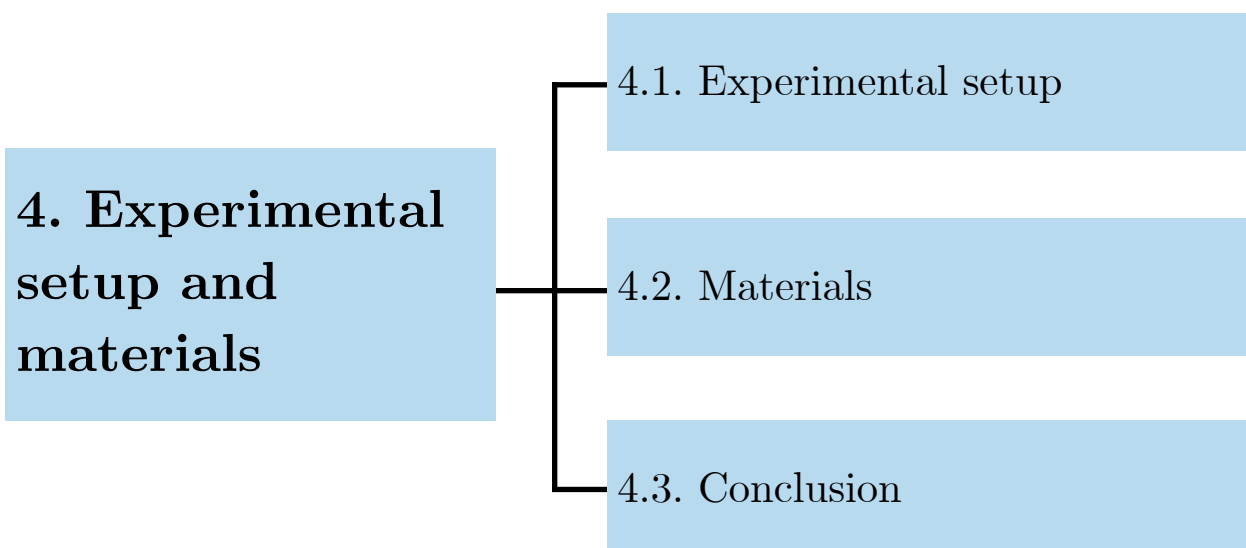
So, besides providing a minimization of mix designs for a large range of mix design parameters, Taguchi also enables to indicate what combination of mix design parameters and their respective value can optimize the mix design. Therefore, the above exemplified Taguchi trends are also presented at research results that are presented as either compressive strength or flexural strength.

3.3. Conclusion

With the upscaling methodology, changes of material properties on conducted experiments are better explained, which increases the efficiency of the development of the final concrete.

Taguchi method provides trends for experimental results, where the performance of the concrete is related to the mix design parameters. The combination of these methodologies and the results of the literature review in chapter 2 enables to describe the experimental setup and used materials in chapter 4.

Experimental setup and materials



The setups of the executed experiments, and used materials are presented in this chapter. Section 4.1 contains the experimental setups used to determine basic material properties. For the experimental setups, only a short description is provided except when the experimental procedure is adapted from the norm. A detailed description of each experiment can be found in appendix H. Section 4.2 continues with the used materials. Finally, section 4.3 concludes the chapter.

4.1. Experimental setup

4.1.1. Aggregate shape

Scope

Determine the shape and flakiness index of the coarse aggregates groups.

Method

This research uses the flakiness and shape index with help from particle classifications of the British standard in order to determine the shape of the aggregates, BS812: Part 1: 1975.

Relevance

When working with unknown aggregates it is important to determine their shape in order to know their external characteristics which influence the behavior in a mix design. For example, particles with a lot of visible pores are more likely to absorb more water and flaky particles tend to be orientated in one plane with greater possibility of bleeding as a result.

4.1.2. Aggregate particle size distribution

Scope

Determine the efficiency of the current particle size distribution of industry provided aggregates by measuring and combining results from the most used aggregates and the amount that is currently used in each cubic meter of concrete.

Method

The particle size distribution is performed in accordance with NEN-EN 933-1:2012 (E).

Relevance

Determining the particle size distributions (PSD) of each aggregate heap used in a concrete mixture is measured as it influences the particle composition within the concrete mix.

4.1.3. Aggregate water absorption and specific gravity

Scope

Determination and comparison of water absorption and specific gravity of all used aggregate groups.

Method

This experiment is in accordance with NEN-EN 1097-6:2013, where a modified pycnometer method is used.

Relevance

The amount of water that is used in any type of concrete mixture is measured as it affects its strength and resistance to durability affecting parameters.

4.1.4. Precursor and activator composition

Scope

Determine the chemical composition of the precursors and activators used in this research.

Method

For SS and SH, only the information provided by the manufacturer is needed to determine its chemical composition. For GGBFS and CC X-ray Fluorescence or XRF needs to be used. XRF is an analytical method to determine elemental composition of materials and phase composition

(Masone, 2015). XRF needs only small specimens and is non-destructive. However, this data is available for both materials and will therefore not be repeated specifically for this research.

Relevance

Determination of the chemical composition of GGBFS, CC, SS and SH is these four components combined form the binder of the alkali activated concrete. The chemical composition significantly influences the mixture composition that should be used.

4.1.5. Workability

Scope

Determination of the workability of alkali activated paste/mortar/concrete mix designs.

Method

Paste phase:	NEN-EN 1015-3
Mortar phase:	NEN-EN 1015-3
Concrete phase:	EN 12350-4

Relevance

Workability is very important when it comes to earth moist concrete. In the end, the mixture should have workability which complies with earth moist concrete, namely consistency class C1. The workability of all three upscaling steps is determined to observe the influence of mix design parameters on workability.

4.1.6. Flexural strength

Scope

Determination of flexural strength development of alkali activated paste/mortar mix designs after 1, 7 and 28 days of curing.

Method

Paste phase:	NEN-EN 1015-11:2019
Mortar phase:	NEN-EN 1015-11:2019.

Relevance

Flexural strength of concrete is important as this introduces tensile stress in the concrete. Concrete generally has a flexural strength capacity which is only 10 % of its compressive strength capacity.

4.1.7. Compressive strength

Scope

Determination of compressive strength development of alkali activated paste/mortar/concrete mix designs after 1, 7 and 28 days of curing.

Method

Paste phase:	NEN-EN 1015-11:2019
Mortar phase:	NEN-EN 1015-11:2019
Concrete phase:	NEN-EN 12390-3:2019 (E)

Relevance

Compressive strength describes the strength of the concrete and it is used through this whole research.

4.1.8. Water absorption

Scope

Mortar phase: Determination of water absorption and voids content for coupling theory to deterioration mechanisms of alkali activated mortar.

Concrete phase: Determination of water absorption of alkali activated concrete and blast furnace cement concrete.

Method

Mortar phase: ASTM-C642

Concrete phase: NEN-EN 1338:2003, appendix E

Relevance

Water absorption can be connected to FT resistance as internal expanding water during a period of frost can cause failure to concrete. It also allows to couple the theory of microstructure formation and reaction kinetics towards the trends of mix design parameters, resulting in finding the origin of why certain mix designs or combinations of parameters result in poor or great freezing and thawing resistance in the case of alkaline activated mortar.

4.1.9. Surface water absorption

Scope

Determine the surface sorptivity of mortar specimens.

Method

No specific standard as experiment is very straight-forward

Relevance

The surface water absorption is of importance for freezing and thawing resistance, like regular water absorption. If surface sorption is high, this means specimens absorb fluids quickly and that the surface is permeable. When the fluid freezes, it expands and this is one of the possible mechanisms that explains scaling of specimens.

4.1.10. Freeze-thaw resistance

Scope

Mortar scale: Determination of effects of mix design parameters on freeze-thaw resistance of alkali activated mortar in order to evaluate trends in freeze-thaw resistance.

Concrete scale: Determine Freeze-thaw resistance of alkali activated concrete and reference concrete by means of measuring mass loss (scaling) according to the CDF method.

Method

Mortar scale: NVN-CEN/TS 12390-9:2016, modification of cube test and CDF test with de-icing solution.

Concrete scale: NVN-CEN/TS 12390-9:2016, CDF test with de-icing solution.

Test setup mortar

The mortar test method is an adaption from the norm, therefore the test setup and procedural steps are further elaborated on the next page.

Figure 4.1 below shows the testing setup used for this experiment.

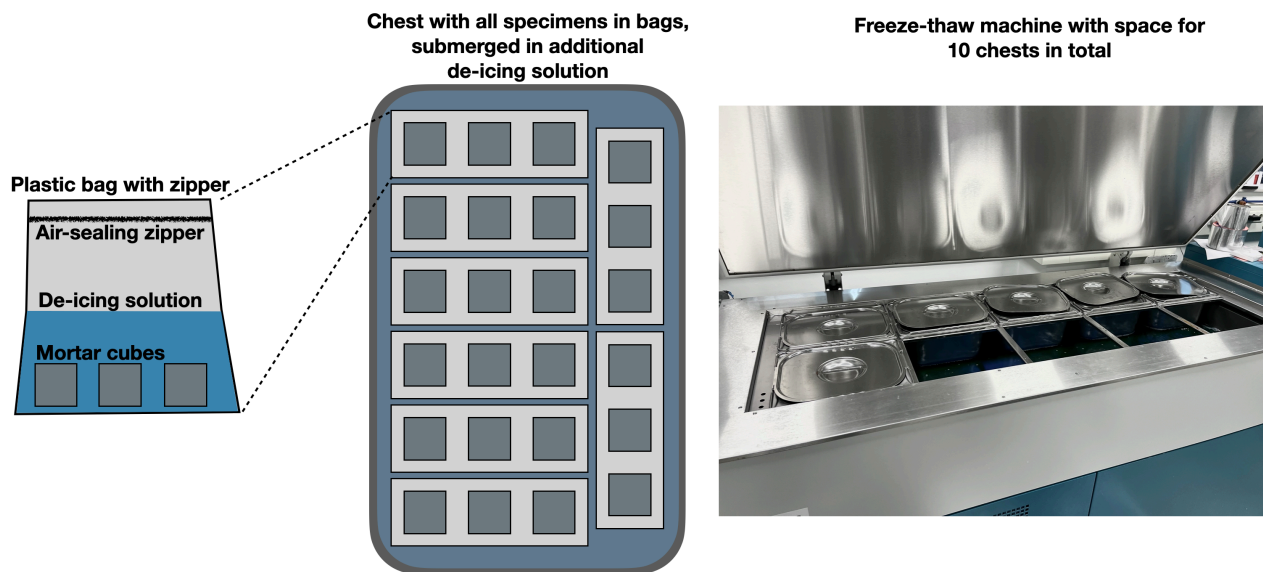


Figure 4.1 - Testing setup for Freeze-thaw resistance of alkali activated mortar

As can be seen in Figure 4.1, the testing setup consists of three parts:

1. The plastic bag which is freeze-resistant and includes a zipper for sealing the bag, preventing evaporation. This plastic bag contains three mortar cubes which are submerged in de-icing solution.
2. The freeze-thaw chest in accordance with the CDF test as described in NVN-CEN/TS 12390-9:2016, containing the 8 mixtures that are subjected to freezing and thawing. Around the bags, additional de-icing solution is added for better heat conduction.
3. The total freeze-thaw machine, containing 10 chests in total.

Process

The regular CDF test requires 150mm cubes of concrete to be partially submerged in water with or without de-icing salts after which the cycles are performed. However, since this part of the research focuses on mortar scale, making such huge cubes would both consume a lot of material and take up a lot of space. Besides, the goal of the freeze-thaw tests in mortar scale is to determine effects of mix design parameters on freeze-thaw resistance of alkali activated mortar in order to evaluate trends in freeze-thaw resistance. As no norm for mortar is available, the following process is used:

By casting 40 x 40 x 40 mm cubes and submerging them completely in the de-icing fluid, the trend of freeze-thaw performance should quickly be visible. In total there are four sets of cubes, which vary in their amount of cycles exposed to freezing and thawing: 2 cycles, 6 cycles, 14 cycles and 28 cycles. To further elaborate these trends, a reference mixture which is un-exposed to freezing and thawing is used.

A temperature controlled chest as described in NVN-CEN/TS 12390-9:2016 is used combined with the following procedural steps:

1. Cast the prisms and store them for 24h with foil on top to prevent evaporation.
2. After 24h, remove foil from prisms, demold prisms and place them in the curing room.
3. After 3 days in the curing room, saw the prisms into 40 x 40 x 40 mm cubes and completely immerse them in water to ensure full saturation. Put the cubes, immersed, in the curing room.

4. After another 3 days, remove the cubes from the curing room and put them in labelled plastic bags which can be sealed to prevent evaporation.
5. Add 3 % NaCl solution in the plastic bags until cubes are completely submerged.
6. Put the bags in the temperature controlled chest, fill the chest with de-icing solution and put the chest in the freeze-thawing machine.

The procedure is different for different durations of freeze-thaw. For all x cycle specimens, the goal is to identify trends in freeze-thaw performance via compressive strength and to determine the scaled material. Therefore, the following procedure is used after x cycles of freezing and thawing:

Procedure (following steps 1-6)

7. After 2, 6, 14 or 28 cycles in the freeze-thaw machine, remove the specimens from the testing setup.
8. Put specimens in ultrasonic bath and subject for ultrasonic cleaning for three minutes.
9. Open the bag and remove the cubes. The cubes are to be brought to saturated surface dry state by using drying towels.
10. Collect scaled material in filter paper using a cut in the plastic bag. Rinse several times with water until all scaled material is collected in the filter. Dry the filters with scaled materials at 110 degrees Celsius for 24h.
11. Weigh the specimens in saturated surface dry state and note result.
12. Test the freeze-thaw specimens for compressive strength.
13. Test the reference specimens for compressive strength.
14. After 24h, remove the dried filters and weigh them. Scaled material can now be determined and can also be validated using the two prior weight measurements (before FT in surface saturated dry state and after FT in surface saturated dry state).
15. Put results in table and graph.
16. Interpretation of results and discussion.

Relevance

Described in chapter 2.

4.1.11. Sulfate attack resistance

Scope

Determination of effects of mix design parameters on sulfate attack resistance of alkali activated mortar in order to evaluate trends in sulfate-attack resistance so that final concrete-scale mix designs can be created keeping these trends in mind.

Method

A modified testing method as described in NPR-CEN/TR 15697:2008 is used.

Test setup

Figure 4.2 below shows the testing setup used for this experiment.

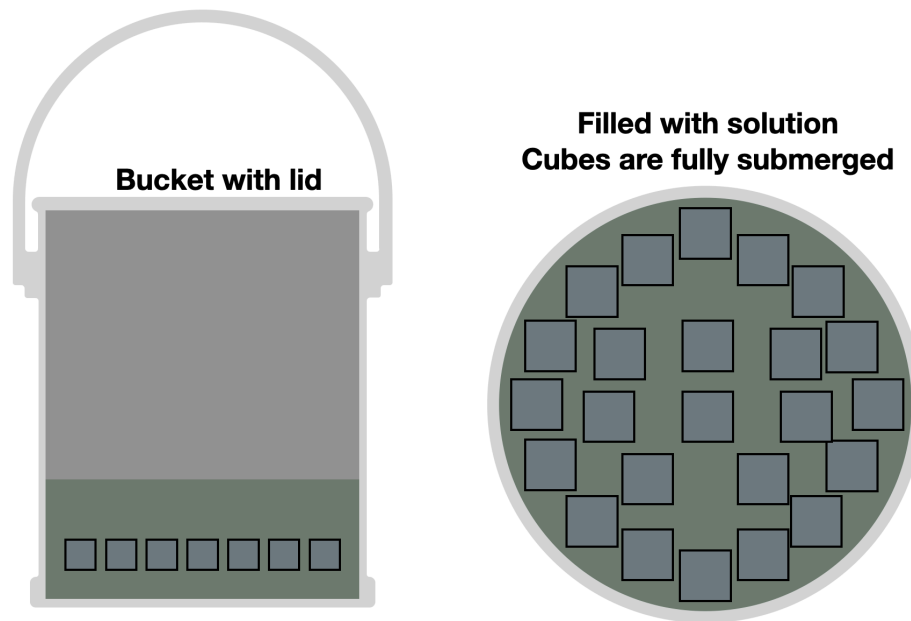


Figure 4.2 - Testing setup for sulfate attack resistance of alkali activated mortar

Process

By casting 40 x 40 x 40 mm cubes and submerging them completely in the MgSO_4 solution, the trend of sulfate-attack performance should quickly be visible. In total there will be four sets of cubes, which vary in their amount of days exposed to the solution: 1, 3, 7 and 14 days. To further elaborate these trends, a reference mixture which is un-exposed to sulfate attack is used.

The following procedural steps are performed:

1. Cast the prisms and store them for 24h with foil on top to prevent evaporation.
2. After 24h, remove foil from prisms, demold prisms and place them in the curing room.
3. After 3 days in the curing room, saw the prisms into 40 x 40 x 40 mm cubes and completely immerse them in water to ensure full saturation. Put the cubes, immersed, in the curing room.
4. After 7 days, remove cubes from water bath and dry (surface dry) and weigh them.
5. Put the cubes in the bucket with spacers underneath. Fill the bucket with MgSO_4 solution until the cubes are completely submerged.
6. Run SA for 1, 3, 7 and 14 days.
7. Once removed, dry cubes until the same surface dry state and weigh them, with any loose material removed.
8. Determine mass loss due to Sulfate attack.
9. Determine compressive strength of specimens.
10. Put results in table and graph.
11. Interpretation of results and discussion.

Relevance

Described in chapter 2.

4.2. Materials

4.2.1. Aggregate shape

Results

In total, 4 different aggregate types are provided by the industry which are combined together in the concrete phase. The aggregate shape is determined for three out of four aggregate types, as the test requires aggregates $> 4\text{mm}$ whereas the sand only contains particles in size $0 - 2\text{mm}$:

- Blast furnace slag (2/8 fraction, suitable for shape test after sieving with 4mm sieve)
- Copper slag (2/8 fraction, suitable for shape test after sieving with 4mm sieve)
- Sand (0/2 fraction, not suitable for shape test)
- Secondary concrete aggr. (0/10 fraction, suitable for shape test after sieving with 4mm sieve)

The result of the shape test is visible in Figure 4.3 below.



Figure 4.3 - Shape test results, from left to right: copper slag, secondary aggregate, blast furnace slag

A total of 100 particles from each aggregate group is ranked in terms of its shape. The shape and the amount corresponding with that shape is summarized in Table 4.1 below.

Table 4.1 - Flakiness and shape index results

Shape	Amount in copper slag	Amount in secondary aggregates	Amount in blast furnace slag	Total amount
Rounded	1	9	15	25
Angular	89	75	65	229
Elongated	6	14	19	39
Flaky	4	2	1	7

Interpretation of results

Different shapes in aggregates influence the behavior in a concrete mix. Angular particles require more water for better workability and elongated and flaky aggregates tend to be oriented in one plane with possible bleeding and air voids formation as a result (Neville, 2002). The particles researched are mostly angular and in the case of the secondary aggregate and blast furnace slag they also contain quite a lot of visible pores, meaning they will absorb more water than the other shapes. The elongated and flaky particles represent 15 % of the aggregate shape, so possible bleeding underneath these particles might occur. Not a lot of rounded shapes are found, but this is

logical as it are all crushed materials whereas rounded materials are expected when originating directly from natural resources (e.g. river sand).

Conclusion

Shape of aggregates is as expected for the type of material and therefore water demand is expected to be higher and is further verified with water absorption test of the aggregates in section 4.2.3.

4.2.2. Aggregate particle size distribution

Results

For the concrete trials, aggregates from TU Delft and the industry are used. From TU Delft, aggregates in different size fractions are used and the cumulative particle size distribution of these particles is matched to the industry specimen. The industry provides 4 aggregate types: blast furnace slag, copper slag, sand and secondary aggregates. These four aggregates are used in the concrete mix design in certain proportions, resulting in a particle size distribution which elaborates the distribution of the four combined aggregate groups in the composition it is used for casting concrete. The goal of the concrete trial is to determine which aggregate group is best used for casting of the final concrete specimens, either using TU Delft aggregates or industry provided aggregates. Therefore, the particle size distribution of the TU Delft aggregates is determined such that it matches the industry combined aggregate groups as identical as possible. The combined particle size distribution of the industry provided aggregates and the matched TU Delft aggregates particle size distribution can be found in Figure 4.4 below.

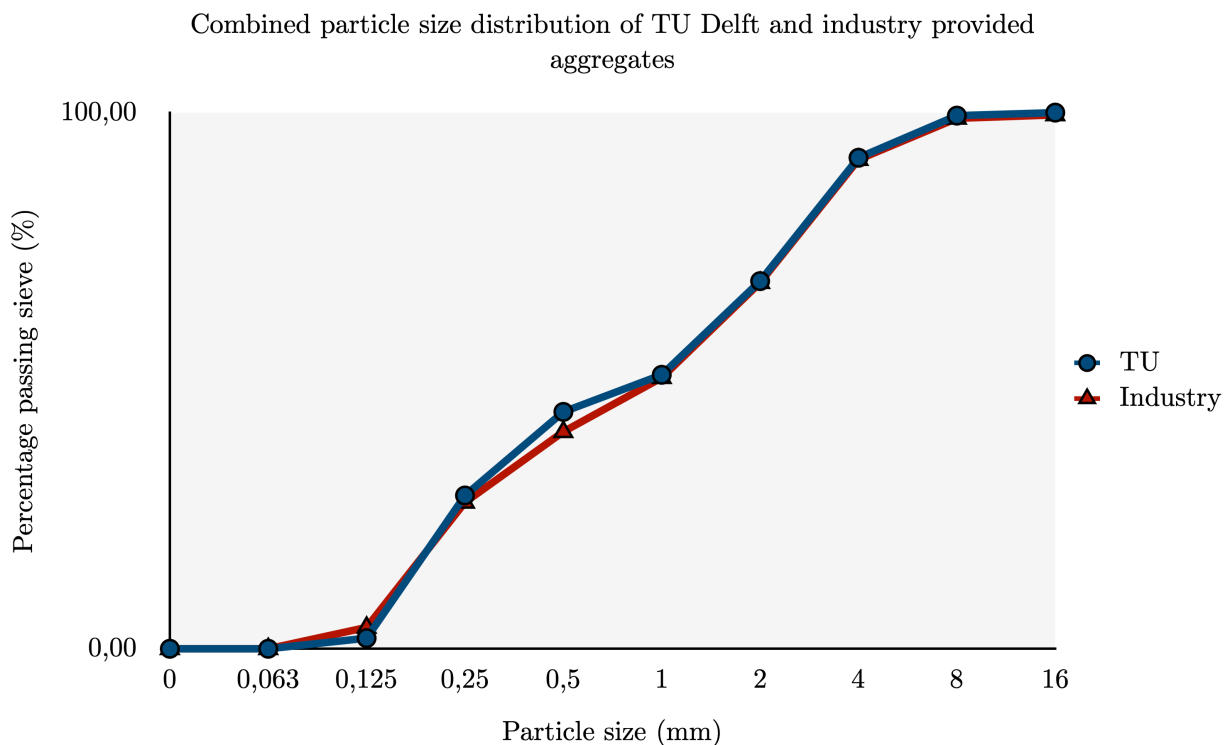


Figure 4.4 - Particle size distribution of used aggregates in final combined composition

Interpretation of results

As is clearly visible, particle sizes are 8 mm or lower which is in line with expectations as the industry uses aggregates up to 8 mm, with some small exceptions in the secondary concrete aggregate fraction, which goes up to size 10 mm. As most of the particles are 2 mm or less, packing of the aggregates is well suitable for earth moist concrete as determined with help of a file of the Betonvereniging (Betonvereniging, s.d.).

Conclusion

The combined particle size distribution is in line with expectations and has good packing for earth moist concrete.

4.2.3. Aggregate water absorption

Results

The water absorption for all industry provided aggregates is determined in accordance to the standard. This water absorption test also provides information concerning the specific gravity of the aggregate which plays an important role in mix design for volumetric calculations. The results can be observed in Table 4.2 below. Please note that the sand only contains results of the fine aggregates, up to 2 mm, as the sand contains no aggregates with size > 2 mm.

Table 4.2 - Water absorption and specific gravity of industry aggregates

Water absorption blast furnace slag - coarse aggregates						
Sample name	A	B	C	D	SG (kg/m ³)	WA (%)
BFS 1	646,7	439,9	358,1	344,5	2,28	4%
BFS 2	744,1	433,4	491,8	468,7	2,59	5%
Water absorption blast furnace slag - fine aggregates						
Sample name	A	B	C	D	SG (kg/m ³)	WA (%)
BFS 1	689,1	423,1	446,2	369,9	2,05	21%
BFS 2	690,1	453,1	447,1	365,4	1,74	22%
Water absorption copper slag - coarse aggregates						
Sample name	A	B	C	D	SG (kg/m ³)	WA (%)
BFS 1	799,1	441,2	493,1	492,4	3,64	0
BFS 2	921,6	446,2	642,8	638,7	3,82	1%
Water absorption copper slag - fine aggregates						
Sample name	A	B	C	D	SG (kg/m ³)	WA (%)
BFS 1	800,1	435,7	572,2	492,4	2,37	16%
BFS 2	787,8	446,2	539,5	470,6	2,38	15%
Water absorption sand - fine aggregates						
Sample name	A	B	C	D	SG (kg/m ³)	WA (%)
BFS 1	658,7	442,4	430,6	342,6	1,60	26%
BFS 2	641,4	432,3	394,1	313,5	1,69	26%
Water absorption secondary concrete aggregates - coarse aggregates						
Sample name	A	B	C	D	SG (kg/m ³)	WA (%)
BFS 1	623,9	434,8	357,7	343,5	2,04	4%
BFS 2	629,8	424,4	345,7	331,4	2,36	4%
Water absorption secondary concrete aggregates - fine aggregates						
Sample name	A	B	C	D	SG (kg/m ³)	WA (%)
BFS 1	596,6	435	407,3	290,6	1,18	40%
BFS 2	578,8	435	386,3	272,9	1,13	42%

Interpretation of results

It is immediately very apparent that the fines of all fractions have very high water absorption, in the region of 15 - 42 %. This means that when using the aggregates in completely dry state, a lot of additional water should be added to the mix to compensate for this absorption as the mix otherwise becomes too dry. As the aggregates are delivered in a slight moist state, it is recommended to use the aggregates in that state. This prevents the need to add excessive amounts of water and the need to dry hundreds of kilograms of material in the oven. However, moisture content should be monitored for constant results.

Conclusion

The water absorption of the aggregates is very high, especially for the secondary concrete aggregate. However, the material is delivered in a slightly moist state and is used that way to prevent addition of excessive amounts of water and to prevent the need to dry hundreds of kilograms of materials in the oven.

4.2.4. Precursor and activator composition

The composition of the following four materials:

- Ground Granulated Blast Furnace Slag
- Calcined Clay
- Sodium silicate
- Sodium hydroxide

are obtained by information provided by TU Delft and industry. The origin and composition are provided in Table 4.3, Table 4.4 and Figure 4.5 below.

Results

Table 4.3 - Compositional data of material

Material	Origin	Compositional data
Ground Granulated Blast Furnace Slag	Ecocem Benelux B.V.	See Figure 4.5/Table 4.4
Calcined Clay	Company in Belgium	See Figure 4.5/Table 4.4
Sodium silicate		44.1 % concentration and Ms = 2 (SiO ₂ = 29,3%, Na ₂ O 14,7%, H ₂ O = 56%)
Sodium hydroxide		10M, 50 % concentration

Table 4.4 - Oxide composition of GGBFS and CC in (%) (top) and mineral composition calcined clay (bottom)

	SiO ₂	Al ₂ O ₃	CaO	MgO	Fe ₂ O ₃	SO ₃	K ₂ O	TiO ₂	Other	LOI
GGBFS	31,77	13,25	40,5	9,27	0,52	1,49	0,34	0,97	0,21	1,31
CC	56,6	16,58	9,7	1,8	5,61	2,48	2,65	0,86	0,23	3,49
	Quartz (%)	Mullite (%)	Anatase (%)	Microcline (%)	Muscovite (%)	Illite (%)	Kaolinite (%)	Amorphous (%)	Density (g/cm ³)	
CC	27	5	< 0,5	5	2	6	2	53	2,42	

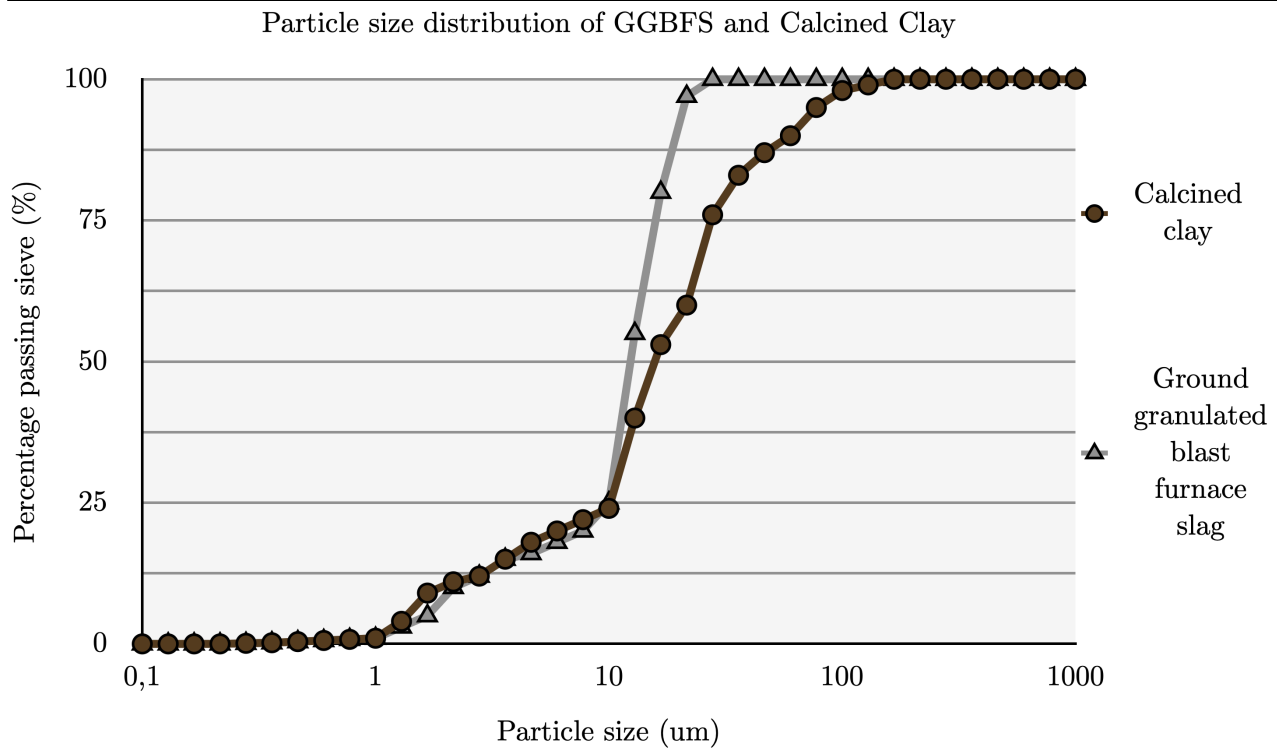


Figure 4.5 - PSD of GGBFS and CC

4.3. Conclusion

Most experiments follow the norm with the exception of freeze-thaw on mortar level and sulfate attack on mortar level, where the explanation of the used method is expanded to account for adaptations to the norm.

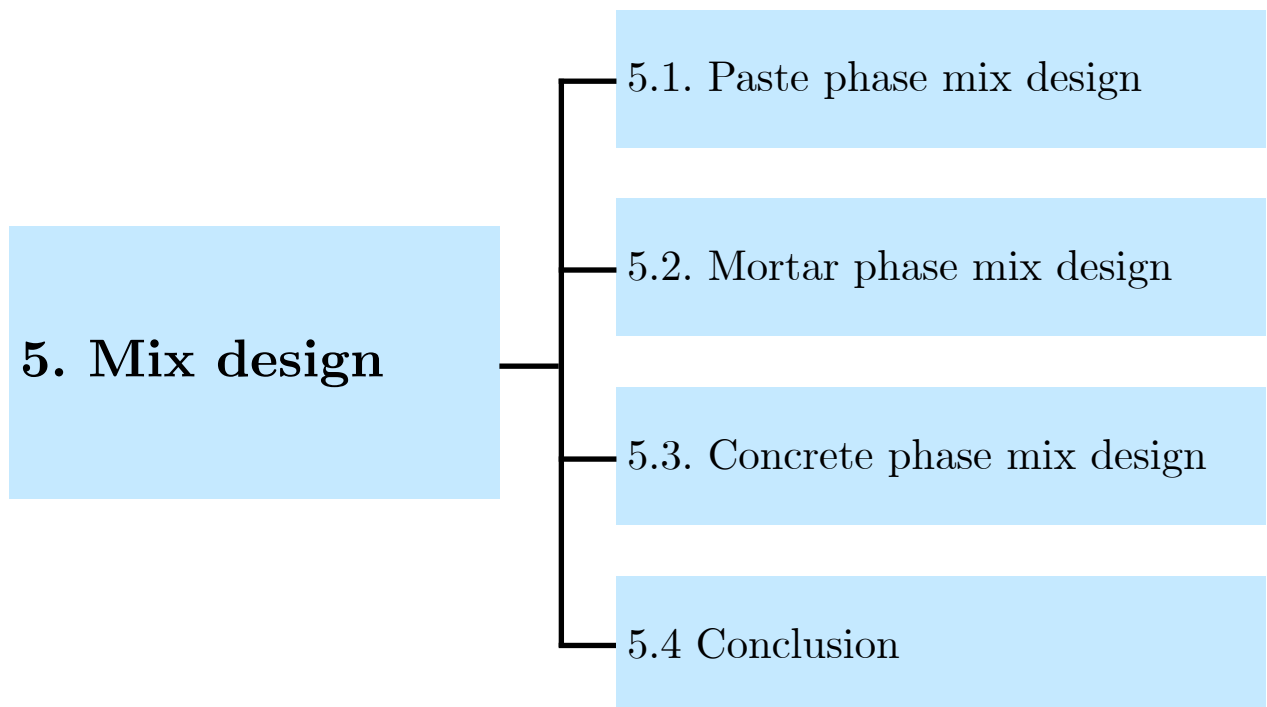
The aggregates that are used for the concrete phase have suitable particle size distribution for earth-moist application, but high water absorption.

The GGBFS has a composition similar to GGBFS found in literature and contains relatively fine particles, so reactivity is high. The CC has relatively low reactivity due to the low amorphous content and the more coarse particles, so expectation is that CC can only be applied to the precursor blend in limited amounts and acts mainly as a filler and not as a reaction contributing precursor.

With the combination of chapter 2, 3 and 4 the mix designs can be developed, which are in a later stage tested at paste, mortar and concrete scale. The mix designs can be found in the next chapter.

5.

Mix design



This chapter contains mix designs that are used in this research. Section 5.1 starts with the paste phase mix designs, followed by the mortar mix designs in section 5.2. Section 5.3 contains the concrete phase mix designs. The chapter is concluded in section 5.4.

5.1. Paste phase mix design

The first phase in this research is to create paste mix designs. With these pastes, the objective is to determine compressive strength, flexural strength, workability and to learn to work with the materials. By varying a list of parameters, each of which is stated below, the influence of these parameters on the behavior of the material can be determined.

Description of used mix design parameters

For formulation of mix designs, the following parameters are used in this research:

- The modulus or “Ms” value: $\frac{SiO_2}{Na_2O}$ molar ratio of the activators.
- Water-to-binder: $\frac{W}{B}$ ratio of total water divided by the sum of solid precursor and solid activator.
- Sodium oxide percentage by mass of precursor: Na_2O % .
- Precursor ratio: $\frac{GGBFS}{GGBFS+CC}$ ratio. If value is 1.0, this means that precursor consists of 100 % GGBFS. If this value is 0.3, this means that precursor consists of 30 % GGBFS and 70 % CC.
- Precursor/Activator: How much activator is used relative to the amount of precursor (used in spreadsheets to tweak the sodium oxide percentage).
- Precursor amount: How much precursor is used (fixed amount in this research).

Trial

The starting point for the paste mix designs is a trial, consisting of three different mixtures where only ground granulated blast furnace slag is used as precursor. The aim of the trial is to learn to work with the equipment used in the paste phase and to discover the behavior of the material. A full description of the paste trial can be found in appendix B.

Parameter variation

The design parameters are chosen such that there is quite a wide range for each parameter. This way there is a better possibility to find optimum values for each property the final concrete should contain. Besides finding optimum values, the wide range also allows for observing trends in the various properties that are to be tested.

As the final concrete should be dry-cast, the W/B ratios are chosen such that it allows for a decrease in workability due to inclusion of calcined clay, but at the same time not being too high as it would be too far from the final earth moist-concrete target. The W/B is also kept relatively low due to its negative contribution to strength development.

The amount of calcined clay in the precursor is varied from no calcined clay until partial replacement as the calcined clay used in this research is not very pure and does not have a significant amorphous content. However, its contribution to the development of mechanical and durability properties in the mix should be investigated thus the amount of calcined clay added should not be too little. Due to the combination of low amorphous content and analyzing the effects of calcined clay in the mix design, the range of 1.00 - 0.5 is chosen, such that the calcined clay content varies between 0 % and 50 % in the total precursor blend.

The sodium oxide percentage has a large influence on the environmental impact of the final material, as it determines the total amount of activator that is used in the mix design. As low environmental impact is one of the goals of this research, this parameter should not be too high.

However, too low sodium oxide content results in bad mechanical properties, which is undesired. Therefore, a range of 3 % to 5 % is chosen.

The modulus determines the relative amount of sodium silicate to sodium hydroxide in the total activator solution. Higher modulus equals higher amount of sodium silicate and lower amount of sodium hydroxide. Sodium plays an important role in geopolymer formation as it charge balances ions. With too much sodium, water will not evaporate resulting in a hindrance of structural formation (Alanazi et al., 2017). The modulus also allows for influencing the rate of strength development of the specimens. A lower modulus results in faster strength development, therefore the modulus contains the values 0.4, 0.7 and 1.0.

Based on the paste trials, the variation of mix design parameters as visualized in Table 5.1 is used in the paste phase.

Table 5.1 - Variation of mix design parameters for paste phase

Level	W/B	GGBFS/GGBFS+CC	Na ₂ O (%)	Ms
1	0.35	1.0	3	0.4
2	0.40	0.75	4	0.7
3	0.45	0.5	5	1.0

Paste phase mix composition

The total precursor content remains constant in this research. Combined with a Taguchi L9 array, the 9 mix designs in Table 5.2 below, based on the parameters of Table 5.1, are used. The exact composition of the pastes can be found in appendix C.

Table 5.2 - Mix designs for paste phase

Mix number	W/B	GGBFS/ GGBFS+CC	Na ₂ O (%)	Ms
P01	0.35	1.0	3	0.4
P02	0.35	0.75	4	0.7
P03	0.35	0.5	5	1.0
P04	0.40	1.0	4	1.0
P05	0.40	0.75	5	0.4
P06	0.40	0.5	3	0.7
P07	0.45	1.0	5	0.7
P08	0.45	0.75	3	1.0
P09	0.45	0.5	4	0.4

5.2. Mortar phase mix designs

When upscaling from paste to mortar, sand is added. The inclusion of this sand results in changes to the mix design. While the four used mix design parameters can remain equal (W/B ratio, precursor ratio, sodium oxide percentage and modulus), the total amount of binder and sand should be determined as this does change. The volume of binder is known. The sand content is determined equal to gravel content (for each kg gravel there is a kg of sand). By subtracting the volume of binder by a unit volume (1 m^3) the total amount of aggregates is determined. The volume of sand is $\frac{1}{2}$ the volume of the total aggregates. With the total volume of binder and sand known, both volumes are combined and volumetrically converted resulting in mix designs expressed in kg/m^3 .

The mortar phase uses the exact same Taguchi array as used in the paste phase. The Taguchi is kept equal for several reasons:

- Direct effects of introduction of sand into the mixture can be observed as the rest of the mix design remains equal
- The range of application possibilities for the material is still very wide, allowing to observe other possible applications then concrete revetments
- Narrowing down the range could result in missing the sweet spot for certain mix design parameters in terms of the freeze-thaw and sulfate attack performance, which are the experiments added to the experimental program in the mortar phase

Mortar phase mix composition

As the Taguchi array is kept equal, only the mix design composition is presented in Table 5.3 below. The exact composition of each individual mix design can be found in appendix D.

Table 5.3 - Mix designs for mortar phase

Mix number	W/B	GGBFS/ GGBFS+CC	Na ₂ O (%)	Ms
M01	0.35	1.0	3	0.4
M02	0.35	0.75	4	0.7
M03	0.35	0.5	5	1.0
M04	0.40	1.0	4	1.0
M05	0.40	0.75	5	0.4
M06	0.40	0.5	3	0.7
M07	0.45	1.0	5	0.7
M08	0.45	0.75	3	1.0
M09	0.45	0.5	4	0.4

5.3. Concrete phase mix designs

When upscaling from mortar to concrete, gravel is added. The desired minimum density of the mix designs should equal 2300 kg/m³ as prescribed by the norm. As the precursor content is known, the total volume of binder is determined. The total volume (1 m³) minus the binder volume results in the total volume that is available for aggregates. The industry provides the ratios in which the four aggregate groups that are used for the concrete phase should be applied. With the determined specific gravity, the amount of each aggregate group is determined. This results in the concrete phase mix designs.

The concrete phase mix designs are based on the results of both the paste and mortar phase. From these two phases, it is concluded that mix design 1 and 5 (P01/M01 and P05/M05) are most suitable for upscaling towards concrete. For details why these mix designs are chosen, refer to the conclusion of section 6.2. The main contents of the mix designs are stated in Table 5.4 below. The exact contents of the mix designs can be found in confidential appendix F.

Table 5.4 - Mix designs for concrete phase

Mix number	W/B	GGBFS/ GGBFS+CC	Na ₂ O (%)	Ms
C01	0.35	1.0	3	0.4
C05	0.40	0.25	4.5	0.4

5.4. Conclusion

Both the paste and mortar phase contain 9 mix designs using the same mix design parameters and variations. W/B ratio is chosen low as the final developed concrete should be earth-moist, so water content and workability should be on the low side. Calcined clay is included in the precursor blend but in an amount that is expected to show its contribution to mechanical properties but still has an upper limit of 50 % as it is expected not to contribute too much due to low amorphous content and large particle size. The sodium oxide percentage is kept as reasonable value to ensure the material develops good mechanical properties, but to prevent very fast setting time. It is also at reasonable values as it directly impacts the environmental footprint of the final concrete. The modulus is kept on the low side for good development of early age strength, necessary in the production process for dry-cast revetment products.

The concrete phase contains the two most promising mix designs but upscaled to concrete. With the mix designs known, the experiments described in chapter 4 can now be conducted and the results can be found in the next chapter.

Experimental results and analysis

6. Experimental results and analysis

6.1. Paste phase results

6.2. Mortar phase results

6.3. Concrete phase results

6.4. Conclusion

This chapter provides all experimental results of the research. Section 6.1 starts with elaborating the experimental results of the paste phase. Section 6.2 follows with results from the mortar phase. The concrete phase results are presented in section 6.3 and the chapter is concluded in section 6.4.

6.1. Paste phase

6.1.1. Workability

Results

The workability results of the 9 mix designs that are used during the paste phase are presented in Figure 6.1 below.

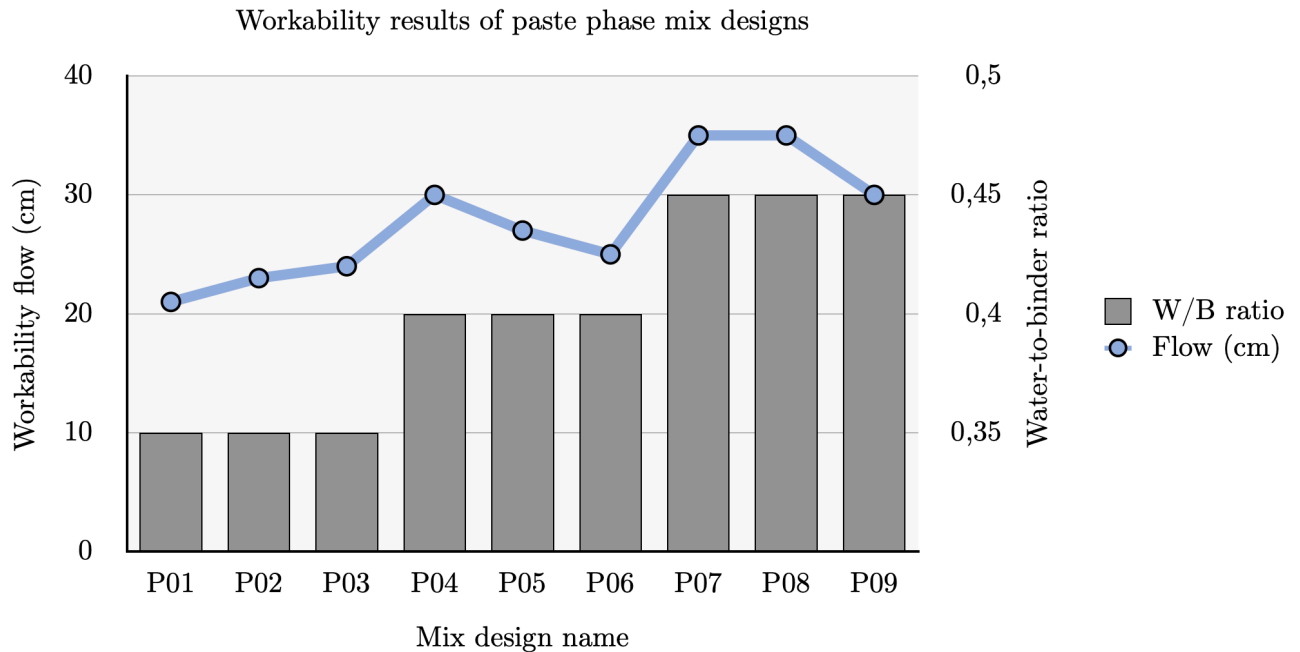


Figure 6.1 - Workability paste phase mix designs

Figure 6.1 shows the water-to-binder ratio with the use of grey bars. The corresponding flow has been indicated with the ● icons.

Interpretation of results

Workability in terms of measured flow is roughly as expected. As the water-to-binder ratio of the mixtures increases, so does the flow, which is in line with other researches. This statement is validated by comparing mixture P01, P04 and P07. All three consist of the highest percentage of slag (100 %) and workability increases with each increase of water-to-binder ratio. Another interesting observation is the influence of increasing the amount of calcined clay to the paste workability. Mixtures P01, P04 and P07 contain 100 % slag and 0 % calcined clay. Mixtures P02, P05 and P08 contain 75 % slag and 25 % calcined clay. Mixtures P03, P06 and P09 contain 50 % slag and 50 % calcined clay.

For all mixtures with water-to-binder ratio of 0.40 and 0.45, it is clear that including calcined clay decreases workability with the mixtures. However, the same trend is imperceptible for mixtures P01-P03 which all contain a 0.35 water-to-binder ratio. The results of P01-P03 are odd, since an increase of Na_2O content and increase of calcined clay should result in a decrease of flow (Huseien et al., 2018), (Hadi, 2017). A reason why it is expected to have a higher workability going from P01 to P03 is the increase in the modulus within these three mixes. The increase of modulus results in a lower pH value of the binder which increases the workability (Jiang et al., 2019), (Choi & Lee, 2019).

Conclusion

Most workability results, except for mix P01-P03, are in line with expectations. The deviation from expected results in mix P01-P03 is partially attributed to the difference in modulus in these mix designs. However, it is not yet possible to provide clear indication for the results of this workability

test to the final required workability which is in line with earth moist concrete as the testing methodology for earth moist concrete has a significantly different testing setup. The results from the mortar phase will already be more interesting, as inclusion of sand to the paste already increases the simulation of actual concrete. However, the workability trends of both the paste- and mortar phase are used to adapt the final concrete mixtures to have the desired workability.

6.1.2. Flexural strength

Results

The results of flexural strength of the 9 paste mixtures after 1, 7 and 28 days of curing is provided using three figures in total, of which the contents of each figure is shortly elaborated below before presenting them:

Figure 6.2: Shows the flexural strength results after 1, 7 and 28 days of curing of each mix design.

Figure 6.3: Shows the trend that each mix design parameter (W/B ratio, precursor ratio, sodium oxide % and modulus) has on the development of flexural strength using the signal-to-noise values that are determined with the Taguchi method. Here, the higher the signal-to-noise ratio of a mix design parameter, the better a mixture with the corresponding value should develop higher flexural strength (remember, the Taguchi method signal-to-noise ratio equation is in this case based on maximizing the flexural strength performance).

Figure 6.4: Shows the influence each mix design parameter has on the development of flexural strength for the 9 geopolymer paste mixtures, coupled to curing age. This influence is determined with the S/N ratio's from Taguchi. For each mix design parameter, the delta is determined (maximum S/N minus minimum S/N for the mix design parameter concerned). The ratio between the delta of the mix design parameter concerned and the summed delta of all mix design parameters yields the ratio that determines the influence of each mix design parameter on performance. Here, a higher influence means that development of flexural strength is more sensitive to the mix design parameter corresponding with the higher influence. The influence of the 4 mix design parameters, per curing age, combined add up to 100 %.

The figures can be found on the next page.

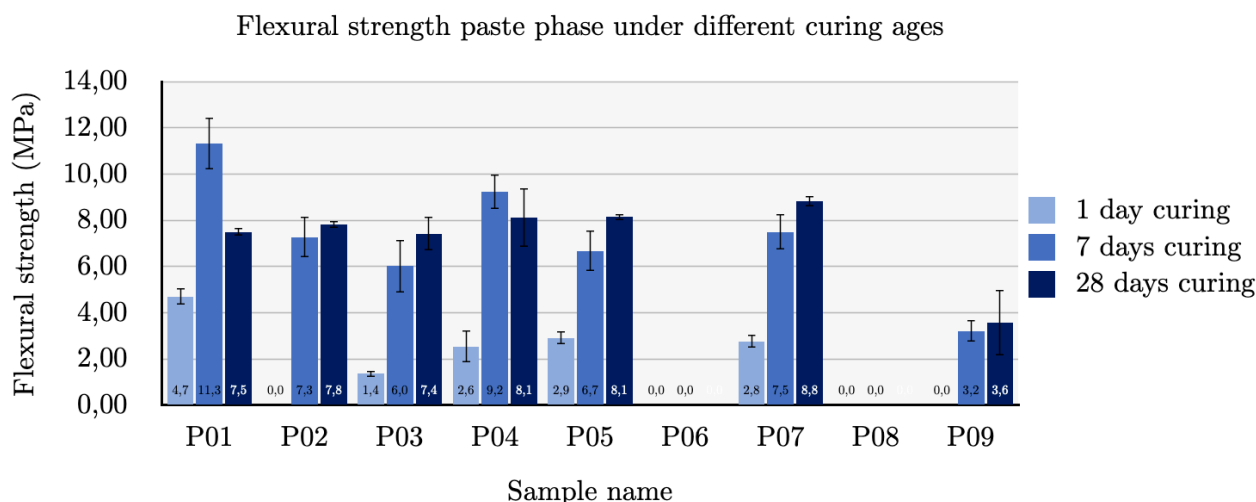


Figure 6.2 - Flexural strength development of paste specimens under different curing ages

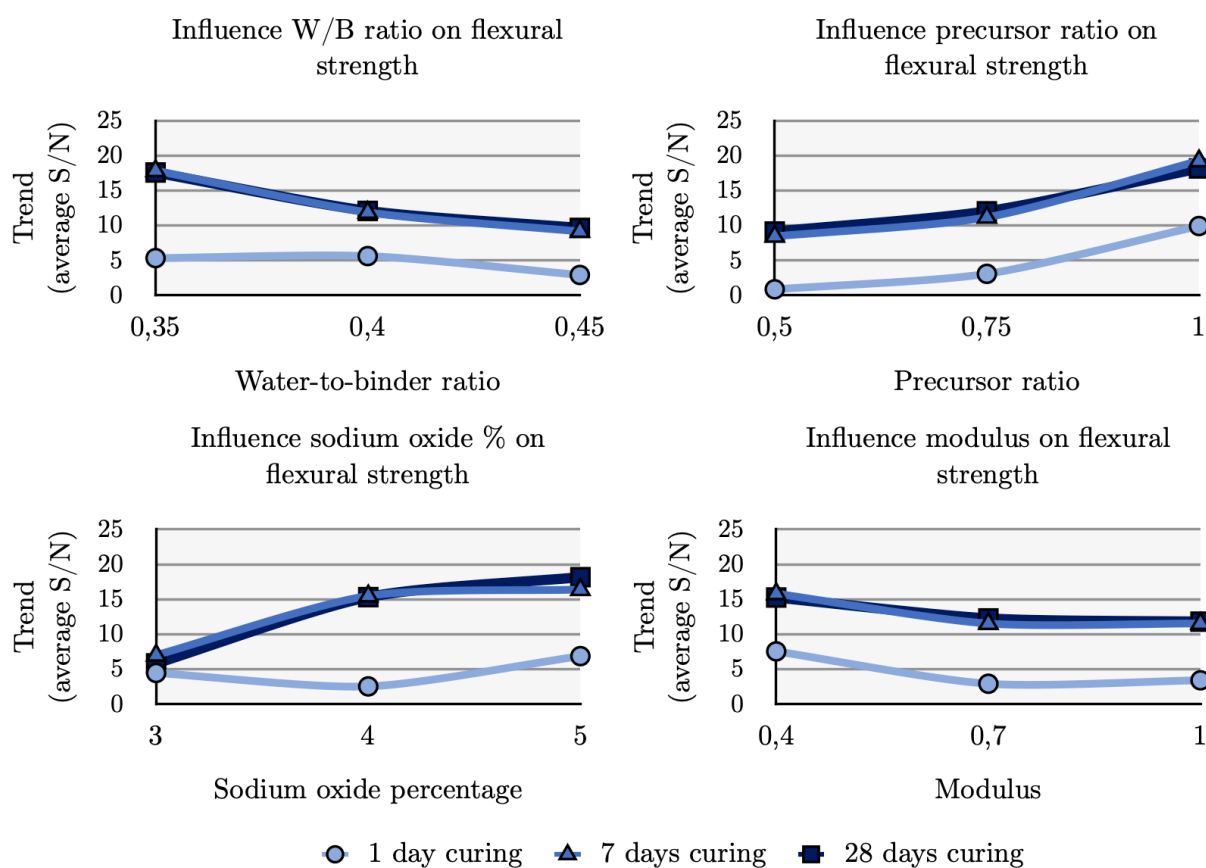


Figure 6.3 - Taguchi trends for flexural strength development at different curing ages

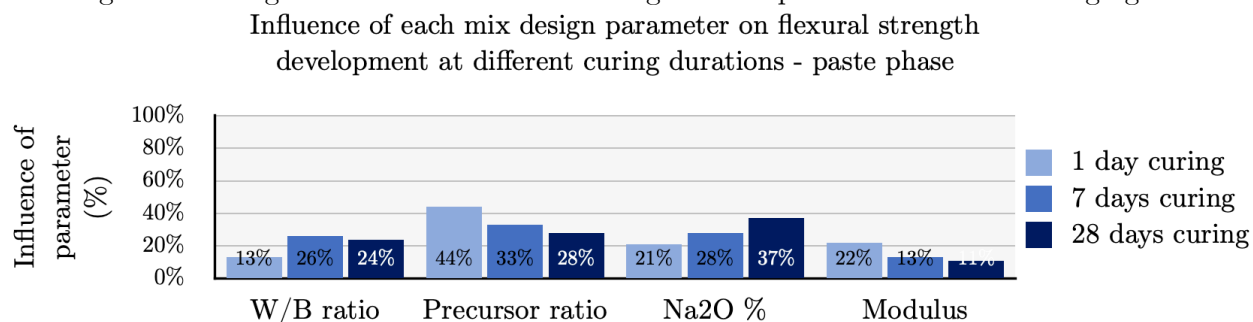


Figure 6.4 - Influence of each mix design parameter on flexural strength development

Interpretation of results

Flexural strength development of paste specimens under different curing ages - Figure 6.2

The flexural strength development at 1 day curing age tends to be rather low for all mix designs. Looking at 28 day curing flexural strength, 6 out of 9 mixtures ended up around the 8 MPa mark. Especially notable in these results is the decrease in flexural strength going from 7 to 28 days curing for mixtures P01 and P04. This odd result is due to a leaching effect in the curing room, where the very high humidity causes pore alkalis to leach out of the paste specimens through capillary action, resulting in less alkalis available for development of mechanical properties (M. Dong et al., 2020). This effect is confirmed by casting P01 and P04 once more, but in sealed conditions. Strength degradation is not noticed in that case. However, the leaching effect does result in less reliable result comparison as the amount of leaching may vary between mix designs.

Also noticeable are mixtures P06 and P08 which do not develop any flexural strength at all. The reason why P06 and P08 do not develop any flexural strength at all is explained with a combination of the trends (Figure 6.3) and the alkalinity of the activators used for these mixtures. The pH of the activator in P06 and P08 is the lowest (13.8) out of all mix designs (ranging from 13.8 to 14.6), due to the combination of the total sodium oxide percentage by mass and modulus used in these mixtures. Due to this lower alkalinity, the environment for these mixtures can be unsuitable for development of mechanical properties.

Taguchi trends for flexural strength development at different curing ages - Figure 6.3

The Taguchi trends show the preferred mix design parameter for flexural strength development at different curing ages by using the signal-to-noise ratio as described by the Taguchi method. The higher the signal-to-noise ratio, shown on the vertical axis, the more preferred the mix design parameter value is.

What is very interesting to see is that for the most part, the trend between all curing ages follows an equal curve, with only a shift in height. An upward shift means that the result should be higher flexural strength. However, for the 1 day flexural strength development, the trend is different for both the water-to-binder ratio and the sodium oxide percentage. This is mainly due to the fact that at 1 day flexural strength development, some mixtures (P02 with 0.35 W/B and 4 % sodium oxide, P03 with 0.35 W/B and 5 % sodium oxide and P09 with 0.45 W/B and 4 % sodium oxide) have no development of flexural strength at all, thus changing the overall trend.

The following development in trends can be observed:

- W/B ratio: For both early and late age flexural strength development, a W/B of 0.35 or 0.40 is preferred. The highest W/B of 0.45 yields in (very) low flexural strength development.
- Precursor ratio: For both early and late age flexural strength development, a value of 1.0 or 0.75 is preferred. This means that the use of 100 % GGBFS and 0 % CC or a combination of 75 % GGBFS and 25 % CC is preferred for good flexural strength development. Using more than 25 % CC results in very low flexural strength development.
- Sodium oxide percentage: For early flexural strength development, a high sodium oxide percentage is preferred. This is still true for late age flexural strength development, but the difference between the 4 % and 5 % sodium oxide is very small, thus an optimum might be present between these values.
- Modulus: A modulus of 0.4 is preferred for both early and late age flexural strength development. This translates to a higher content of sodium hydroxide present in the activator. This is logical, as sodium hydroxide has a fast reaction process due to its high initial heat evolution, resulting in fast early age mechanical properties.

Influence of each mix design parameter on flexural strength development - Figure 6.4

The higher the influence percentage is, the more sensitive the development of flexural strength is to the corresponding mix design parameter. The graph clearly shows that the precursor ratio has the highest influence on the development of flexural strength at early age, with a value of 44 %. However, at later age the most influence slowly shifts towards the sodium oxide percentage. At 28 days curing, the sodium oxide has the most influence on flexural strength development with 38 %.

Conclusion

Development of flexural strength at early age is tricky and no outstanding values after 28 days of curing are found, where most mix designs top out with a value around 8 MPa. However, some clear trends are observed, which are shortly concluded below:

- Low W/B ratio is preferred, where a 0.35 value results in highest flexural strength.
- Using GGBFS as precursor is preferred as introduction of CC yields much lower flexural strength at both early and late age. The precursor ratio is most sensitive for early age flexural strength development.
- Higher sodium oxide (thus more activator) percentage is preferred, where an optimum value is in the range of 4 % and 5 %. This parameter is the most sensitive for late age flexural strength development.
- A lower modulus (meaning more sodium hydroxide and less sodium silicate) results in higher flexural strength both at early and late age.
- A high alkaline environment is required for good development of mechanical properties. When increasing the modulus or increasing the amount of CC in a mixture, the alkaline conditions of the activator should be determined and adjusted towards a more alkaline environment if needed.
- Curing of specimens which are fully exposed in higher than 95 % humidity can result in leaching of silica out of the geopolymer matrix resulting in a decrease of mechanical properties performance, as water is the transporting agent of both the precursor and activator. Therefore, lower than 95 % humidity is the preferred curing methodology.

6.1.3. Compressive strength

The results of compressive strength of the 9 paste mixtures after 1, 7 and 28 days of curing are provided using three figures in total, of which the contents of each figure is shortly elaborated below before presenting them:

Figure 6.5: Shows the compressive strength results after 1, 7 and 28 days of curing of each mix design.

Figure 6.6: Shows the trend that each mix design parameter (W/B ratio, precursor ratio, sodium oxide % and modulus) has on the development of compressive strength using the signal-to-noise values that are determined with the Taguchi method.

Figure 6.7: Shows the influence each mix design parameter has on the development of compressive strength for the 9 geopolymer paste mixtures, coupled to the curing age. The influence of the 4 mix design parameters combined add up to 100 %.

The figures can be found on the next page.

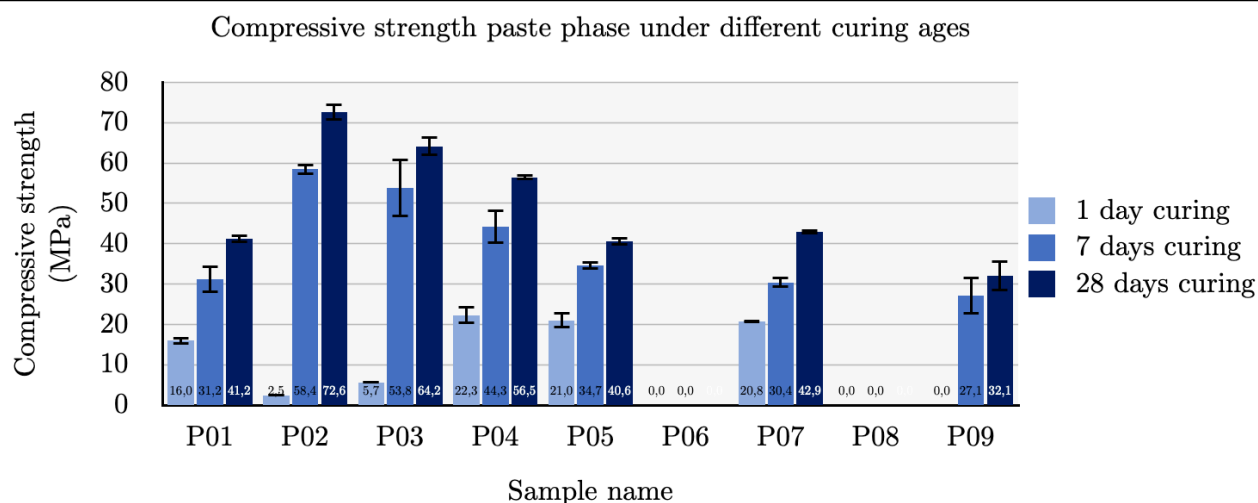


Figure 6.5 - Compressive strength development of paste specimens under different curing ages

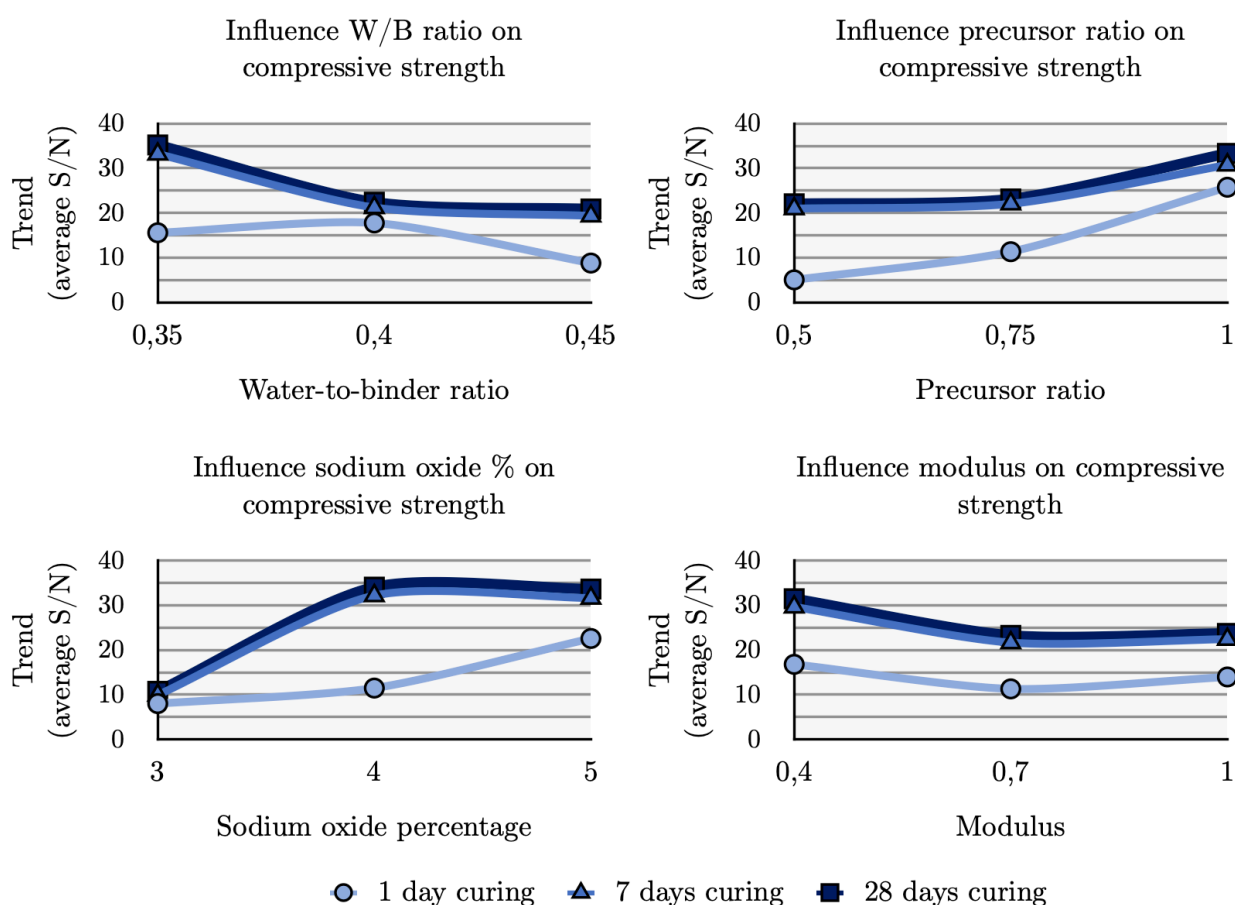


Figure 6.6 - Taguchi trends for compressive strength development at different curing ages

Influence of each mix design parameter on compressive strength development at different curing durations - paste phase

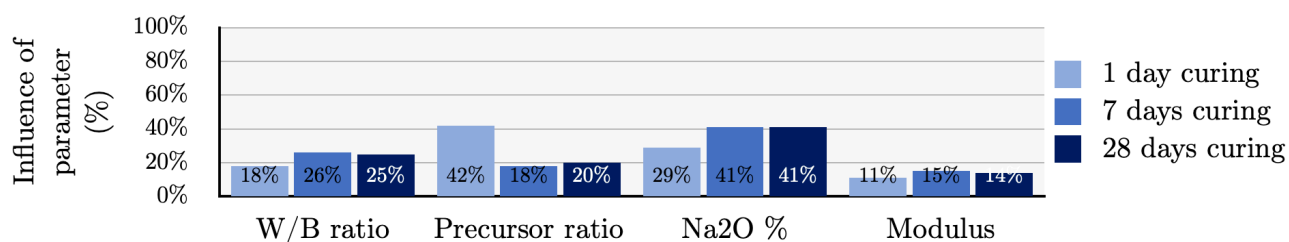


Figure 6.7 - Influence of each mix design parameter on compressive strength development

Interpretation of results

Compressive strength development of paste specimens under different curing ages - Figure 6.5

4 of the paste mix designs (P01, P04, P05 and P07) meet the 1 day compressive strength requirement (≥ 12 MPa) and 6 mix designs (P01 - P05 & P07) meet the 28 day compressive strength requirement (≥ 37 MPa). Still, other mix designs come close to the requirements where mild modifications of these mix designs should be sufficient to also meet the requirements. The compressive strength development is logical as compressive strength increases with increase of curing age. However, the rate of development is different due to the different modulus used in the mix designs. The strongest mix, P02, reaches 72.55 MPa after 28 days of curing. Like flexural strength, P06 and P08 do not develop any compressive strength.

It is interesting to observe that leaching clearly has little to no effect on the development of compressive strength. A possible cause for this can be attributed not to leaching, but to shrinkage of the specimens. Shrinkage of alkaline activated slag is reported often and can cause micro cracks in the internal structure of the paste (Collins & Sanjayan, 1998), (Puertas et al., 2003). These cracks especially have large influence on flexural strength due to tensile stresses induced by this test, but little influence on compressive strength as it induces only compressive stresses in the specimens.

Taguchi trends for compressive strength development at different curing ages - Figure 6.6

Once again, a slight shift in the trend can be seen for early age strength development for both the water-to-binder ratio and sodium oxide percentage.

The following developments in the trends can be observed:

- W/B ratio: For both early and late age compressive strength development, a lower W/B yields in higher compressive strength. For early age specifically, a 0.35 and 0.40 value both are fine, but for late age strength development a 0.35 value is clearly strongly preferred due to the much higher signal-to-noise ratio.
- Precursor ratio: Once again the incorporation of CC in the precursor blend has negative effect on both early age and late age compressive strength development, as expected due to the relatively large particles and low amorphous content. However, it can clearly be seen that the inclusion of CC is especially detrimental for early age compressive strength development. Using more than 25 % CC most likely results in not meeting the 1 day compressive strength requirement if activator amount is kept low. This is further substantiated when also observing the results of Figure 6.5, mix designs P03, P06 and P09. These mixtures all contain 50 % calcined clay. Only P03 shows some early age strength development, but that mixture also contains high activator content.
- Sodium oxide percentage: For early age compressive strength development it is very clear that a higher sodium oxide percentage yields higher results. However, it is very interesting to once again observe that for this set of mix design parameters, an optimum for late age compressive strength development seems to be found between 4 % and 5 % sodium oxide.
- Modulus: Once again an identical trend when compared to development of flexural strength is observed: A lower modulus (higher sodium hydroxide and lower sodium silicate) results in higher compressive strength development, both at early and late age.

Influence of each mix design parameter on compressive strength development - Figure 6.7

Once again, the precursor ratio is very important and the most sensitive mix design parameter for early age strength development, with a value of 42 %. Sodium oxide is also a sensitive mix design parameter, both for early- and late age strength development. For early age, it has 29 % influence, but at late age this influence increases to 41 %. Both the W/B ratio and modulus are less sensitive once again.

Conclusion

Compressive strength development for the paste phase is satisfactory, as many of mixtures show good potential for the application considered in this research. Some clear trends are visible and concluded below:

- Low W/B ratio is preferred, resulting in the highest compressive strength
- Using GGBFS as precursor is preferred as introduction of CC results in large impact of the early age compressive strength development. However, the CC does show potential for late age compressive strength development, with P02 (medium CC) and P03 (high CC) containing the highest compressive strength results at late age.
- Lower modulus is preferred for both early and late age compressive strength development.
- Curing conditions and the alkaline environment are still very important as they can hinder development of mechanical properties and prevent reactions from happening at all, as proven by P06 and P08.

6.1.4. Paste phase conclusion

2 out of 9 mix designs fail to develop mechanical properties in the paste phase and from the remaining mix designs there are already some promising results with the requirements in mind. Due to the good results, the same mix designs are used for the mortar phase, but sand is of course added as the additional ingredient.

Using identical mix designs in the mortar phase also allows for both a wider range of possible applications of the concrete that is in development and the possibility to directly compare the correlations between the paste- and mortar phase. Besides that, narrowing down the ranges of the mix design parameters at this stage can result in missing the sweet spot for freeze-thaw resistance and sulfate attack resistance, the two additional performance parameters that are investigated in the mortar phase.

Combining the trends from the paste- and mortar phase allows for development of concrete mix designs based on these trends. These mix designs should result in checkboxes for all requirements, to prove the effectiveness of the Taguchi method in an experimental study.

6.2. Mortar phase

6.2.1. Workability

The workability of the 9 mix designs that are used during the mortar phase is presented in Figure 6.8 below.

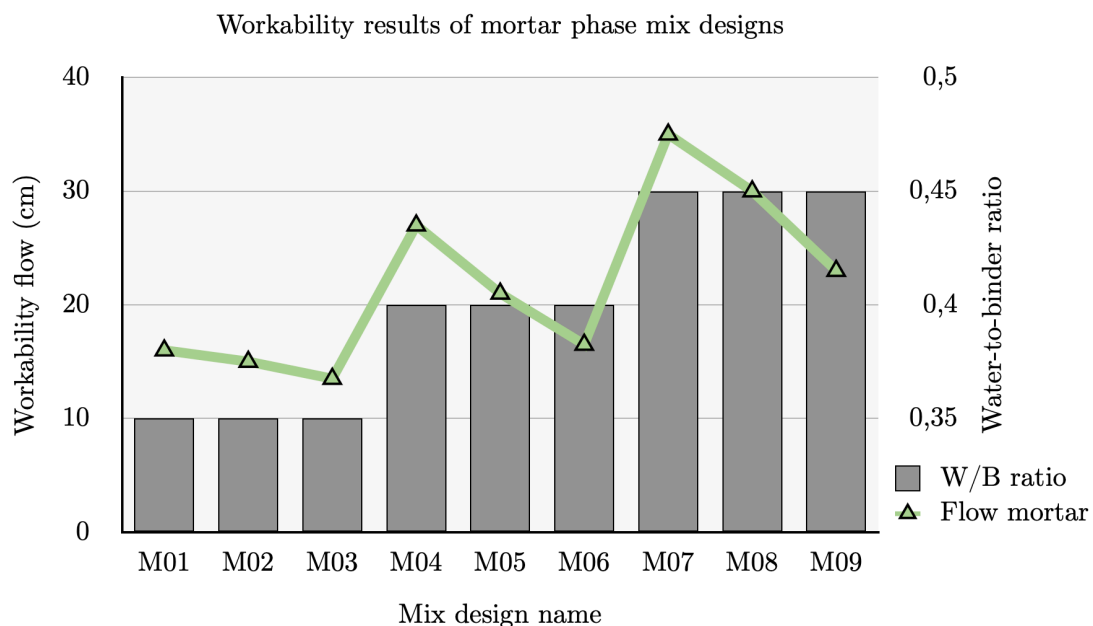


Figure 6.8 Workability mortar phase mix designs

Figure 6.8 shows the water-to-binder ratio with the use of grey bars. The corresponding flow is indicated with the Δ icons.

Interpretation of results

Workability is once again as expected. It is very clear from the graph that increasing the water-to-binder ratio yields an increase in the workability, just as observed in the paste phase mix designs. However, it is also clear that replacement of ground granulated blast furnace slag by calcined clay decreases workability. This can now be seen throughout all 9 mix designs.

When comparing workability to the paste phase, see Figure 6.9 below, the general trend is that overall workability is lower which can be explained due to the relative lower amount of fluids in the total mixture and the sand which also absorbs a little bit of water.

Workability results of mortar phase mix designs

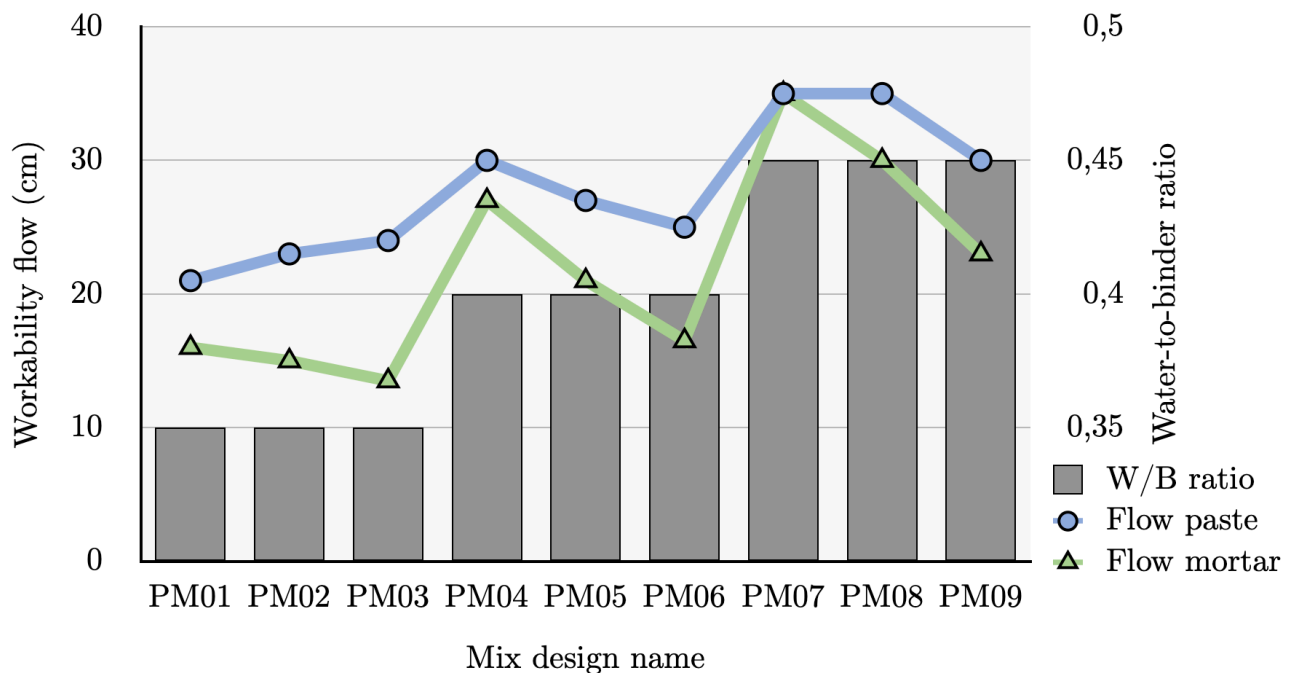


Figure 6.9 - Comparison of workability of paste- and mortar phase mix designs

As can be seen in Figure 6.9, the introduction of both the sand but also the amount of calcined clay in the precursor causes larger decreases in workability. The decrease of workability due to introduction of sand can clearly be seen by generally lower values, while the influence of the calcined clay is visible in the decreasing slope of the line within each water-to-binder value (PM01-PM03, PM04-PM06 & PM07-PM09).

Conclusion

Workability is as expected for the mortar phase mix designs. Introduction of sand in the mixture, causing some of the water to be absorbed in the sand, and a relative lower amount of total water compared to total amount of dry materials resulted in overall lower workability when compared to the paste phase. Introduction of calcined clay also clearly results in strong reduction of the workability, as can be seen by the downward trend. However, the influence calcined clay on workability is less when the water-to-binder ratio decreases (difference of 2.5 cm for M01-M03, difference of 10.5 cm for M04-M06 and difference of 12 cm for M07-M09).

This workability test is in not comparable to the workability test which is coupled to earth moist concrete due to the very different testing methodology. However, it still provides valuable information if other application possibilities are considered besides earth moist concrete. During the

concrete phase, trials are performed on workability allowing the mix designs to be adjusted towards the desired workability.

6.2.2. Flexural strength

Results

The results of flexural strength of the 9 mortar mixtures after 1, 7 and 28 days of curing is provided using three figures in total, of which the contents is shortly elaborated below.

Figure 6.10: Shows the flexural strength results after 1, 7 and 28 days of curing of each mix design.

Figure 6.11: Shows the trend that each mix design parameter (W/B ratio, precursor ratio, sodium oxide % and modulus) has on the development of flexural strength using the signal-to-noise values that are determined with the Taguchi method. Here, the higher the signal-to-noise ratio of a mix design parameter, the better a mixture with the corresponding value should develop higher flexural strength (remember, the Taguchi method signal-to-noise ratio equation is in this case based on maximizing the flexural strength performance).

Figure 6.12: Shows the influence each mix design parameter has on the development of flexural strength for the 9 geopolymer paste mixtures. Here, a higher influence means that development of flexural strength is more sensitive to the mix design parameter corresponding with the higher influence. The influence of the 4 mix design parameters, coupled to the curing age, combined add up to 100 %.

The figures are presented on the next page.

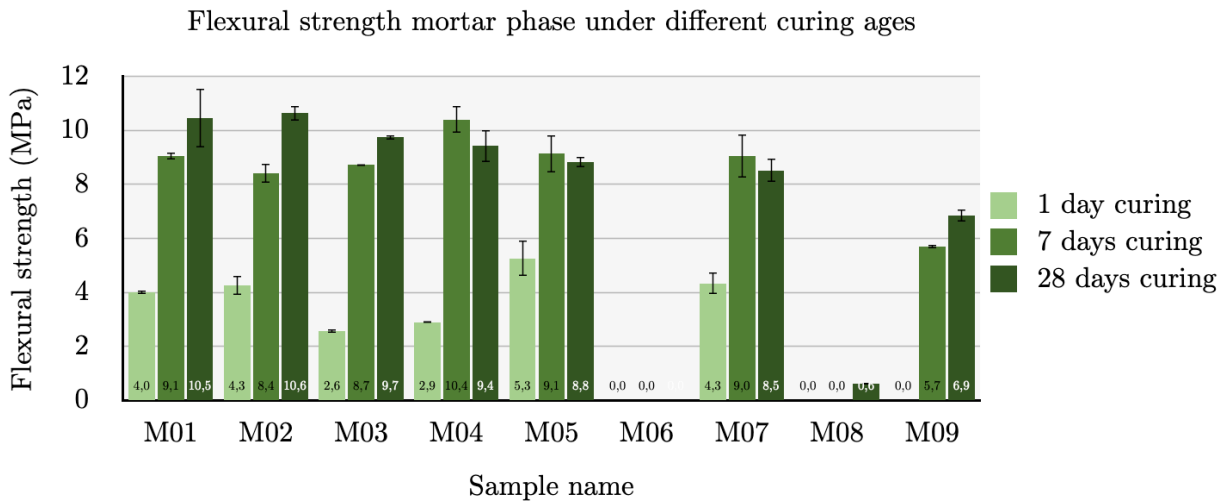


Figure 6.10 - Flexural strength development of mortar specimens under different curing ages

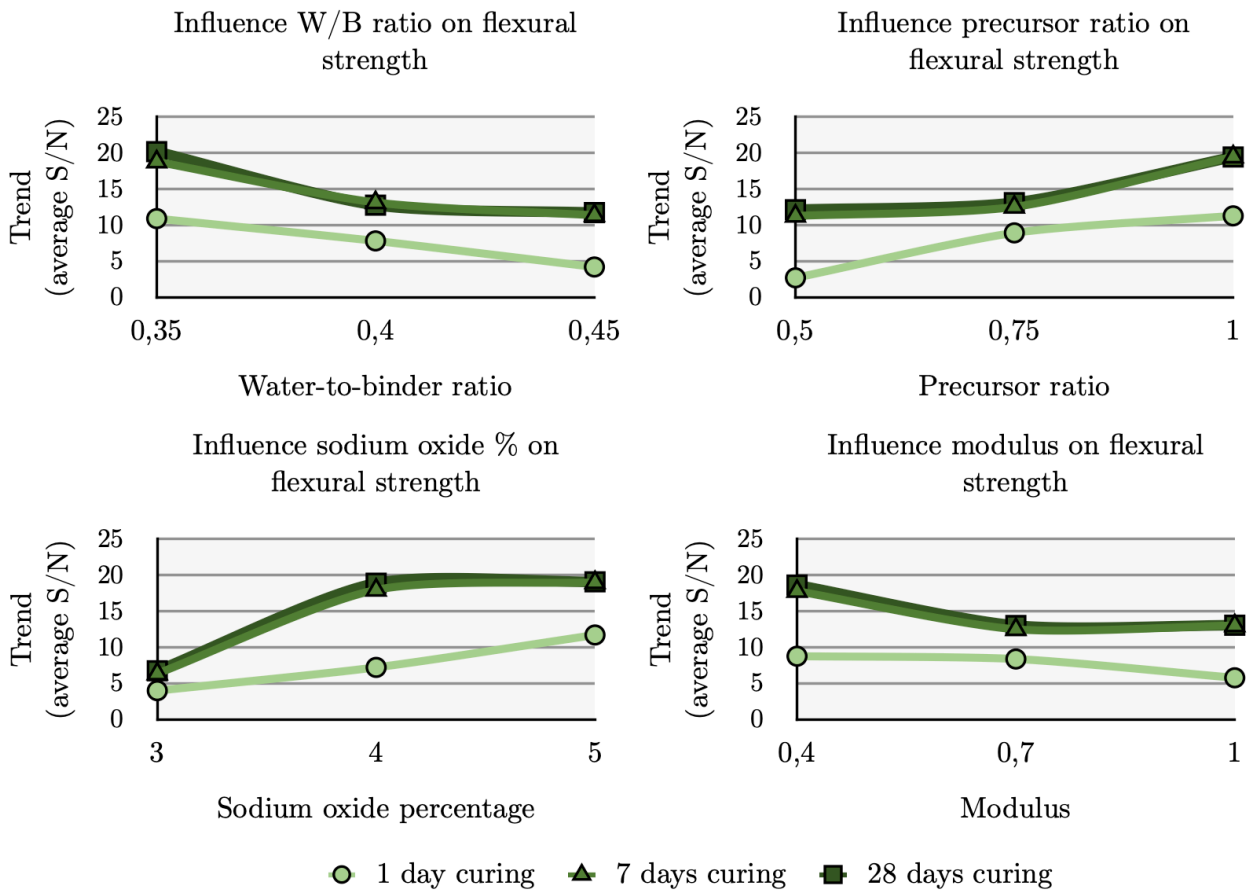


Figure 6.11 - Taguchi trends for flexural strength development at different curing ages

Influence of each mix design parameter on flexural strength development at different curing durations - mortar phase

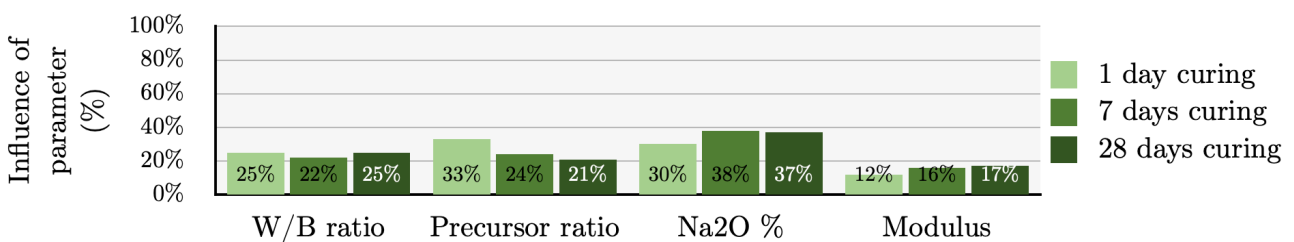


Figure 6.12 - Influence of each mix design parameter on flexural strength development

Interpretation of results

Flexural strength development of mortar specimens under different curing ages - Figure 6.10

The flexural strength development after 1 day curing is decent for most mix designs. After 28 days of curing, 6 out of 9 mixtures end up around the 9 MPa mark, which is 1 MPa higher than the results in the paste phase. Just like the paste phase, some mixtures have a decrease in flexural strength going from 7 to 28 days of curing, possibly due to the same leaching effect as concluded in the paste phase. In general the flexural strength is higher for mortars than for pastes, most likely due to the additional water absorption of the sand.

Once again, mixtures 6 and 8 are (nearly) not able to develop mechanical properties with the exception being mixture 8 at 28 days, but with a very low 0.6 MPa. As the rest of the mix design parameters are kept equal to the paste phase, once again these mixtures have non-suitable alkaline environment for development of mechanical properties.

Taguchi trends for flexural strength development at different curing ages - Figure 6.11

The trends show nearly equal results when compared to the paste phase. The only differences in comparison to the paste phase are that for early flexural strength development:

- The medium precursor ratio (0.75) has a less negative effect on development of flexural strength.
- A low W/B ratio now has more positive effect on early flexural strength development compared to the paste.
- The sodium oxide percentage now has more preference for 4 % over 3 % for early flexural strength development, where the paste phase has opposite results due to more failed early flexural strength mixtures.
- A medium modulus (0.7) is not as detrimental to early flexural strength development when compared to the paste phase. This effect is related to mixture M02 containing 1 day flexural strength result, whereas P02 in the paste phase does not have any result for flexural strength after 1 day. This favors the 0.7 value for the modulus.

For late age flexural strength development, the trends are equal to the paste phase.

Influence of each mix design parameter on flexural strength development - Figure 6.12

For early age flexural strength development once again the precursor ratio has the most influence with a value of 33 %. For late age flexural strength development the sodium oxide percentage has most influence, with a value of 37 %. However, sodium oxide percentage also has a rather high influence on early age flexural strength development and should be well considered when designing the final concrete mixtures.

Conclusion

Development of flexural strength at early age is better than the paste phase, with 6 out of 9 mixtures developing flexural strength. Once again, mixtures M06 and M08 fail to develop any flexural strength. After 28 days of curing, most mixtures reach 9 MPa of flexural strength. Even though this result is better than the paste phase, it still is not an outstanding value, but sufficient for the application this research focuses on.

The trends can be concluded as follows:

- Low W/B is preferred as this is the value resulting in highest flexural strength.
- Using GGBFS is preferred as precursor with introduction of CC slightly reducing early age flexural strength development. The precursor ratio is the most sensitive parameter for early age flexural strength development.
- A high sodium oxide percentage is clearly preferred for early age flexural strength development. However, for late age flexural strength development there once again seems to be an optimum between the 4 % and 5 % sodium oxide. This parameter is most sensitive for late age flexural strength development once again.
- A lower modulus results in higher flexural strength at both early and late age flexural strength.

- During the mortar phase, overall flexural strength increased a little compared to the paste phase. Differences in trends can mainly be explained due to 1 day flexural strength development of M02, where P02 had a 0 value there.

6.2.3. Compressive strength

The results of compressive strength of the 9 mortar mixtures after 1, 7 and 28 days of curing are provided using three figures in total, of which the contents of each figure is shortly elaborated below before presenting them:

Figure 6.13: Shows the compressive strength results after 1, 7 and 28 days of curing of each mix design.

Figure 6.14: Shows the trend that each mix design parameter (W/B ratio, precursor ratio, sodium oxide % and modulus) has on the development of compressive strength using the signal-to-noise values that are determined with the Taguchi method.

Figure 6.15: Shows the influence each mix design parameter has on the development of compressive strength for the 9 geopolymer mortar mixtures. The influence of the 4 mix design parameters, coupled to the curing age, combined add up to 100%.

The figures are presented on the next page.

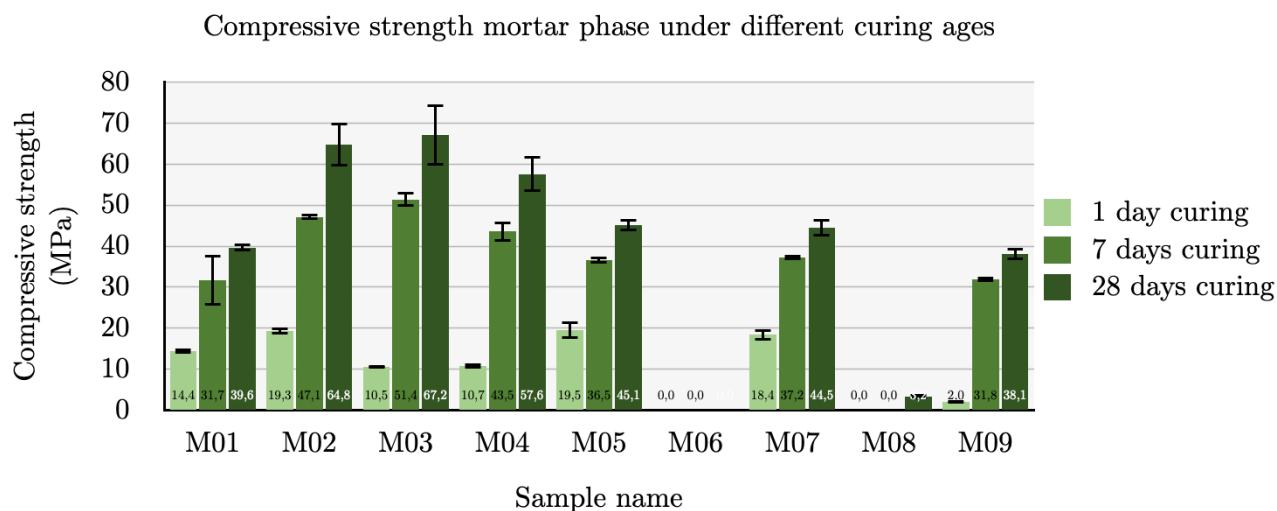


Figure 6.13 - Compressive strength development of mortar specimens under different curing ages

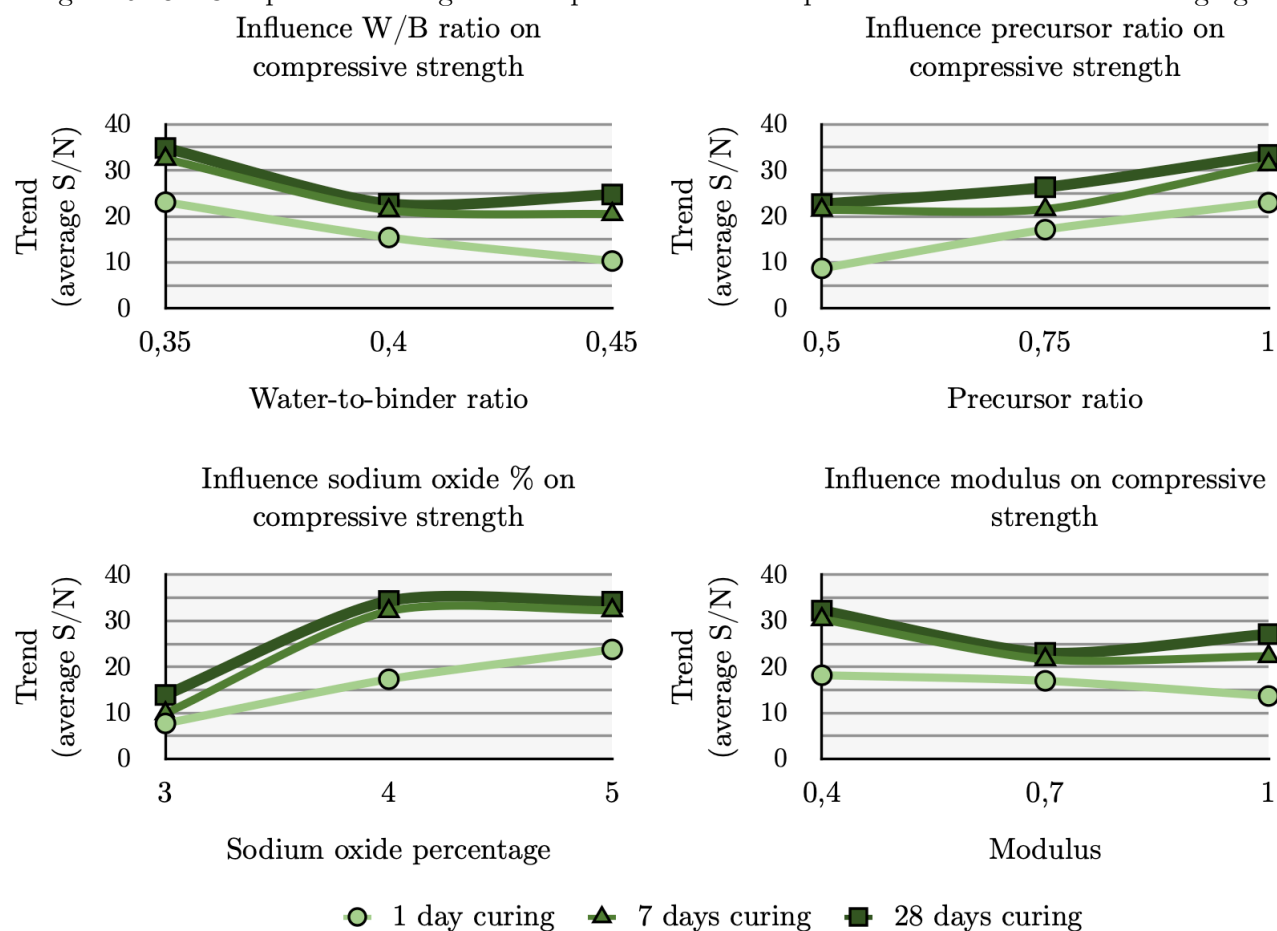


Figure 6.14 - Taguchi trends for compressive strength development at different curing ages

Influence of each mix design parameter on compressive strength development at different curing durations - mortar phase

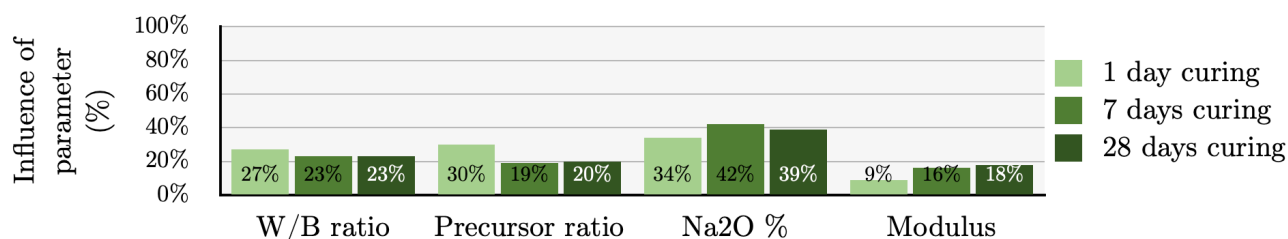


Figure 6.15 - Influence of each mix design parameter on compressive strength development

Interpretation of results

Compressive strength development of mortar specimens under different curing ages - Figure 6.13

4 of the mortar mix designs (M01, M02, M05 and M07) meet the 1 day compressive strength requirement (≥ 12 MPa) and 7 mix designs (M01-M05, M07 & M09) meet the 28 day compressive strength requirement (≥ 37 MPa). Compressive strength development is once again logical, with continuous increase of compressive strength with longer curing. The strongest mix is M03, with an average value of 67,17 MPa after 28 days of curing.

Taguchi trends for compressive strength development at different curing ages - Figure 6.14

The trends show many similarities with the paste phase, with the only differences once again in the 1 day curing situation:

- The medium precursor ratio (0.75) has a less negative effect on development of compressive strength.
- A low W/B ratio now has more positive effect on early compressive strength development compared to the paste.
- The sodium oxide percentage now has more preference for the 4 % over the 3 % value for early compressive strength development.
- A medium modulus (0.7) not as detrimental to early compressive strength development when compared to the paste phase. This effect can be related to mixture M02 containing much higher 1 day compressive strength result compared to P02 in the paste phase. This favors the 0.7 value for the modulus.

Influence of each mix design parameter on compressive strength development - Figure 6.15

This time, the sodium oxide percentage has the most influence on development of both early age and late age compressive strength with values of 34 %, 42 % and 39 % after 1 day, 7 days and 28 days of curing respectively. During the paste phase, the precursor ratio has more influence on early age compressive strength development, whereas the sodium oxide percentage has most influence in the mortar phase. However, this shift is not extremely significant, as the precursor ratio still has 30 % influence for 1 day compressive strength development.

6.2.4. Water absorption

Results

Surface water absorption is tested by casting prisms of size 40 x 40 x 160 mm where both the casting surface and bottom surface are in a 5 mm layer of water. Water absorption is tested by casting cubes of 100 x 100 x 100 mm which are subjected to a combination of complete submersion (until mass difference in a period of 24h is < 0.5 %), submersion in boiling water (for 5 hours, then cooling down for 14 hours) and drying (until mass difference in a period of 24h is < 0.5 %). The results for water absorption are provided in Table 6.1 below. The results for surface water absorption as function of time are presented in Figures 6.16 and 6.17 on the next page.

Table 6.1 - Water absorption of mortar cubes

Mix design	Water absorption (A)	Volume of permeable pore space (B)
M01	5,90%	11,13%
M02	6,62%	12,62%
M03	7,45%	14,08%
M04	7,91%	13,94%
M05	8,84%	14,25%
M07	9,07%	15,95%
M09	9,20%	16,87%

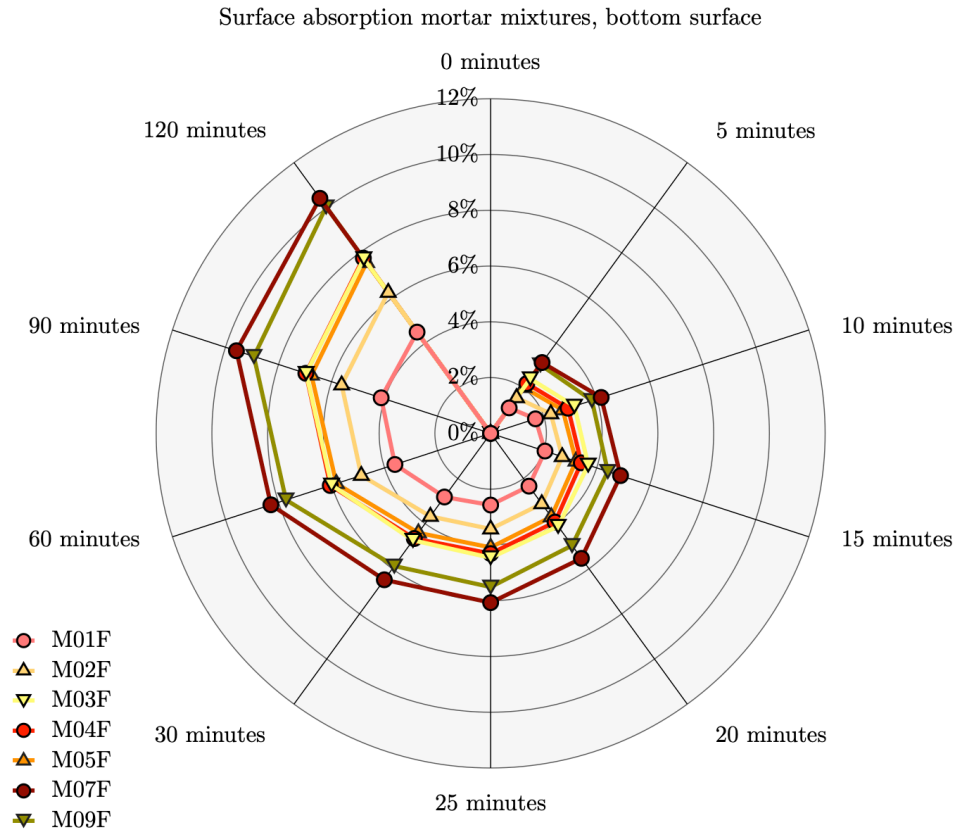


Figure 6.16 - Surface absorption mortar mixtures, bottom surface

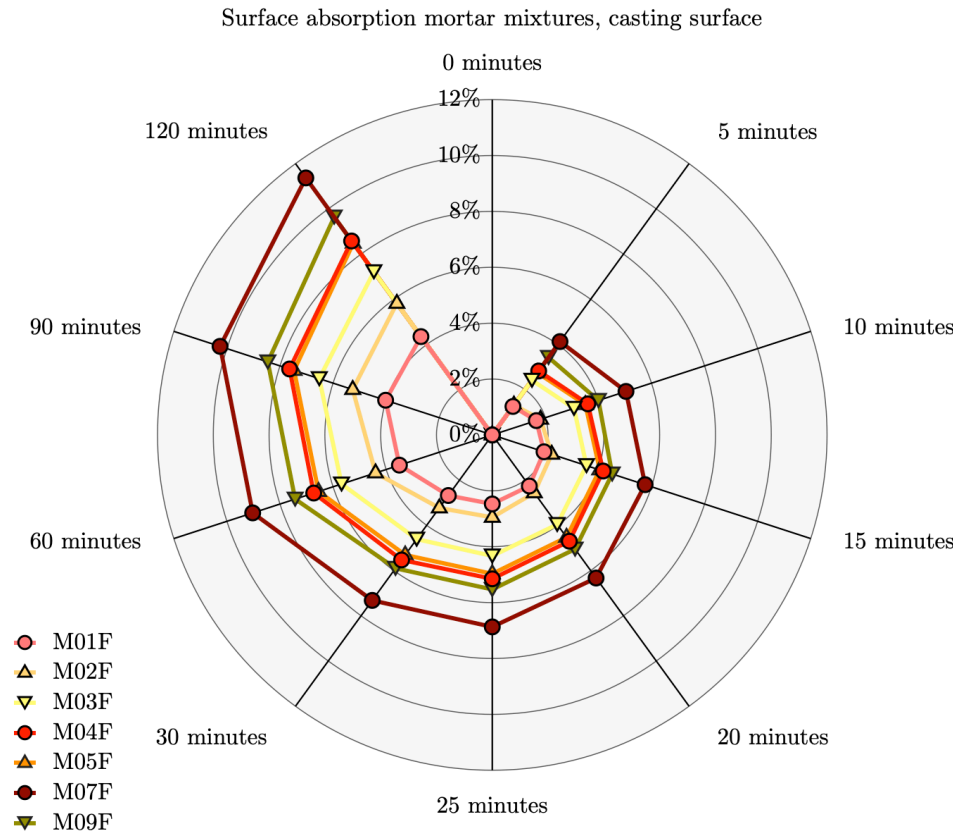


Figure 6.17 - Surface absorption mortar mixtures, casting surface

Interpretation of results

First of all it is very interesting to see that both the surface water absorption and the cube water absorption follow an equal trend when it comes to what mix design has most water absorption and what mix design has least water absorption.

There are two other clear observations: With increasing water-to-binder ratio, the water absorption of the specimens seems to increase (M01, M02, M03 with 0.35 W/B, M04, M05 with 0.4 W/B and M07, M09 with 0.45 W/B). There is also a slight tendency for the calcined clay specimens to absorb more water. When comparing M01, M02 and M03, it is clear that the water absorption keeps increasing whilst the calcined clay content also increases (M01 = 0 %, M02 = 25 %, M03 = 50 %).

Another interesting parameter to observe is the volume of permeable pore space from Table 6.1. The total volume of permeable pore space provides an indication of the amount of pores that is present in the specimens. Here, pore means the open spaces in the specimens that can be filled and permeability means the interconnectivity of the pores. A high permeable pore space means that a specimen contains large pores which are permeable. In the case of freezing and thawing this implies that a lot of water can be absorbed and can easily penetrate into the specimens. If these pores then have insufficient size, internal cracking occurs. Increased penetration with de-icing solution results in increased scaling.

Specifically observing the results from M03 and M09, reason of highlighting these two mixes becomes clear when presenting the freeze-thaw results, it is clear that both specimens have (within their W/B ratio category) the highest water absorption and the highest volume of permeable pore space. This means that the water is quite easily absorbed but is also able to easily penetrate into the specimens. This water expands during freezing and thawing and internal cracking can result in decrease of compressive strength and when subjected in a de-icing solution, scaling also increases.

6.2.5. Freeze-thaw

Results

For fully elaborating the results of the freezing and thawing resistance, the results from water absorption are combined with two additional parameters:

- Freeze-thaw scaling: Main measurement for determining freeze-thaw resistance. Provides insight how much material is removed from the specimen due to freezing and thawing cycles.
- Freeze-thaw compressive strength: Strength degradation is a good indicator for freezing and thawing performance. Also provides estimation of the amount of internal cracking as internal cracking causes degradation in compressive strength.

The next pages first provide the scaling results (Figure 6.18), compressive strength results (Figure 6.19) and Taguchi trends (Figure 6.20 and Figure 6.21) and these results are first elaborated with visualizations of the degradation as well. These results combined with the water absorption results from section 6.2.4. provide insight in the deterioration mechanisms due to freezing and thawing cycles in alkaline activated mortars.

Please note the two different types of results in Figures 6.19 and 6.20. Specimens are cast and tested at the same time, but once ready for freezing and thawing a distinction is made:

- Samples that *are* subjected to freezing and thawing, named as: x cycle FT
- Samples that *are not* subjected to freezing and thawing and remain in the curing room, named as: x cycle no-FT

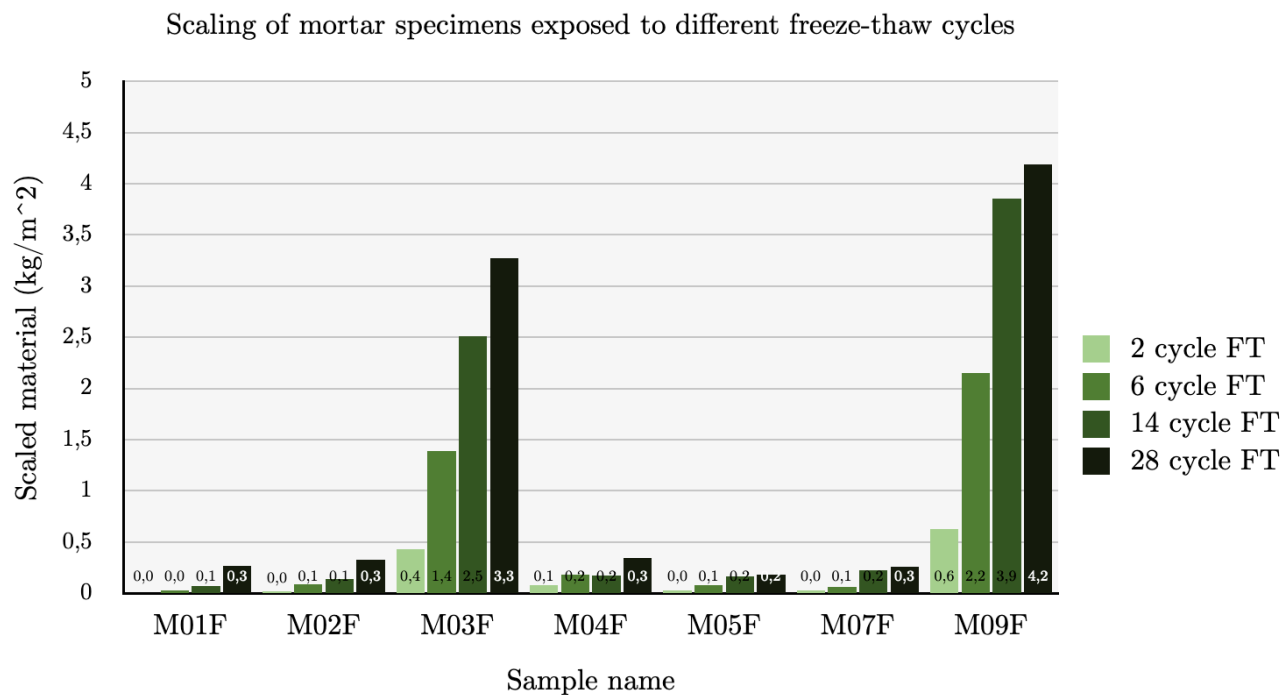


Figure 6.18 - Freeze-thaw scaling of mortar specimens

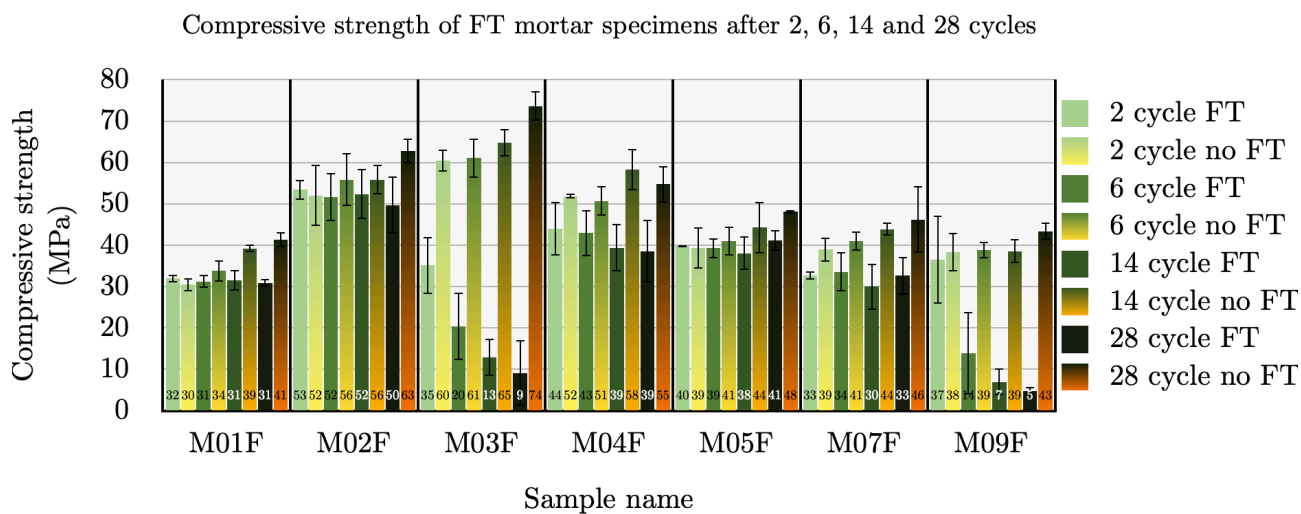


Figure 6.19 - Compressive strength with and without freeze-thaw cycles of mortar specimens

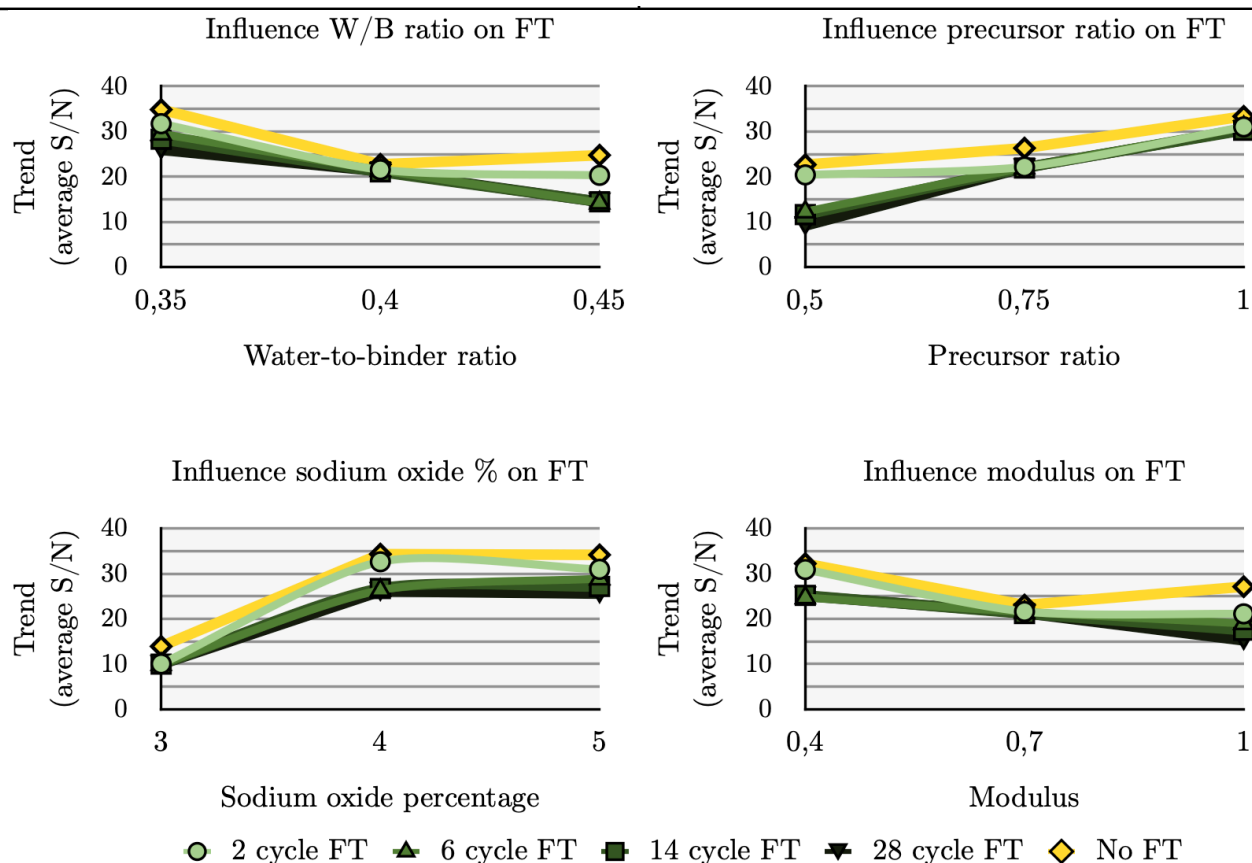


Figure 6.20 - Taguchi trends for freeze-thaw resistance of mortar specimens

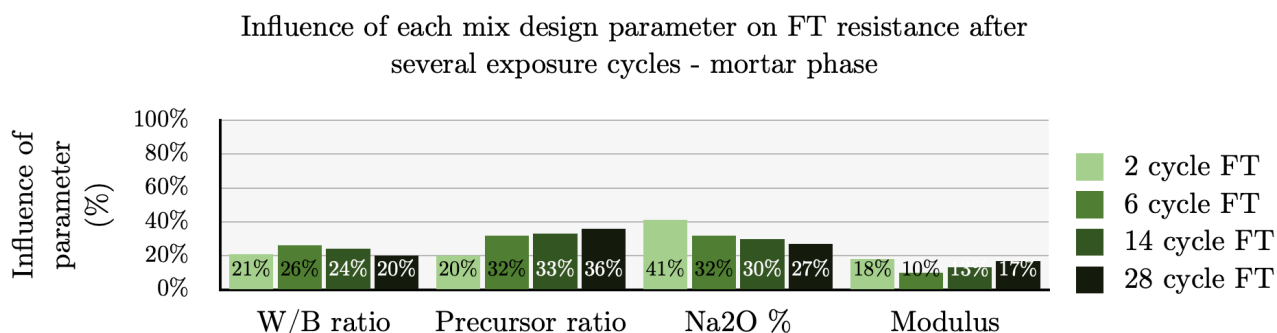


Figure 6.21 - Influence of each mix design parameter on freeze-thaw performance

Please note that Figure 6.23 on the next page contains images of three side views of each cube. When a cube is cut out of the prism, the following definitions are used:

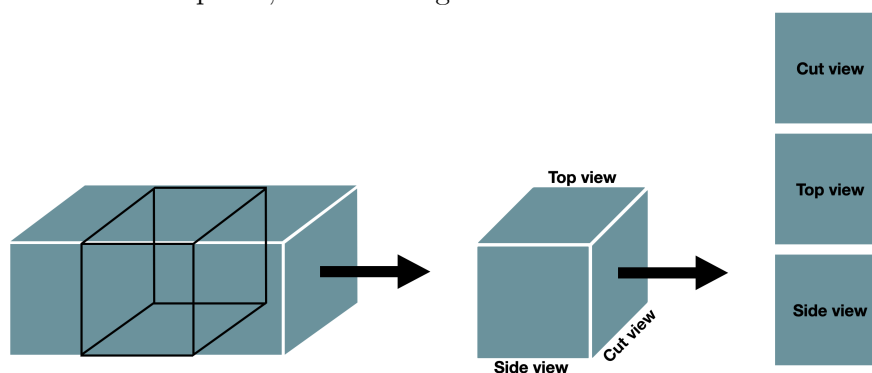


Figure 6.22 - View of specimen definition

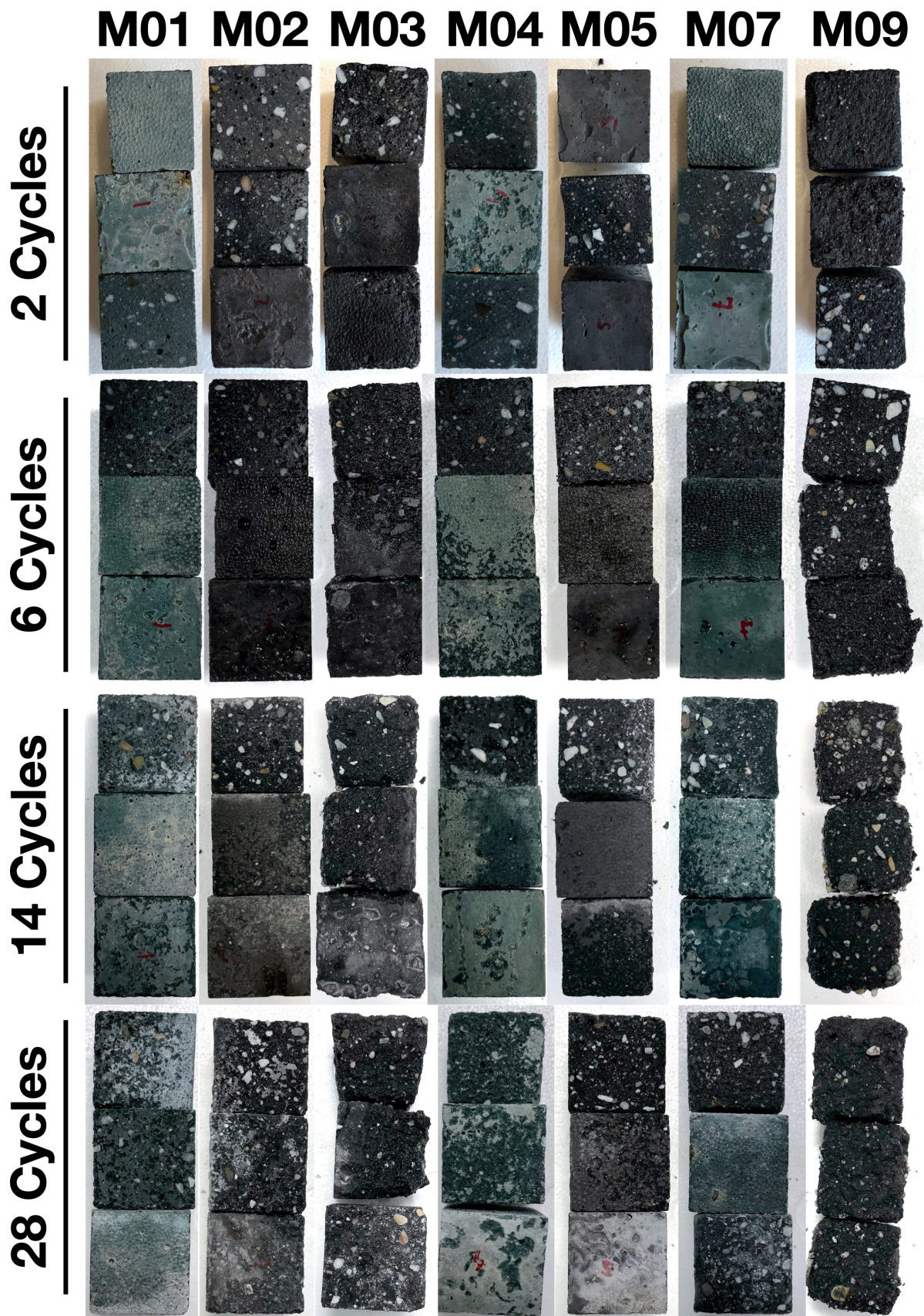


Figure 6.23 - Freeze-thaw degradation visualization over testing period (For each block, a cut view (top), top view (middle) and side view (bottom) is provided)

Interpretation of results

Freeze-thaw degradation visualization over testing period - Figure 6.23

From left to right, mix designs M01, M02, M03, M04, M05, M07 and M09 are visible. At only 2 cycles, damage starts to occur for some specimens, like M03 and M09. However, it is most interesting to observe the damage at 28 cycles. It is quite clear that specimens M03 and M09 suffer a lot of damage. It also seems M01, M02 and M05 barely got damaged at all.

Freeze-thaw scaling and compressive strength capacity - Figures 6.18 and 6.19

Figure 6.18 shows the total amount of scaling of the specimens after x amount of cycles. This scaling can be interpreted as the total material loss of the specimens, converted to kg/m^2 which is normal in the standard. According to the requirements, the scaling should average below $1.5 \text{ kg}/\text{m}^2$. However, this is not a concrete but a mortar and the testing method is adapted from the concrete norm. These specimens show a good indication for performance under freezing and thawing as the testing conditions are very harsh due to the complete submersion in the de-icing fluid. The results from Figure 6.18 can be very nicely coupled to the visualizations in Figure 6.23. It is indeed true that specimens M01, M02 and M05 prove to have little scaling after 28 cycles and that specimens M03 and M09 have significant amount of scaling.

Figure 6.19 also shows interesting results. For each mix design, both a reference (not subjected to freezing and thawing) and tested (subjected to freezing and thawing) specimen have been compressed until failure. The reference specimens remain in the curing room whilst the tested specimens are situated in the freeze-thaw setup. It is immediately clear that specimens M03 and M09 show a decrease in compressive strength with increasing freeze-thawing cycles. This correlates well with the scaling graph as these specimens also lost most material. Specimen M04 is also very interesting. With a continuous increase of scaling, the effect on compressive strength capacity is clear: it continuously decreases. M04 also uses the 1.0 value for modulus. A higher modulus decreases permeability and porosity. Due to lower permeability and less pores, internal cracking is more likely to occur. This phenomenon can be further explained as there is a decrease in compressive strength visible, but no significant increase in scaling. Finally, M01, M02 and M05 do show a decrease in compressive strength capacity compared to their references, but no decrease of compressive strength over time. It seems like the cold climate of the freeze-thaw specimens does stop development of compressive strength but as the specimens have little scaling it does not seem that the specimens are likely to fail soon.

Taguchi trends for freeze-thaw resistance of mortar specimens and influence of each mix design parameter - Figures 6.20 and 6.21

The trends that are observed in previous experiments are similar until after 6 or more freeze-thaw cycles. The trends start to shift a little until a near-equilibrium is reached. These shifts in trends are the first indications that alkali activated mortar has certain preferences for the mix design parameters in order to have sufficient freeze-thaw performance. While these preferences are in line with previous results, they are way more sensitive.

According to the precursor ratio, introduction of more calcined clay results in worse freeze-thaw performance. This is clearly visible in the results, as the samples with the high content calcined clay are the worst performers (M03, M09). This worse performance is a logical result, as the stickiness of the calcined clay affects compaction of the mortar, increasing porosity of the specimens. This increase in porosity also increases permeability of the specimens and calcined clay also results in a less refined pore structure (Zhu et al., 2021). Besides, the calcined clay also causes higher percentage of permeable pores and higher capillary sorptivity (Borges et al., 2016). High amount of permeable pores and high sorptivity results in large amounts of scaling, as proven by M03 and M09. As the scaling is so significant, a very large decrease of compressive strength, as clearly visible, is expected.

A preference in modulus is also visible, with higher preference for a lower modulus. Higher modulus is known to increase shrinkage, which in turn can cause micro cracking within the specimen, therefore promoting penetration of the de-icing solution in the specimens resulting in more scaling (Chen et al., 2021). This increased effect is also visible in Figure 6.21, which shows the effect of each mix design parameter on compressive strength development for specimens subjected to freezing and thawing. The precursor ratio plays an increasingly more significant role in freezing and thawing performance when the amount of cycles increases.

Conclusion

While freezing and thawing performance is sufficient for most specimens, it is clear that some trends start to develop. The precursor ratio seems to play a significant role in final freeze-thaw performance as inclusion of too much calcined clay is likely to hinder the pore refinement and therefore weakening the specimens subjected to freezing and thawing. It is also clear that a strong specimen does not necessarily mean that it performs well under freezing and thawing, as clearly proven by specimen M03. While it is difficult to exactly pinpoint the degradation mechanisms for alkali activated mortars subjected to freezing and thawing, it is clear that these mechanisms are different to regular cement concrete. Internal pore structure, precursor content and possible formation of microcracks define freeze-thaw performance for alkali activated mortar specimens. The Taguchi trends confirm this tendency, as it is clear that the precursor ratio has large influence on freeze-thaw performance as does the modulus.

6.2.6. Sulfate attack resistance

Results

The sulfate attack resistance results are presented in the same manner as previous sections, thus with three figures containing strength and trend information. Besides these figures, an additional figure is presented which shows the degradation of the specimens throughout the testing period.

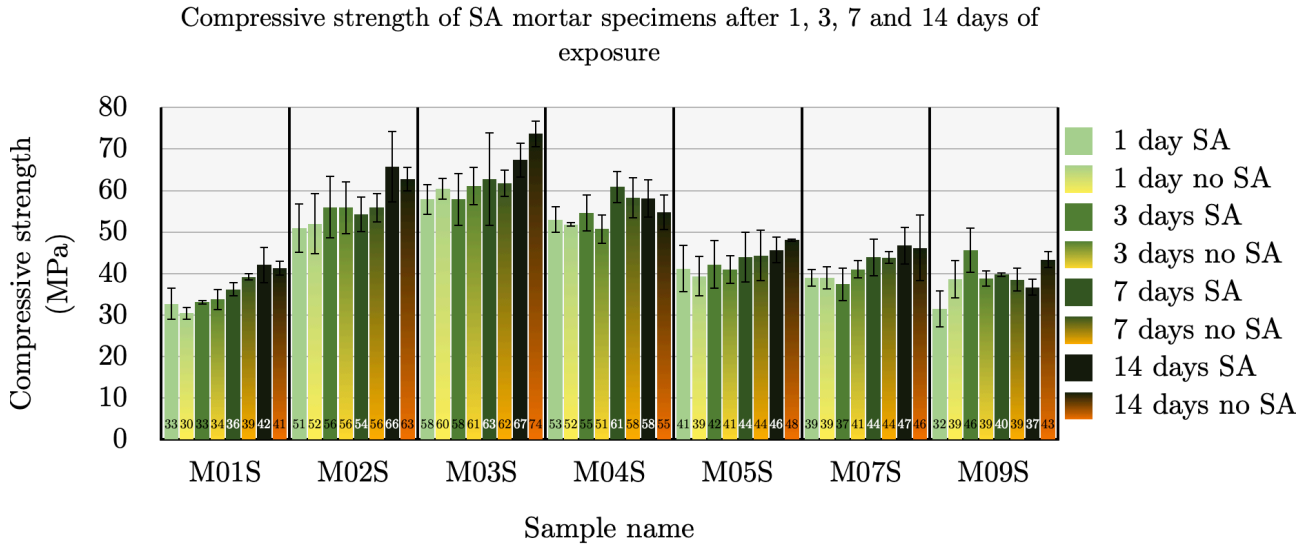


Figure 6.24 - Compressive strength development of specimens with and without exposure to sulfate attack

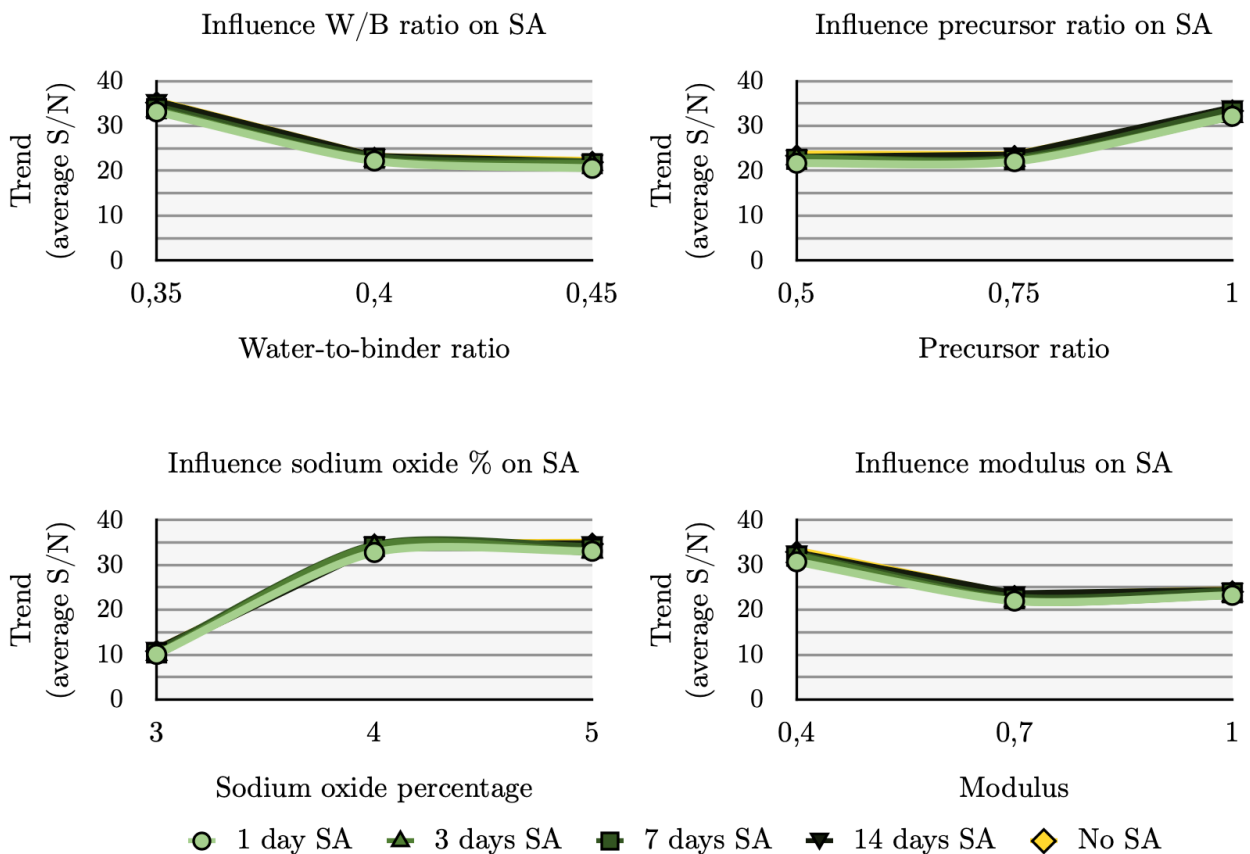


Figure 6.25 - Taguchi trend development of mortar specimens with and without exposure to sulfate attack

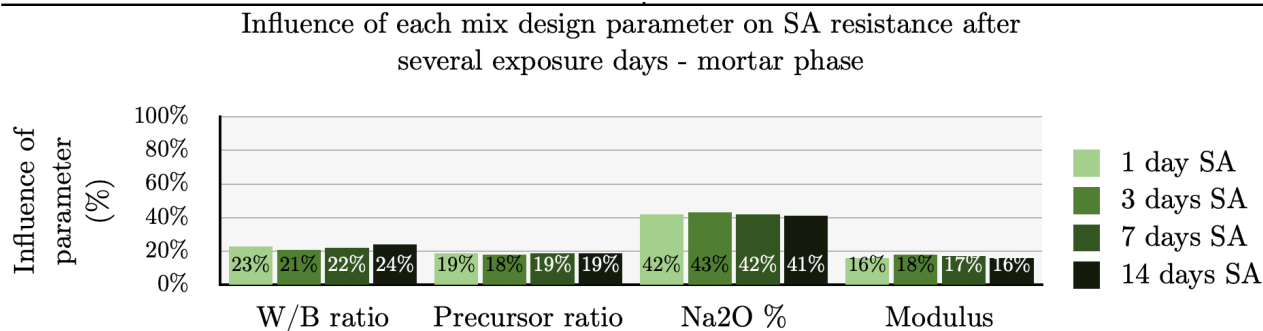


Figure 6.26 - Influence of each mix design parameter on sulfate attack resistance strength development

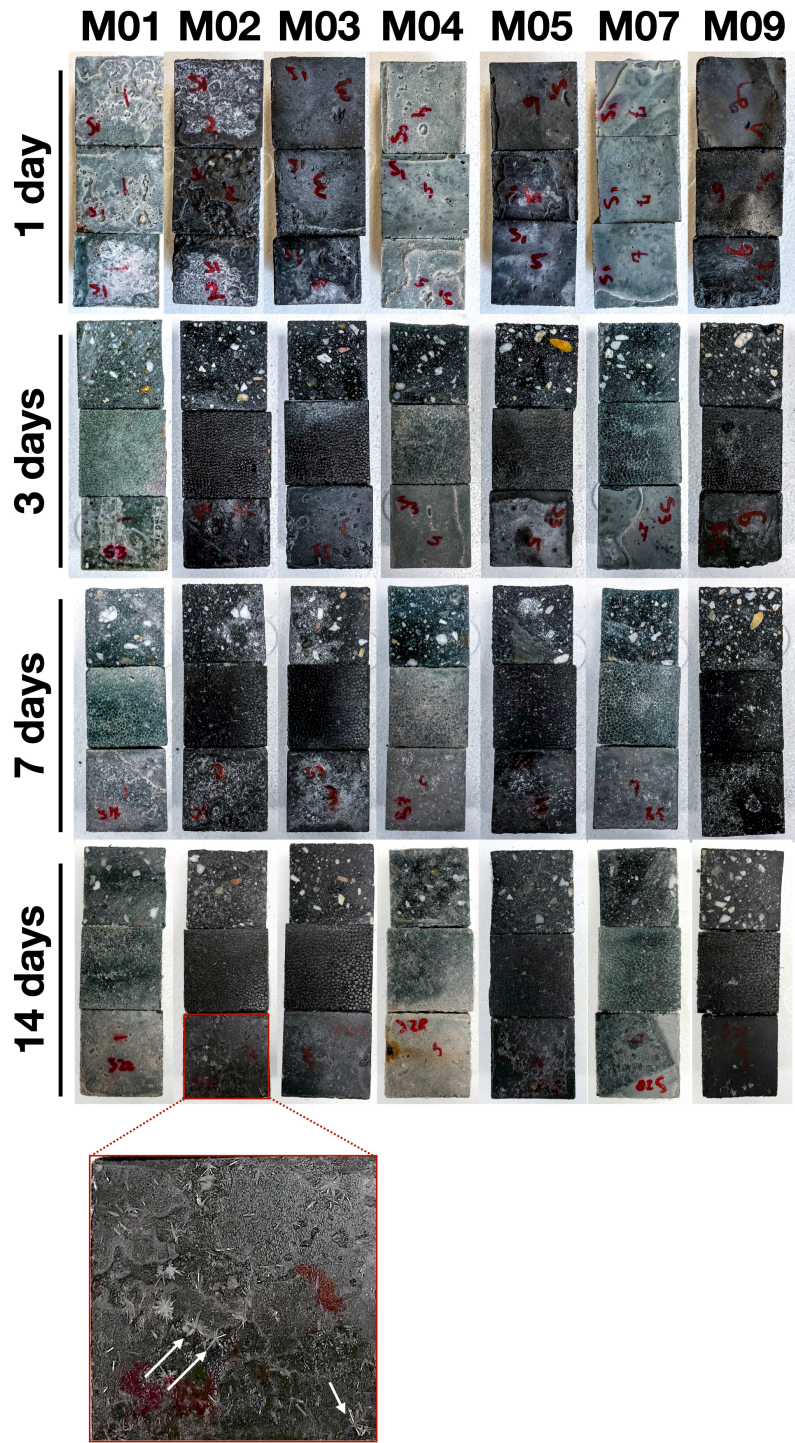


Figure 6.27 - Sulfate attack degradation visualization over testing period (For each block, a cut view (top), top view (middle) and side view (bottom) is provided).

Interpretation of results

Compressive strength development of specimens with and without exposure to sulfate attack - Figure 6.24

For all specimens there is no significant degradation of strength development due to exposure to sulfate attack as both the exposed and non-exposed specimens have nearly equal result and deviations fall within the margin of error.

Taguchi trend development and influence of mix design parameter on sulfate attack resistance - Figures 6.25 and 6.26

As there is no decrease in compressive strength capacity compared to non-exposed sulfate attack specimens, it is no surprise that both the Taguchi trend development and the influence of mix design parameters on strength development show similar results to normal strength development.

The fact that no damage is really observable can be due to the fact that the testing duration is too short for actual damage to occur, but as the solution to which the specimens are exposed has very high concentration and actual sulfate concentrations in natural resources is very low ($< 1\%$) and there is no visible degradation in strength development the sulfate attack resistance seems no problem for the tested mix designs.

Sulfate attack degradation visualization over testing period - Figure 6.27

The figure shows what the specimens look like during the testing period and in most cases shows three different faces of the specimen. All specimens show some degree of crystallization, indicating formation of ettringite on the surface of the specimens. Ettringite formation can result in expansion of the material causing internal or external cracks. While cracks are not visible after 14 days in the high concentration solution, formation of ettringite can indicate some degradation in later stages. More interesting is that more ettringite is observed for the specimens that contain calcined clay, of which Figure 6.27 shows the specimen with most ettringite, where crystals are indicated with the white arrows.

Conclusion

Sulfate attack does not result in actual degradation of the mortar specimens, but some formation of ettringite is observed. At this stage the ettringite forms no problems as no degradation of strength is observed. As concentration of sulfates in sea-water is much lower than the tested concentration and as no strength degradation is observed for the testing period, sulfate attack is not considered a problem for alkali activated mortar specimens. As testing duration during the concrete phase is also 14 days, the concrete is not tested for sulfate attack as no damage is expected and because sulfate attack should be tested for a longer period of time.

6.2.7. Mortar phase conclusion

The mortar phase proves to be a success. Just like the paste phase, only 2 out of 9 specimens (same mix designs) do not develop any strength. Of the other 7 mix designs, nearly all reach the compressive strength target after 1 and 28 days. Workability is still good but now that sand is introduced the spread in workability is quite different. The mix designs with calcined clay prove to be more difficult to handle even though the workability does not indicate that due to the cohesiveness of the mix design. Still, workability is heading in the right direction and it is expected that not too much modifications are required for the best candidates to be have like earth moist concrete in the concrete phase.

This time freeze-thaw resistance and sulfate attack resistance are tested as well. Sulfate attack resistance proves to be very good due to no degradation in strength development of the specimens. Therefore it is decided to not test sulfate attack resistance in the concrete phase. Freeze-thaw resistance is generally good with quite some specimens falling within the required scaling criterion, but the calcined clay definitely has negative influence on freeze-thaw resistance.

The mix designs generally behave well, but a decision must be made for the mix designs that are used in the concrete phase. The performance and environmental footprint, of which the latter is determined in the next chapter, of all mix designs are evaluated and presented in Table 6.2 below.

Table 6.2 - Mortar phase performance comparison

Mix design	Workability	1 day compressive strength	28 day compressive strength	Sulfate attack	Freeze-thaw resistance	MKI
M01						
M02						
M03						
M04						
M05						
M06						
M07						
M08						
M09						

Table 6.2 represents relative performance of the mix designs coupled to the types of experiments. Green represents a clear pass, yellow represents tight performance and red represents insufficient performance. Of all 9 mix designs there are only two that score green on each requirement and are therefore the two mix designs that are to be upscaled: M01 and M05.

M01: This mix design has the lowest environmental footprint of all mix designs whilst also fulfilling all requirements. It is a great mix design to prove what alkali activated concrete can do with minimal environmental footprint and is expected to fulfill the requirements once directly upscaled to concrete.

M05: This is a great performance mix design. The calcined clay proves that it is a little more difficult to handle due to the cohesiveness of the mix. However, it has great performance in all tests from the mortar phase and still has quite a low environmental footprint. Therefore it is a great mix design to be upscaled to concrete.

6.3. Concrete phase

This final section of the experimental research is of great importance. After investigating 9 mix designs at a paste and mortar level the two best mix designs, M01 and M05, are directly upscaled to concrete where M05 needed slight modifications in total moisture content as the resulting mix design as upscaled proved to be little too moist for the small compaction machine provided by industry. The final two alkali activated concrete mix designs are from now on referred to as C01 and C05. C01 and C05 are put through the entire testing program, consisting of workability, compressive strength, water absorption and freeze-thaw resistance and are directly compared to a reference concrete from the industry whilst using the exact same aggregate proportions and aggregate types.

For compacting the concrete, a special compaction machine from the industry is used which allows to combine vibration and application of pressure for optimal compaction. However, due to scale differences with regards to the compactor used in the factory process, results of the concrete phase may still deviate from the actual concrete produced in the factory. This effect, caused by compaction, is elaborated more thoroughly in appendix E.

6.3.1. Workability

Requirement

The concrete should be classified as “earth moist” which corresponds with a compaction value between 1.26 and 1.45.

Results

Results are presented in Figure 6.28 below and Table 6.3 on the next page.

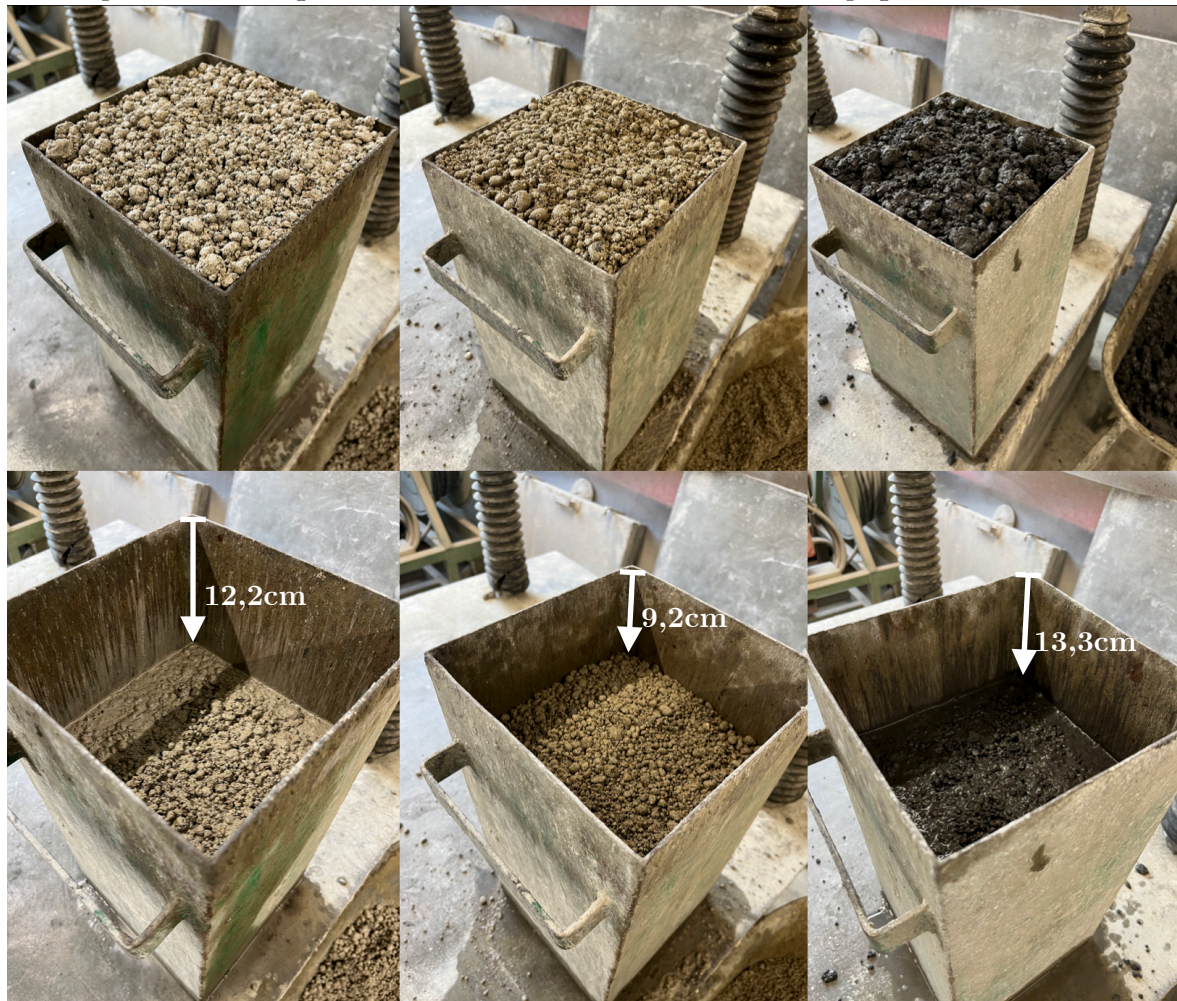


Figure 6.28 - Workability but visualized, from left to right: C01, industry reference, C05

Table 6.3 - Workability results

Mix design	Explanation	Workability result (cm)	Compaction range	Compaction value
C01	Direct upscaling of M01, industry aggregates	12,30	1,26-1,45	1,44
C05	Direct upscaling of M05 with slight adjustment of total activator content	13,30	1,26-1,45	1,50
Industry reference	Testing of industry provided concrete mix design	9,20	1,26-1,45	1,30

From Table 6.3 it is clear that C01 directly falls within the required workability range but as the compaction value is on the high side, it indicates that the concrete is quite dry as drier mixes tend to compact more. C05 falls just outside the boundaries set for earth moist concrete and the industry reference concrete falls within the earth moist concrete requirement.

Interpretation of results

While both alkali activated mix designs barely fall or do not fall within the earth moist concrete region, it does not mean that they are not suitable for production. As Figure 6.28 clearly shows, both the alkali activated mix designs show more signs of moisture than the industry reference. This can partially be attributed to bleeding, where water rises to the upper layer of the concrete, but after removal of the mould it is clear that the alkali activated mix designs show more moisture signs than the industry mixture, even though they are on the dry side as implied by the ranges provided by the norm.

According to the concrete norm, the alkali activated mixtures do not meet the earth moist requirement. However, the alkali activated mixtures behave differently compared to regular cement concrete. Therefore it is possible and also recommended that adjustments to the workability ranges are made for the definition of earth moist concrete when it comes to utilizing alkali activated concrete instead of cement concrete. The tested alkaline activated concrete proves suitable for earth moist application, whilst the norm states that it is barely (not). After all, the norms are all based on cement concrete, but that does not necessarily mean that it can all be directly be applied to alkali activated concrete.

Conclusion

C01 reaches the norm requirement for earth moist concrete, but C05 does not. However, due to visual observation and due to the fact that the mixtures perform great in the small compaction machine provided by the industry, both mix designs are considered successful in terms of their workability.

6.3.2. Compressive strength Requirement

The concrete should be at least of grade C30/37 to be used in concrete revetment products and as set requirement by the industry. The industry also has a 1 day strength development requirement as the concrete only cures for 24h and is then stacked on top of each other. Therefore, the concrete should reach at least 12 MPa 24 hours after casting and should reach at least 37 MPa 28 days after casting.

Results

Before providing the final results, the curing method should be elaborated as it deviates from the paste- and mortar phase due to a different production location.

The alkali activated concrete spent the first 24 hours of curing in the hall where it is produced. According to KNMI (the royal Dutch meteorological institute), the average temperature during those 24 hours was 23 °C. This means that it cured under slightly higher than ambient temperature.

The reference mix spent the first 24 hours of curing in the curing hall of the industry, where the temperature averaged 32 °C with 99 % humidity.

The alkali activated concrete mix designs and the reference mix design had different curing conditions in the first 24 hours to prove that alkali activated concrete does not need the high 32°C curing temperature to reach high early strength, which the reference concrete does need. After the first 24 hours, all mix designs are transported by car and put in the curing room at TU Delft (20 °C with 99 % humidity) where conditions are identical to the paste- and mortar phase.

The compressive strength results are presented in Figure 6.29 below:

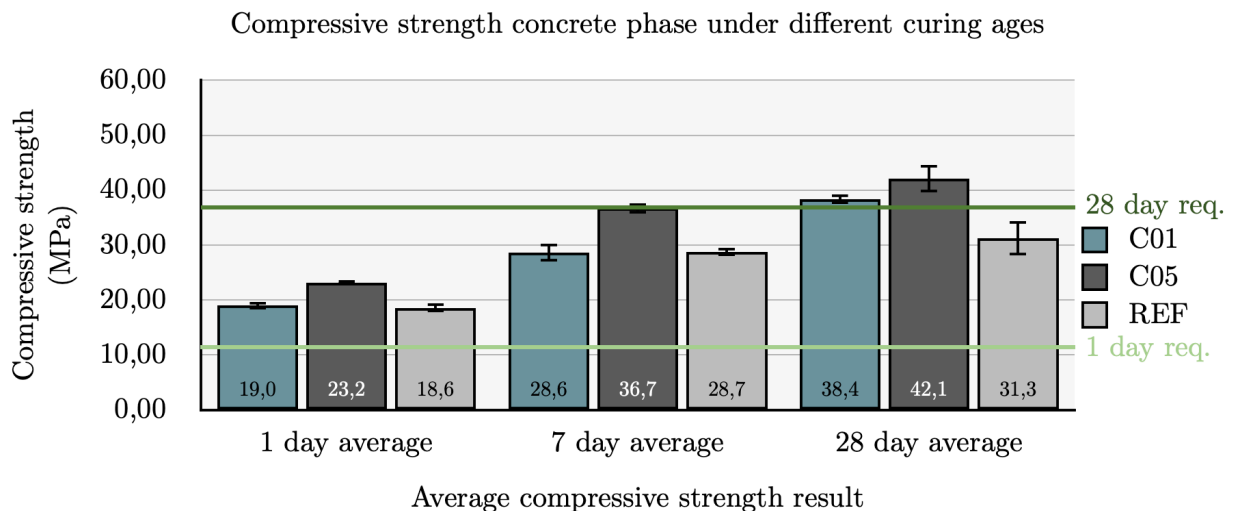


Figure 6.29 - Compressive strength development of concrete specimens at different curing ages

Interpretation of results

Figure 6.29 displays the average compressive strength at 1, 7 and 28 days curing of C01, C05 and the reference mix design. The light green line represents the 1 day compressive strength requirement, every mix design above this line fulfills the requirement. The dark green line represents the 28 day compressive strength requirement, every mix design above this line fulfills the requirement.

As can clearly be observed, all three mix designs successfully succeeded in reaching the compressive strength target after 24 hours of curing. Strength develops continuously where after 7 days C01 and the reference mix perform nearly equal with 28,63 MPa and 28,73 MPa respectively. Mix C05 averages at 36,67 MPa after just 7 days, almost meeting the requirement after 28 days of curing. C01 and C05 follow a nearly equal trend to both their paste and mortar mix designs.

After 28 days curing, both C01 and C05 manage to reach the requirement of 37 MPa. C01 averages 38,4 MPa with no values below 37 MPa and C05 averages 42,1 MPa. Interestingly, the industry reference concrete does not reach the 37 MPa target, but it does so in the factory. The original factory mix design is adjusted slightly as the mix design had higher water demand for the small compaction machine, but as the water-to-cement ratio is a strength controlling parameter for cement concrete, more cement was added to prevent significant changes in this ratio. However, from visual observation for all specimens, it is clear that the small compaction machine manages to properly compact the bottom half of the specimens, but not necessarily the top half of the specimens. This is further visualized in Figure 6.30 on the next page.

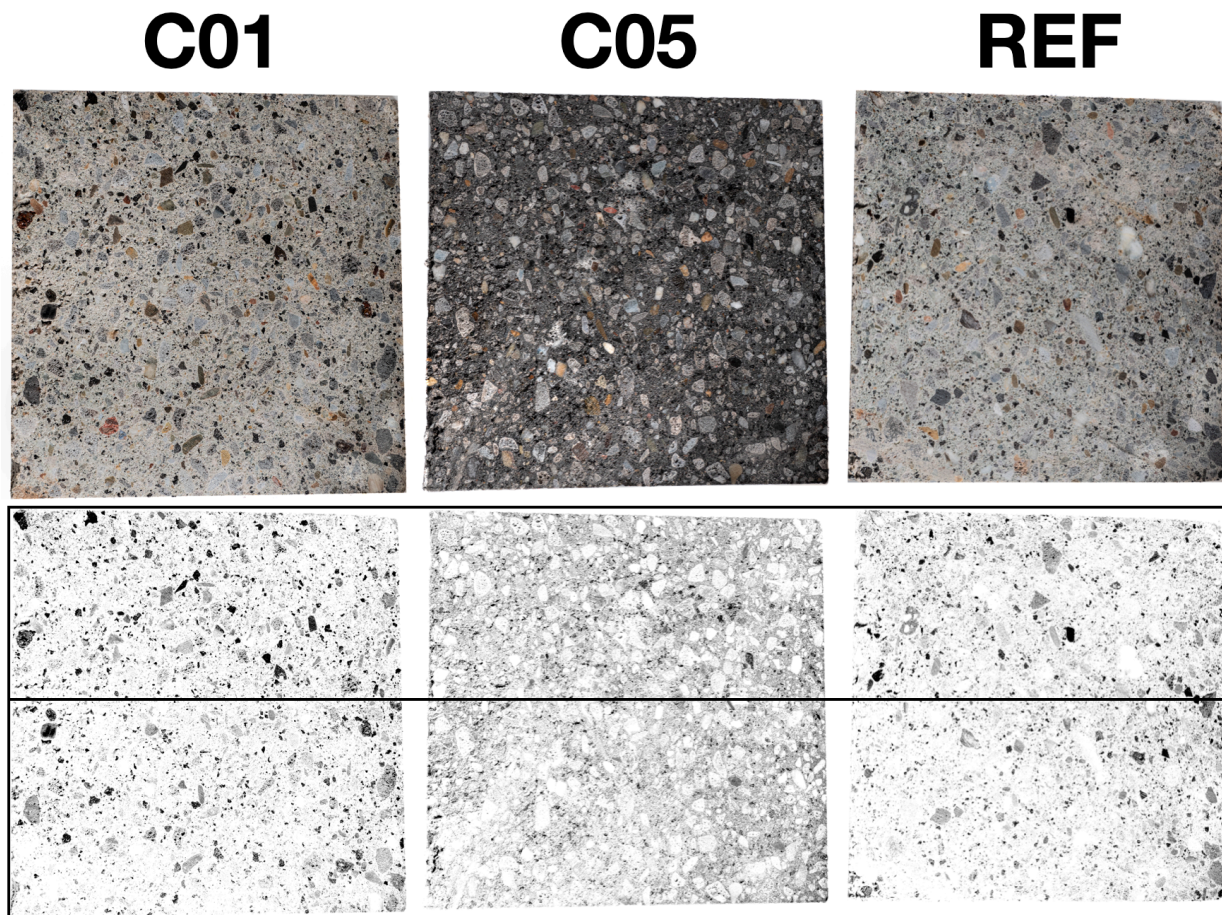


Figure 6.30 - Visualization of pores of concrete specimens

The top images show C01, C05 and the reference mixes in color, while at the bottom images some modifications have been made to better visualize the pores. As can be seen for all three specimens, there are more dark spots (which equal pores) in the upper half of each cross section. While some dark spots originate from aggregates, the difference between the amount of dark spots between the lower half and top half of the cross sections is quite clear. It verifies that compaction of the cubes is not optimal and it also does not completely represent the concrete that is produced at the factory.

Still, this means that results are an underestimate of what the concrete is able to do in the actual production process. As the alkali activated mix designs already agree with the compressive strength requirement there should be no problem for this requirement should these mix designs be used in the production process.

Once again the requirement by the concrete norm should be mentioned. It is clear that the alkali activated mix designs meet the 37 MPa requirement, but alkali activated concrete continues to develop strength over time, which means if the norm incorporates this effect, the total activator content can be lowered and optimized resulting in a reduction of environmental footprint (Human et al., 2021).

Conclusion

Compressive strength development of the alkali activated mix designs fulfill the requirements with strong early strength development and good 28 day strength development. While the industry concrete does develop good early strength, it does not reach the 28 day requirement, but that is mainly a result to non-optimal compaction. For the alkali activated mix designs, the need to incorporate optimization in the industrial production process is clear due to non-optimal compaction by considering different aggregate proportions, but also different precursor amounts

and the possibility for a decrease in activator solution under the condition that the workability requirement is still met.

6.3.3. Water absorption

Requirement

The total average water absorption should stay below or equal 6 %.

Results

The results are presented in Table 6.4 below.

Table 6.4 - Average water absorption values of concrete specimens

Mix design	Saturated weight (g)	Dry weight (g)	Water absorption (%)
C01	3940,9	3670,6	6,86%
C05	3938,1	3650,4	7,31%
REF	3858,8	3587,8	7,02%

Interpretation of results

The table shows the average values of the saturated weight, dry weight and total water absorption. As clearly visible, all three specimens do not meet the requirement. C01 averages at 6.86 %, C05 at 7.31 % and the reference at 7.02 %. Most likely the compaction of the specimens has influence on the water absorption. As mentioned, the amount of pores in the specimens in the top half is very high as seen in Figure 6.30. This is true for all cast specimens, meaning that the top half of each specimen is considered not to be compacted sufficiently. Whilst the small compaction machine of industry provides the best possible compaction results during the span of this thesis, it is not capable enough to accurately represent the compaction of the machine inside the factory process.

As even the reference concrete from the industry does not meet the requirement, ongoing investigation is necessary to confirm the worse compaction caused by the small compaction machine which is unfortunately not possible in the timespan of this thesis. However, C01 manages to have less water absorption than the reference and C05 only has little more water absorption than the reference, meaning that if the mix designs can be compacted in a better way, it is well possible that both alkali activated mix designs fulfill the requirement.

Conclusion

Water absorption for all three specimens is too high, mainly caused by the limited compaction from the small compaction machine provided by the industry. Still, it is considered the best possible compaction method for the timespan of this thesis. However, it is expected that optimizing the mix designs to the industrial production process can lower the water absorption to meet the standard requirement.

6.3.4. Freeze-thaw resistance

Requirement

Scaling should be $\leq 1,5 \text{ kg/m}^2$ after 28 cycles of freezing and thawing.

Results

The visualized scaling is presented in Figure 6.31 on the next page and the numerical values are presented in Table 6.5 on the page after.

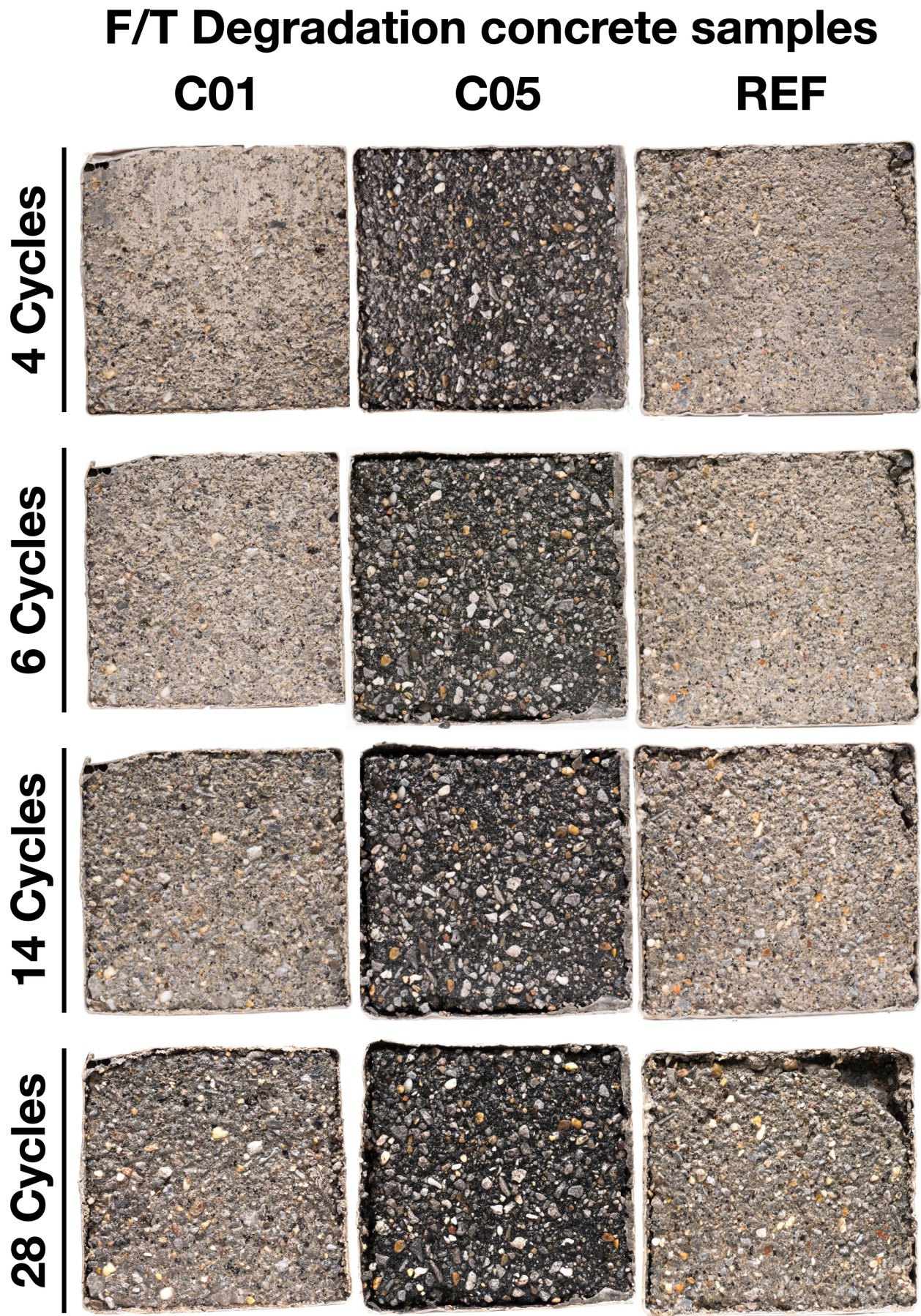


Figure 6.31 - Freeze-thaw deterioration of 3 concrete mix designs up to 28 cycles

Table 6.5 - Freeze-thaw scaling results

Mix design	Average scaling (kg/m ²)	Standard deviation	Scaling best specimen (kg/m ²)
C01	5,00	1,70	3,04
C05	5,17	0,16	5,07
REF	6,62	4,10	3,94

Interpretation of results

All mix designs fail to meet the requirement for the CDF test as clearly stated in Table 6.5. C01 has the lowest average scaling after 28 cycles, followed by C05 and lastly the reference. The reference has high average scaling and high standard deviation due to 1 specimen with very significant scaling. Without this specimen, the average is in the region of 4.30 kg/m³. Still, the best specimen even surpasses the norm stated 1.5 kg/m³ by 2 times. One of the potential causes for the insufficient performance is the compaction of the specimens. As mentioned before, the specimens have a lot of visible pores and not a very dense matrix. A worse compaction resulting in increase in scaling and decrease in mechanical properties after freezing and thawing cycles is also observed by Shahrajabian & Behfarnia (2018) and Coppola et al. (2020). The reference concrete also fails to meet the requirement, further substantiating the effect of compaction on freeze-thaw performance, as the same recipe but produced in the factory does fulfill the freeze-thaw requirements.

As explained in appendix E, the small compaction machine provides an optimal compaction region with parabolic shape. Outside of this region, compaction is lower and worse freeze-thaw performance is expected at those regions. Now observe Figure 6.32 below.

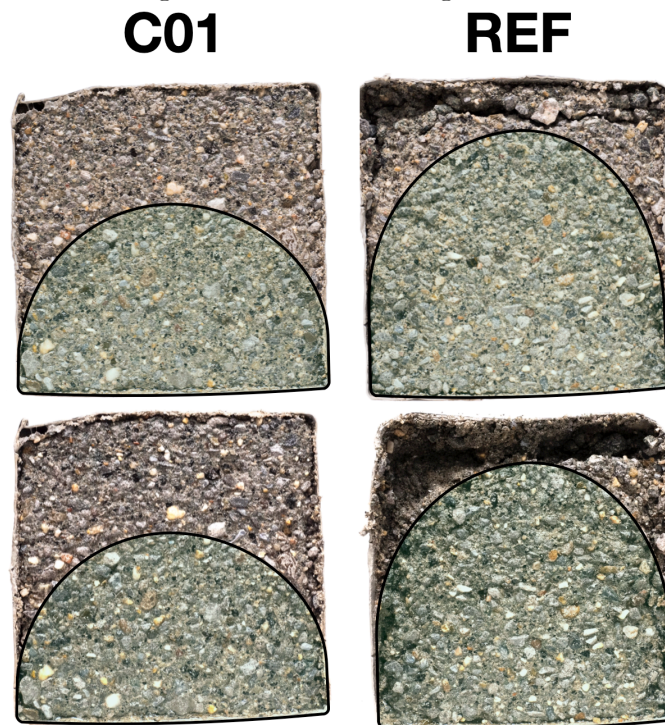


Figure 6.32 - Compaction and freeze-thaw resistance

For some specimens, the parabolic shape is observed. This implies that for both the reference concrete and alkali activated concrete, compaction is very important to make the concrete resistant to freezing and thawing. One of the reference specimens, Figure 6.32 right, clearly shows the parabolic shape of optimal compaction.

While compaction is most likely the main contributor to the non-sufficient freeze-thaw performance, other mechanisms also influence the alkali activated concrete freeze-thaw resistance.

The specimens contain high water absorption. This indicates that the outer surface of the concrete allows the de-icing solution to penetrate through the surface. As known, this results in formation of a weakened layer which causes scaling once subjected to freezing.

The concrete mix designs also incorporate different aggregates compared to the mortar phase. These aggregates are provided by the industry and also contains recycled concrete aggregates. Recycled aggregates can induce micro strain in the concrete. This effect is more pronounced for alkaline activated slag concrete than regular concrete whilst recycled aggregates also increase internal porosity. As the specimens have a lot of visible pores, it allows for the de-icing solution to penetrate which once again results in more scaling (Nanayakkara et al., 2020).

The precursor also influences freeze-thaw results. As mentioned at mortar level, inclusion of calcined clay hinders pore refinement and increases pore connectivity. This explains why there is both more scaling and higher water absorption for C05. It is clear that calcined clay can only be applied in limited amount and that optimizations to mix design are necessary for good durability performance.

The progress of scaling is also very interesting. As pointed out in the literature review, scaling progress can be described via an accelerating, linear or retarding curve. The average scaling over time is presented in Figure 6.33 below.

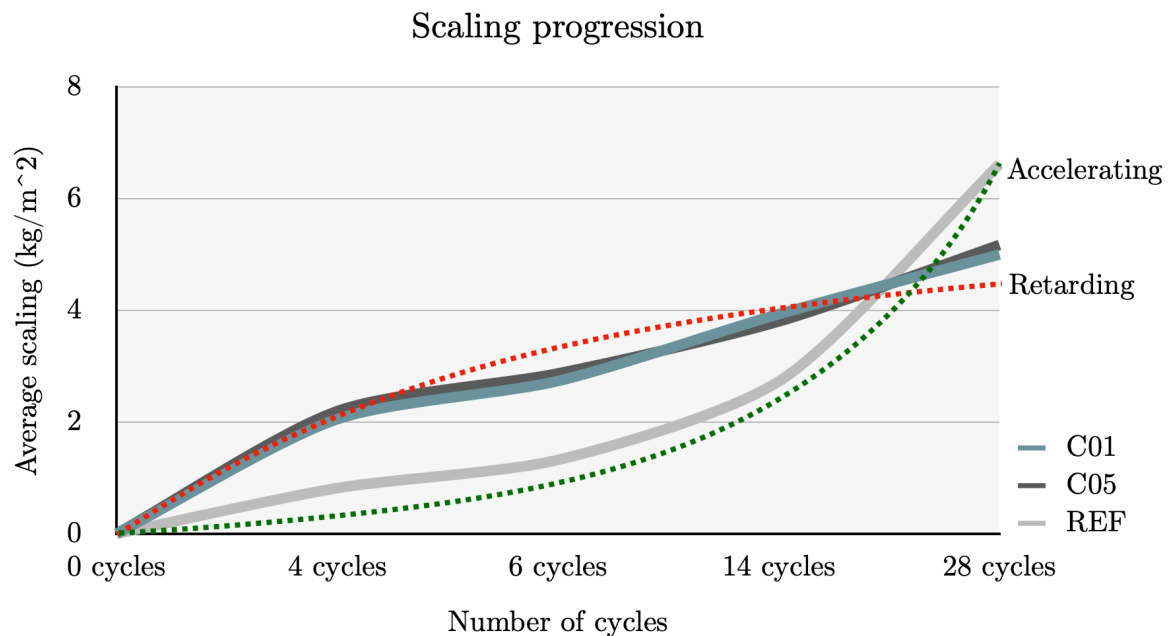


Figure 6.33 - Scaling progression at concrete scale

Both alkaline activated concrete mix designs show retarding behavior (red line): much scaling at the start, but strong decrease in additional scaling while the cycles continue. This indicates that even though scaling is high for the alkaline activated mixtures, it is still rather resistant against freezing and thawing. The reference concrete shows accelerating behavior (green line): little scaling at the start, but much scaling once the number of cycles increases. In terms of lifespan expectation in terms of freezing and thawing, retarding behavior promotes longer lifespan whilst accelerating behavior can cause more sudden failure of the concrete.

Logically, compaction should first be verified to be one of the reasons why all concrete mix designs exceed the requirement stated by the norm. Once verified, it is interesting if the mix designs still show the same scaling progression, as the retarding behavior of the alkaline activated concrete mix designs indicates that testing according to the current concrete norms may not be the way alkaline activated concrete should be tested. Longer testing periods are required and if scaling

continues to follow this retarding line, alkaline activated concrete may reach much longer service life for freezing and thawing than the current norm suggests.

Conclusion

The lack of performance of the alkali activated mix designs is largely attributed to insufficient compaction as the reference concrete also performs far worse compared to the in-factory produced concrete. However, the inclusion of recycled aggregates may also partially attribute to the low performance and it is clear that calcined clay can only be substituted in the precursor blend in limited amount. The retarding behavior of the alkaline activated concrete looks promising and shows that adaptations to the norm can be necessary for alkaline activated concrete.

Still, the performance of alkaline activated concrete should be further investigated by means of ESEM and MIP and by expansion of the freeze-thaw testing variables, also including curing ages and curing conditions, to accurately monitor freeze-thaw performance of alkali activated concrete and to further improve the durability of the material.

6.3.5. Concrete phase conclusion

The concrete phase is partially successful. While the development of compressive strength and suitability in terms of workability of the alkali activated mix designs is clearly proven, its durability is not proven as of yet. The industry reference concrete performs worse on most tests compared to the alkali activated mixtures. This worse performance by the industry reference concrete is largely attributed to the amount of compaction which is as optimal as can be for this research, but clearly is insufficient to provide a proper representation of compaction that is possible in the factory process. The clearly worse performance of the alkali activated mix designs in terms of water absorption and freeze-thaw resistance is therefore also partially attributed to compaction, but the effects of inclusion of recycled aggregates, precursor content and pore size distribution should be investigated more thoroughly to better explain the degradation mechanisms at concrete level.

6.4. Conclusion

The paste phase results in directly usable results for the chosen Taguchi mix design array. The mix designs develop good flexural strength, but leaching and shrinkage are mechanisms that are important to consider as they decrease performance. Workability and compressive strength both provide a nice spread of results even though 2 mix designs do not develop mechanical properties due to a too low alkaline climate.

The mortar phase shows that the introduction of sand causes overall lower workability due to the additional water absorption and causes changes to the influence of the mix design parameters on workability. Flexural strength does not provide signs of leaching anymore and compressive strength is very good, where 4 out of 9 mix designs already reach the final required compressive strength at both 1 day and 28 days of curing. Sulfate attack resistance is sufficient for all specimens where only slight formation of ettringite is visible. As testing duration at concrete level is equally long, the concrete is not tested for sulfate attack resistance. Freeze-thaw for most mixtures is very good. Including calcined clay shows decrease in freeze-thaw performance due to more difficult compaction and higher water absorption and more pore connectivity, resulting in more scaling. Lower modulus causes better performance due to better microstructure development. The water-to-binder ratio and precursor content also has direct influence on water absorption, where samples with high water absorption perform worse in freezing and thawing.

During the concrete phase the final developed alkaline activated mix designs often perform better than the reference cement concrete. The reference fails to meet the compressive strength requirement after 28 days of curing, while the alkali activated concrete meets both compressive strength requirements. Workability results show that the norm may not be completely applicable for alkali activated concrete due to different material behavior. In terms of water absorption and

freeze-thaw, all specimens fail to meet the requirements. However, after visual inspection it is clear that compaction may be one of the contributors to this performance. Inclusion of recycled aggregates is also one of the reasons why performance is lower. Furthermore, the alkaline activated concrete shows very promising retarding scaling behavior, indicating that while scaling numbers are high, it is possible that it is able to withstand more freezing and thawing cycles compared to cement concrete as that clearly shows accelerating behavior.

With these results known, the life cycle analysis or LCA is presented in the next chapter.

Life cycle analysis

7. Life cycle analysis

7.1. LCA reference concrete

7.2. LCA alkali activated performance mix

7.3. LCA alkali activated low MKI mix

7.4. LCA comparison and conclusion

A life cycle analysis is a method to determine the environmental performance of a material or product. This chapter elaborates the life cycle analysis for three different concrete mix designs: The reference mix design in section 7.1, an alkali activated concrete performance mix design in section 7.2 and a low MKI alkali activated concrete mix design in section 7.3. Finally, the data is summarized and concluded in section 7.4.

A brief introduction

A Life Cycle Analysis or LCA is a method that is used to evaluate the environmental impact of a product through its lifecycle (Ecochain, s.d.). Specifically in the Netherlands, the environmental impact consists of at least 11 environmental impact categories. Each category represents a certain harmful emission type, for example the global warming potential category (GWP) is represented by the amount of CO₂ emitted. In order to clearly represent the environmental impact of a product, the shadow price is introduced in the Netherlands. The shadow price represents the governments highest prevention costs per unit emissions control (also known as the amount of money needed in order to compensate for the amount of emitted harmful substances).

For example, the shadow price of 1kg emitted CO₂ is 5 cents. This means that 5 cents should be invested in preventing harmful emissions in order to compensate for the emissions. The MKI, milieukostenindicator, represents the total environmental impact of a product and includes the shadow costs of all 11 environmental impact categories. The higher the MKI, the higher the investment is to compensate for the emitted harmful substances (stichting nationale milieudatabase, 2022). This research strives to develop a new concrete mix design suitable for concrete revetment products with equal performance, but lower environmental impact. This means that the concrete should have a lower MKI value than the reference concrete considered.

General information

This LCA describes product stage A1-A3 of the production of concrete, where three different mix designs are considered: reference concrete, alkali activated performance mix and alkali activated low MKI mix.

Author:	Tom van Rijswijk
Company for which it is performed:	Industry & TU Delft
Date:	27-07-2022

This LCA is performed using demands from Bepalingsmethode Bouwwerken. Note that exact input values are missing for the concrete mix designs due to confidentiality. The alkali activated mix designs can be found in appendix F and the industry mix design in confidential appendix G.

Goal & Scope

Intended application

This LCA is used for TU Delft and the revetment industry to provide information with regards to the MKI of the concrete mix designs, evaluated using reliable quantitative information.

Goal and audience

The goal of the LCA is to provide insight in the MKI of the reference concrete produced at the revetment industry compared to two mix designs developed in this research using reliable sources of information. Both the value of MKI and relative influence of each component is considered. The audience of this LCA is industry and TU Delft.

Functional unit

This LCA describes the production of concrete mix designs, so other life cycle phases are not considered. These are not included as more research is necessary to correctly predict the life span of alkali activated concrete. The concretes in this LCA perform in accordance with the concrete revetment products, meaning that they should be of strength class C30/37, have equal freeze-thaw performance and equal water absorption. As mentioned, full life cycle is not used in this LCA and end of life situation is also not considered, but both are mentioned in the discussion. As the mix designs are not always equal, the functional unit is difficult to define in much detail. All of the

above results in the following functional unit:

“Production of 1 m³ of (alkali activated) concrete of strength class C30/37, with equal performance in water absorption and freezing and thawing.”

Scope

Section A1-A3 are considered in this LCA, meaning that it is a cradle-to-gate situation. Therefore, section A1-A3 form the system boundary within this LCA. The output of the LCA consists of a MKI or Milieu Kosten Indicator score. To determine this score, set one of “Bepalingsmethode milieuprestatie bouwwerken / versie 1.1 (march 2022)” combined with EN15804/A1:2013 is used. The environmental categories considered in this LCA are:

- Abiotic depletion, non-fuel	(ADnf)
- Abiotic depletion, fuel	(ADf)
- Global warming potential	(GWP)
- Ozone layer depletion	(ODP)
- Photochemical oxidation potential	(POCP)
- Acidification potential	(AP)
- Eutrophication potential	(EP)
- Human toxicity potential	(HTP)
- Ecotoxicity potential, fresh water	(FAETP)
- Ecotoxicity potential, marine water	(MAETP)
- Ecotoxicity potential, terrestrial environment	(TETP)

From this point on, the LCA is split into three sections, each describing the entire process of the concrete considered. In the end, the output data is compared.

7.1. LCA reference concrete

Full product description

The concrete mix from the industry is mainly used in revetment products. The most produced revetment product is the quattrobloek, consisting of four polygonal shaped concrete pillars, applied in water-retaining structures like sea dikes. Production takes place in Alphen aan den Rijn. The complete version of Table 7.1 can be found in confidential appendix G.

Table 7.1 - Mix design and transportation distances

Component	Amount (kg/m ³)	Transport type	Distance (km)
CEM III/B	CONFIDENTIAL	Inland vessel	235
Fly ash		Truck	235
Plasticizer		Truck	80
Concrete sand 0-4mm		Inland vessel	165
Copperslag 0-4mm		Inland vessel	164
Blast furnace slag 2-8mm		Inland vessel	252
Concrete recycled sand 0-10mm		Inland vessel	60
Water		-	-
Total		-	-

Quality of used data

The quality of used data can be found in appendix I.

Life cycle inventory

Figure 7.1 shows the process tree. A larger image is available in Appendix I.

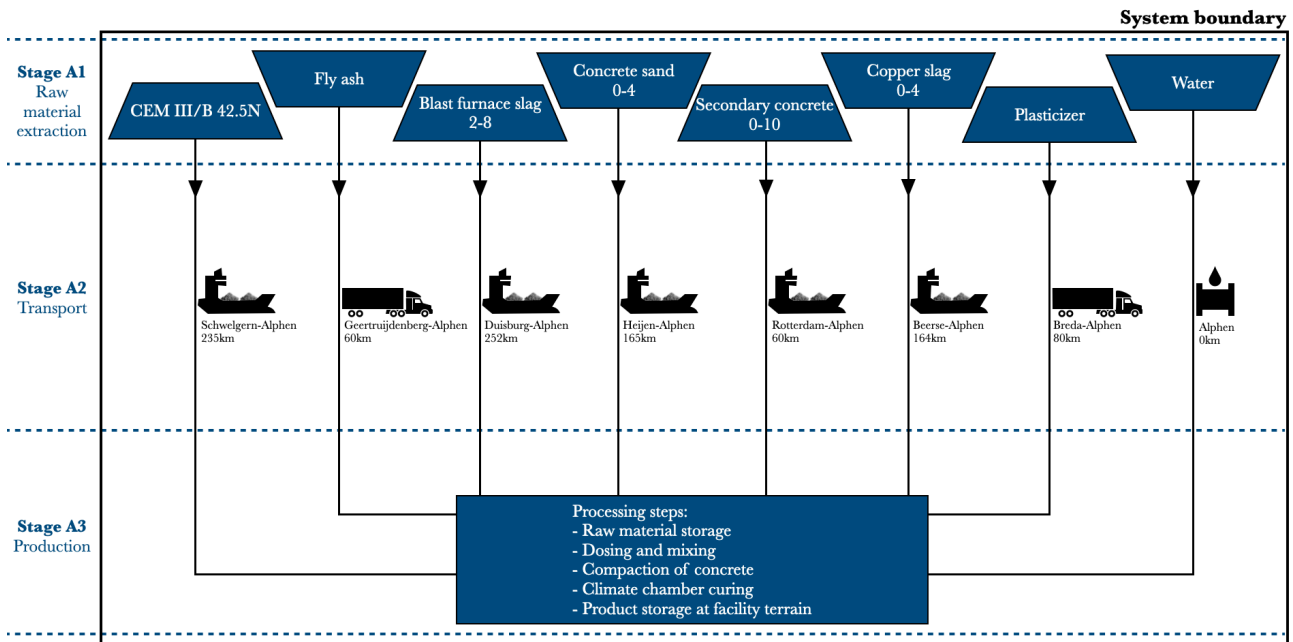


Figure 7.1 - Process tree reference concrete

Elaboration on the process tree

Blast furnace cement (CEM III/B)

The main binder component used for the concrete production is CEM III/B 42.5N, produced by a company in Schwelgern, Germany. Environmental data is retrieved directly from Ontwerptool Groen Beton. The cement is transported by inland vessel over a distance of 235 km.

Fly ash

Another component in the binder is fly ash, a product originating from coal power plants. The fly ash is delivered from both Geertruijdenberg and Rotterdam by truck. The average transportation distance of 60 km is used and for the truck, an average value including empty return trip is used. Environmental data is retrieved from vliegias unie, MRPI code 20.1.0027.005.

Blast furnace slag

This blast furnace slag is used as surcharge material and not as binding component, meaning that environmental data may be put to 0 (free of burden) as it is considered a by-product from the steel industry. However, transportation does introduce emissions and is accounted for. The blast furnace slag is transported by inland vessel from Duisburg, Germany over a distance of 252 km.

Concrete sand

The sand is supplied by Teunesen Zand en Grint B.V., a member of Cascade. Therefore, environmental data from “Industriezand, in en nabij Nederland geproduceerd door Cascade leden, c2” from the Nationale Milieu Database is used. The sand is transported by inland vessel from Heijen over a distance of 165 km.

Secondary concrete

This is concrete that has been in a construction before, but broken down and reused in fresh concrete and therefore originates from construction and demolition waste. Recycling aggregates are end-of-waste, meaning that it is considered free of burden (0 values for all impact categories). Transportation takes place by inland vessel from Rotterdam with a distance of 60 km.

Copper slag

Like blast furnace slag, copper slag is a by-product from the steel industry. It is used as surcharge material and not part of the binder, meaning it may be considered free of burden (0 values for all impact categories). The copper slag is transported by inland vessel from Metallo, Beerse over a distance of 164 km.

Plasticizer

Plasticizer originates from Cugla, specifically Cugla Paving Plast 2500 con.20% PL. General plasticizer impact data from Ontwerptool Groen Beton has been used (SBK BD normal plasticizers, 30 - 45 % active content, 2002). Plasticizer is transported by truck from Breda over a distance of 80 km.

Water

Water is locally used.

Production of the concrete

The raw materials are all stored at the production facility. The cement and fly ash are stored in silo's. The superplasticizer is stored in a small sealed container and the aggregates are stored in non-covered bunkers exposed to outdoor air. Products are weighed and mixed together into a dry cast mixture. This mixture is transported to a mold which is pressed and vibrated to form the final concrete product. This product remains in a climate chamber for 24 hours and are then stored for a minimum of two weeks at the outdoor terrain, exposed to the elements.

During production, a combination of electricity and diesel is used. Per cubic meter of concrete, the following energy is used:

1. Green energy, 11.63 kWh (modeled after NMDv3.0, 53 % wood and biogas, 45 % wind)
2. Diesel, 0.27 l (modeled after Diesel, burned in building machine {GLO}|market for|cut-off)

Total production waste is determined to be 1.2 % and is accounted for in the calculations of the total environmental impact. This waste is deposited as sludge. The design life span for the concrete is 100 years but is not accounted for as only categories A1-A3 (production) are considered.

Life cycle impact analysis

The life cycle impact analysis is shown in Table 7.2 below.

Table 7.2 - Life cycle impact analysis of reference mixture

Reference mixture per category						
Environmental category	Abbreviation	Unit	A1	A2	A3	Total
Abiotic depletion, non fuel	ADnf	kg Sb eq	3,28E-05	2,15E-04	7,56E-05	3,24E-04
Abiotic depletion, fuel	ADf	kg Sb eq	2,92E-01	1,42E-01	9,94E-03	4,44E-01
Global warming potential	GWP	kg CO ₂ eq	1,03E+02	2,29E+01	2,23E+00	1,28E+02
Ozone layer depletion	ODP	kg CFC-11 eq	2,29E-06	3,44E-06	2,39E-07	5,97E-06
Photochemical oxidation potential	POCP	kg C ₂ H ₄ eq	2,84E-02	1,37E-02	2,57E-03	4,47E-02
Acidification potential	AP	kg SO ₂ eq	2,64E-01	1,63E-01	2,73E-02	4,54E-01
Eutrophication potential	EP	kg PO ₄ eq	5,19E-02	3,64E-02	5,59E-03	9,38E-02
Human toxicity potential	HTTP	kg 1,4-DB eq	1,02E+01	5,74E+00	1,73E+00	1,77E+01
Ecotoxicity potential, fresh water	FAWTP	kg 1,4-DB eq	2,32E-01	1,44E-01	4,01E-02	4,16E-01
Ecotoxicity potential, marine water	MAETP	kg 1,4-DB eq	1,02E+03	4,58E+02	1,16E+02	1,59E+03
Ecotoxicity potential, terrestrial environment	TAETP	kg 1,4-DB eq	2,16E-01	2,65E-02	1,08E-01	3,50E-01
Milieukosten indicator	MKI	€	€ 7,81	€ 2,74	€ 0,45	€ 11,00

Table 7.2 shows the impact of each stage in the LCA per environmental impact category per cubic meter of mix. The total influence of categories A1-A3 combined is shown as well. Table 7.2 allows to see what step in the process has most influence on the MKI.

Results and interpretation

The total MKI of the cradle-to-gate methodology for 1 m³ of reference concrete is € 11,00. From the raw materials (A1) the cement has the most influence on the MKI. However, during transportation towards the production location (A2) both the concrete sand and blast furnace slag have significant contribution, mainly due to large distances and large amounts of material. Of all processes, the green energy contributes the most (A3). Also interesting is that the production of 1 m³ of reference concrete emits 128 kg of CO₂.

The above combined proves that cement is the main contributor to the MKI. Large transportation distances combined with large amount of material also translate to quite large contributions to the total MKI as shown by the concrete sand and blast furnace slag. Lastly, the green energy contributes most to the MKI in terms of process steps. Of all green energy used, most is used by the climate chamber. Lowering the temperature in the climate chamber is therefore an option to lower the MKI as well as either a more energy efficient cement production or using less cement (the latter is in a lot of cement concrete cases restricted due to the minimum cement content stated per climate class in the concrete norms).

Sensitivity analysis

The influence of the most important choices and assumptions made on the final results can be determined using a sensitivity analysis. For the SBK (Stichting Bouwkwiteit) some items are important, of which the results can be found in Table 7.3 below and on the next page.

Table 7.3 - Sensitivity analysis reference mix

Subject of sensitivity analysis	Reasoning
Spread at group-averages	SBK requirement; no sensitivity analysis needed as the concrete is produced at 1 location.
Geographical spread	SBK requirement; no sensitivity analysis needed as the concrete is produced at 1 location.

Technological spread	SBK requirement; no sensitivity analysis needed as the concrete is produced at 1 location.
Spread in composition	SBK requirement; no sensitivity analysis needed as the concrete is produced at 1 location.
Waste scenario's	Not relevant in this research.
Influence of allocation	Performed conform SBK, no sensitivity analysis deemed necessary.
Total conclusion and additional comparisons between the three mix designs is provided in section 7.4.	

7.2. LCA alkali activated low MKI concrete

Full product description

The alkali activated low MKI mix from TU Delft has the goal to be used in the production process of the industry. The final concrete aims to have equal performance but lower MKI. However, some assumptions should be made with regards to input parameters for the calculation model:

- The Sodium silicate and Sodium hydroxide are assumed to be transported by truck with empty retour trip over a distance of 80 km.
- The GGBFS is assumed to be transported by inland vessel from EcoCEM (located between Dordrecht and Breda), which means a distance of 80 km is used.
- The CC is assumed to be transported from a generic location in Belgium by inland vessel. A transportation distance of 250 km is used.
- The climate chamber can be lowered quite considerably in temperature for alkali activated concrete. It is assumed that total energy consumption (11.63 kWh) consists of 50 % climate chamber and 50 % other equipment. The energy reduction of the climate chamber is estimated to be 50 %, meaning that a total green energy consumption of 8.71 kWh is used.

The mix design that is calculated is shown in Table 7.4 below. The full table can be found in appendix F.

Table 7.4 - Mix design and transportation distances

Component	Amount (kg/m ³)	Transport type	Distance (km)
GGBFS	CONFIDENTIAL	Inland vessel	80
SS solution		Truck	80
SH solution		Truck	80
Water		-	-
Blast furnace slag 2-8mm		Inland vessel	252
Concrete sand 0-4mm		Inland vessel	165
Copperslag 0-4mm		Inland vessel	164
Concrete recycled sand 0-10mm		Inland vessel	60
Total		-	-

Quality of used data

The quality of used data can be found in appendix I.

Life cycle inventory

Figure 7.2 shows the process tree. A larger image is available in Appendix I.

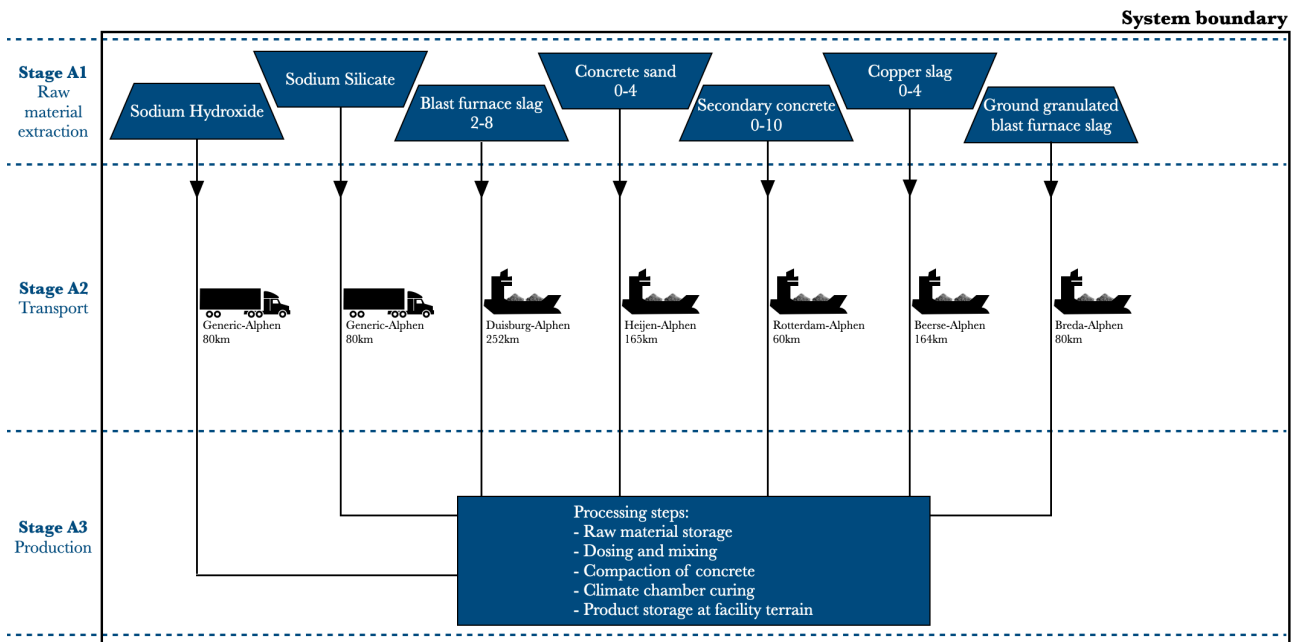


Figure 7.2 - Process tree low MKI concrete

Elaboration on the process tree

Sodium hydroxide

Sodium hydroxide is part of the activator solution of this concrete. It is assumed to be delivered by truck over a distance of 80 km, from a generic location in the Netherlands. Data used in the calculation model originates from Ontwerptool Groen Beton (20 Natronloog (50 %): (Sodium hydroxide, without water, in 50 % solution state {GLO}|market for|cut-off, u, tap water {GLO}|market group for| cut-off)).

Sodium silicate

Sodium silicate is part of the activator solution of this concrete. It is assumed to be delivered by truck over a distance of 80km, from a generic location in the Netherlands. Data used in the calculation model originates from Ontwerptool Groen Beton (20 Waterglas, hydrothermaal (48 %) (Sodium silicate, without water, in 48 % solution state {RER}|Sodium silicate production, hydrothermal liquor, product in 48 % solution state |cut-off + tap water {RER}|market group for | cut-off, u)). However, the data is translated towards the exact sodium silicate used in this research by adding sodium hydroxide until the combination equals the modulus of the sodium silicate that is used in this research.

Blast furnace slag

This blast furnace slag is used as surcharge material and not as binding component, meaning that environmental data may be put to 0 (free of burden) as it is considered a by-product from the steel industry. However, transportation does introduce emissions and is accounted for. The blast furnace slag is transported by inland vessel from Duisburg, Germany over a distance of 252 km.

Concrete sand

The sand is supplied by Teunesen Zand en Grint B.V., a member of Cascade. Therefore, environmental data from “*Industriezand, in en nabij Nederland geproduceerd door Cascade leden, c2*” from the Nationale Milieu Database is used. The sand is transported by inland vessel from Heijen over a distance of 165 km.

Secondary concrete

This is concrete that has been in a construction before, but broken down and reused in fresh concrete and therefore originates from construction and demolition waste. Recycling aggregates are end-of-waste, meaning that it is considered free of burden (0 values for all impact categories). Transportation takes place by inland vessel from Rotterdam with a distance of 60 km.

Copper slag

Like blast furnace slag, copper slag is a by-product from the steel industry. It is used as surcharge material and not part of the binder, meaning it may be considered free of burden (0 values for all impact categories). The copper slag is transported by inland vessel from Metallo, Beerse over a distance of 164 km.

Ground granulated blast furnace slag

Ground granulated blast furnace slag forms the binder when combined with the water and the two activators and is therefore a vital component in the mix design. It is assumed to be transported by inland vessel from EcoCEM towards industry, over a distance of 80 km. Data used for calculations originate from Ontwerptool Groen Beton (eco2cem, Ecocem, Moerdijk, c1 - NMDv3.4).

Water

Water is locally used.

Production of the concrete

The raw materials are all stored at the production facility. The ground granulated blast furnace slag can be stored in silo's. The two activators can either be stored separately in containers and then be combined with water until ready for production or are stored in a large basin which gradually adds the activator components and keeps it moving. The aggregates are stored in non-covered bunkers exposed to outdoor air. Products are weighed and mixed together into a dry cast mixture. This mixture is transported to a mold which is pressed and vibrated to form the final concrete product. This product remains in a climate chamber for 24 hours and are then stored for a minimum of two weeks at the outdoor terrain, exposed to the elements.

During production, a combination of electricity and diesel is used. Per cubic meter of concrete, the following energy is used:

1. Green energy, 8.71 kWh (modeled after NMDv3.0, 53 % wood and biogas, 45 % wind)
2. Diesel, 0.27 l (modeled after Diesel, burned in building machine {GLO}|market for|cut-off)

Total production waste is determined to be 1.2 % and is accounted for in the calculations of the total environmental impact. This waste is deposited as sludge. The design life span for the concrete is 100 years but is not accounted for as only categories A1-A3 (production) are considered.

Life cycle impact analysis

The life cycle impact analysis is shown in Table 7.5 on the next page.

Table 7.5 - Life cycle impact analysis of low MKI mixture

Low MKI mixture per category						
Environmental category	Abbreviation	Unit	A1	A2	A3	Total
Abiotic depletion, non fuel	ADnf	kg Sb eq	3,49E-04	1,57E-04	5,70E-05	5,63E-04
Abiotic depletion, fuel	ADf	kg Sb eq	1,90E-01	1,12E-01	8,90E-03	3,10E-01
Global warming potential	GWP	kg CO2 eq	2,77E+01	1,81E+01	1,89E+00	4,78E+01
Ozone layer depletion	ODP	kg CFC-11 eq	8,70E-06	2,70E-06	2,17E-07	1,16E-05
Photochemical oxidation potential	POCP	kg C2H4 eq	1,04E-02	1,08E-02	2,15E-03	2,34E-02
Acidification potential	AP	kg SO2 eq	1,15E-01	1,31E-01	2,21E-02	2,68E-01
Eutrophication potential	EP	kg PO4 eq	1,59E-02	2,94E-02	4,56E-03	4,99E-02
Human toxicity potential	HTTP	kg 1,4-DB eq	1,12E+01	4,41E+00	1,38E+00	1,69E+01
Ecotoxicity potential, fresh water	FAWTP	kg 1,4-DB eq	2,10E-01	1,09E-01	3,12E-02	3,51E-01
Ecotoxicity potential, marine water	MAETP	kg 1,4-DB eq	1,21E+03	3,43E+02	9,08E+01	1,64E+03
Ecotoxicity potential, terrestrial environment	TAETP	kg 1,4-DB eq	1,02E-01	2,07E-02	8,10E-02	2,04E-01
Milieukosten indicator	MKI	€	€ 3,18	€ 2,17	€ 0,37	€ 5,72

Table 7.5 shows the impact of each stage in the LCA per environmental impact category per cubic meter of mix. The total influence of categories A1-A3 combined is shown as well. Table 7.5 allows to see what step in the process has most influence on the MKI.

Results and interpretation

The total MKI of the cradle-to-gate methodology for 1 m³ of alkali activated low MKI concrete is € 5,72. From the raw materials (A1) it is clear that the ground granulated blast furnace slag has the most influence on the MKI. However, during transportation towards the production location (A2) both the concrete sand and blast furnace slag have significant contribution, mainly due to large distances and large amounts of material. Of all processes, the green energy still contributes the most (A3). Also interesting is that the production of 1 m³ of alkali activated low MKI concrete emits 48 kg of CO₂.

Even though large transportation distances combined with large amount of material also translate to quite large contributions to the total MKI as shown by the concrete sand and blast furnace slag. Lastly, the green energy contributes most to the MKI in terms of process steps. Of all green energy used, most is used by the climate chamber. Lowering the temperature in the climate chamber is therefore an option to lower the MKI.

Sensitivity analysis

The influence of the most important choices and assumptions made on the final results can be determined using a sensitivity analysis. For the SBK (Stichting Bouwkwiteit) some items are important, of which the results can be found in Table 7.6 below and on the next page.

Table 7.6 - Sensitivity analysis low MKI mix

Subject of sensitivity analysis	Reasoning
Spread at group-averages	SBK requirement; no sensitivity analysis needed as the concrete is produced at 1 location.
Geographical spread	SBK requirement; no sensitivity analysis needed as the concrete is produced at 1 location.
Technological spread	SBK requirement; no sensitivity analysis needed as the concrete is produced at 1 location.
Spread in composition	SBK requirement; no sensitivity analysis needed as the concrete is produced at 1 location.

Waste scenario's	Not relevant in this research.
Influence of allocation	Performed conform SBK, no sensitivity analysis deemed necessary.

Total conclusion and additional comparisons between the three mix designs is provided in section 7.4.

7.3. LCA alkali activated performance concrete

Full product description

The alkali activated performance mix from TU Delft has the goal to be used in the production process of the industry. The final concrete aims to have equal performance but lower MKI. However, some assumptions should be made with regards to input parameters for the calculation model:

- The Sodium silicate and Sodium hydroxide are assumed to be transported by truck with empty retour trip over a distance of 80 km.
- The GGBFS is assumed to be transported by inland vessel from EcoCEM (located between Dordrecht and Breda), which means a distance of 80 km is used.
- The CC is assumed to be transported from a generic location in Belgium by inland vessel. A transportation distance of 250 km is used. Several sources of information with regards to EPD's of calcined clay have been found of which 1, originating from NIBE (which uses Ecoinvent v3.6 data), was fully complete for this LCA. It combines data from 1.16 kg kaolin and 2.5 MJ of energy for blast furnaces as input for production of 1 kg calcined clay.
- The climate chamber can be lowered quite considerably in temperature for alkali activated concrete. It is assumed that total energy consumption (11.63 kWh) consists of 50 % climate chamber and 50 % other equipment. The energy reduction of the climate chamber is estimated to be 50 %, meaning that a total green energy consumption of 8.71 kWh is used.

The mix design that is calculated is shown in Table 7.7 below. The mix design that is calculated is shown in Table 7.4 below. The full table can be found in appendix F.

Table 7.7 - Mix design and transportation distances

Component	Amount (kg/m ³)	Transport type	Distance (km)
GGBFS	CONFIDENTIAL	Inland vessel	80
CC		Inland vessel	250
SS solution		Truck	80
SH solution		Truck	80
Water		-	-
Blast furnace slag 2-8mm		Inland vessel	252
Concrete sand 0-4mm		Inland vessel	165
Copperslag 0-4mm		Inland vessel	164
Concrete recycled sand 0-10mm		Inland vessel	60
Total		-	-

Quality of used data

The quality of used data can be found in appendix I.

Life cycle inventory

Figure 7.3 shows the process tree. A larger image is available in Appendix I.

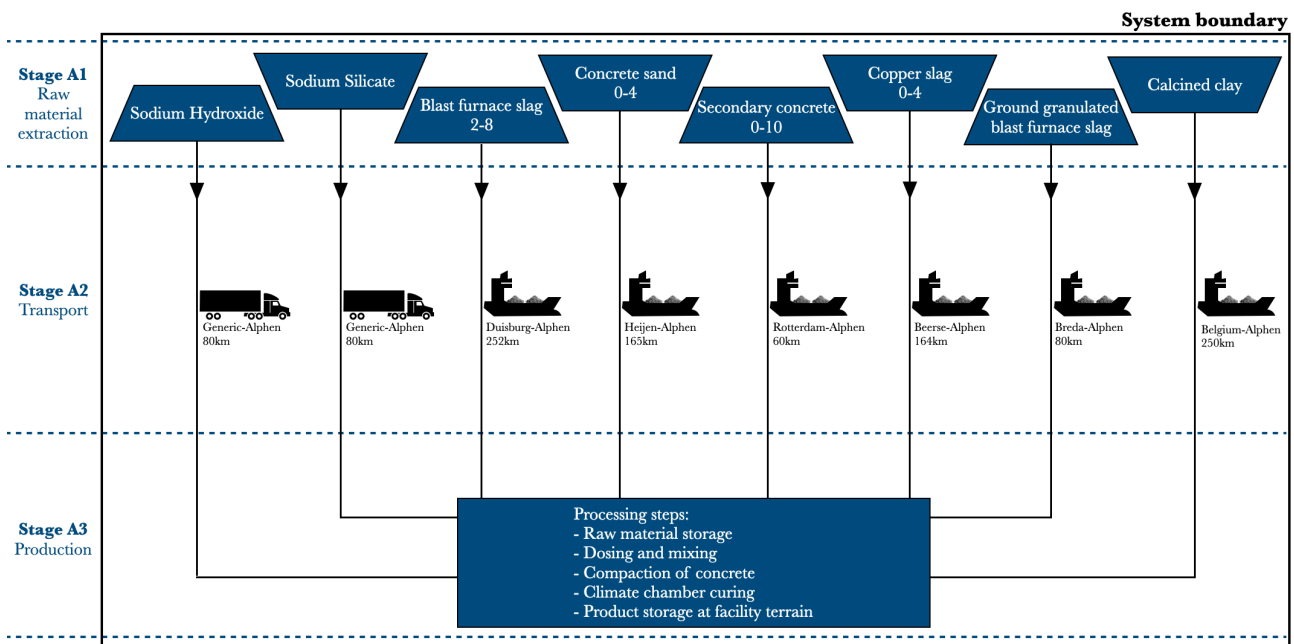


Figure 7.3 - Process tree alkali activated performance concrete

Elaboration on the process tree

Sodium hydroxide

Sodium hydroxide is part of the activator solution of this concrete. It is assumed to be delivered by truck over a distance of 80 km, from a generic location in the Netherlands. Data used in the calculation model originates from Ontwerptool Groen Beton (20 Natronloog (50 %): (Sodium hydroxide, without water, in 50 % solution state {GLO}|market for|cut-off, u, tap water {GLO}|market group for| cut-off)).

Sodium silicate

Sodium silicate is part of the activator solution of this concrete. It is assumed to be delivered by truck over a distance of 80 km, from a generic location in the Netherlands. Data used in the calculation model originates from Ontwerptool Groen Beton (20 Waterglas, hydrothermaal (48 %) (Sodium silicate, without water, in 48 % solution state {RER}|Sodium silicate production, hydrothermal liquor, product in 48 % solution state |cut-off + tap water {RER}|market group for | cut-off, u)). However, the data is translated towards the exact sodium silicate used in this research by adding sodium hydroxide until the combination equals the modulus of the sodium silicate that is used in this research.

Blast furnace slag

This blast furnace slag is used as surcharge material and not as binding component, meaning that environmental data may be put to 0 (free of burden) as it is considered a by-product from the steel industry. However, transportation does introduce emissions and is accounted for. The blast furnace slag is transported by inland vessel from Duisburg, Germany over a distance of 252 km.

Concrete sand

The sand is supplied by Teunessen Zand en Grint B.V., a member of Cascade. Therefore, environmental data from “*Industriezand, in en nabij Nederland geproduceerd door Cascade leden, c2*” from the Nationale Milieu Database is used. The sand is transported by inland vessel from Heijen over a distance of 165 km.

Secondary concrete

This is concrete that has been in a construction before, but broken down and reused in fresh concrete and therefore originates from construction and demolition waste. Recycling aggregates are end-of-waste, meaning that it is considered free of burden (0 values for all impact categories). Transportation takes place by inland vessel from Rotterdam with a distance of 60 km.

Copper slag

Like blast furnace slag, copper slag is a by-product from the steel industry. It is used as surcharge material and not part of the binder, meaning it may be considered free of burden (0 values for all impact categories). The copper slag is transported by inland vessel from Metallo, Beerse over a distance of 164 km.

Ground granulated blast furnace slag

Ground granulated blast furnace slag forms the binder when combined with the water and the two activators and is therefore a vital component in the mix design. It is assumed to be transported by inland vessel from EcoCEM towards industry, over a distance of 80 km. Data used for calculations originate from Ontwerptool Groen Beton (eco2cem, Ecocem, Moerdijk, c1 - NMDv3.4).

Calcined clay

The calcined clay originates from Belgium from a generic location and is one of the binder components like the ground granulated blast furnace slag. It is assumed to be transported by inland vessel from a generic location in Belgium towards industry, over a distance of 250 km. Input of 1.16 kg kaolin and 2.5 MJ heating energy is used for production of 1 kg calcined clay. Inputs are obtained from NIBE, which states the data origins from EcoInvent v3.6 as: Kaolin {RER}|production | Cut-off, U and Heat, district or industrial, natural gas {Europe without Switzerland}|heat production, natural gas, at industrial furnace >100kW | Cut-off, U.

Water

Water is locally used.

Production of the concrete

The raw materials are all stored at the production facility. The ground granulated blast furnace slag and calcined clay can be stored in silo's. The two activators can either be stored separately in containers and then be combined with water until ready for production or are stored in a large basin which gradually adds the activator components and keeps it moving. The aggregates are stored in non-covered bunkers exposed to outdoor air. Products are weighed and mixed together into a dry cast mixture. This mixture is transported to a mold which is pressed and vibrated to form the final concrete product. This product remains in a climate chamber for 24 hours and are then stored for a minimum of two weeks at the outdoor terrain, exposed to the elements.

During production, a combination of electricity and diesel is used. Per cubic meter of concrete, the following energy is used:

1. Green energy, 8.71 kWh (modeled after NMDv3.0, 53 % wood and biogas, 45 % wind)
2. Diesel, 0.27 l (modeled after Diesel, burned in building machine {GLO}|market for|cut-off)

Life cycle impact analysis

The life cycle impact analysis is shown in Table 7.8 below.

Table 7.8 - Life cycle impact analysis of performance mixture

Performance mixture per category						
Environmental category	Abbreviation	Unit	A1	A2	A3	Total
Abiotic depletion, non fuel	ADnf	kg Sb eq	4,75E-04	1,57E-04	5,70E-05	6,89E-04
Abiotic depletion, fuel	ADf	kg Sb eq	5,00E-01	1,11E-01	8,90E-03	6,20E-01
Global warming potential	GWP	kg CO ₂ eq	6,34E+01	1,80E+01	1,89E+00	8,33E+01
Ozone layer depletion	ODP	kg CFC-11 eq	1,52E-05	2,68E-06	2,17E-07	1,81E-05
Photochemical oxidation potential	POCP	kg C ₂ H ₄ eq	1,55E-02	1,07E-02	2,15E-03	2,83E-02
Acidification potential	AP	kg SO ₂ eq	1,48E-01	1,30E-01	2,21E-02	2,99E-01
Eutrophication potential	EP	kg PO ₄ eq	1,98E-02	2,91E-02	4,56E-03	5,35E-02
Human toxicity potential	HTTP	kg 1,4-DB eq	1,40E+01	4,39E+00	1,38E+00	1,97E+01
Ecotoxicity potential, fresh water	FAWTP	kg 1,4-DB eq	2,42E-01	1,09E-01	3,12E-02	3,82E-01
Ecotoxicity potential, marine water	MAETP	kg 1,4-DB eq	1,29E+03	3,42E+02	9,08E+01	1,72E+03
Ecotoxicity potential, terrestrial environment	TAETP	kg 1,4-DB eq	1,07E-01	2,06E-02	8,10E-02	2,08E-01
Milieukosten indicator	MKI	€	€ 5,45	€ 2,15	€ 0,37	€ 7,97

Table 7.8 shows the impact of each stage in the LCA per environmental impact category per cubic meter of mix. The total influence of categories A1-A3 combined is shown as well. Table 7.8 allows to see what step in the process has most influence on the MKI.

Results and interpretation

The total MKI of the cradle-to-gate methodology for 1 m³ of reference concrete is € 7,97. From the raw materials (A1) it is clear that the calcined clay has the most influence on the MKI. However, during transportation towards industry (A2) both the concrete sand and blast furnace slag have significant contribution, mainly due to large distances and large amounts of material. Of all processes, the green energy contributes the most (A3). Also interesting is that the production of 1 m³ of alkali activated performance concrete emits 83 kg of CO₂.

The above combined proves that calcined clay is the main contributor to the MKI. Large transportation distances combined with large amount of material also translate to quite large contributions to the total MKI as shown by the concrete sand and blast furnace slag. Lastly, the green energy contributes most to the MKI in terms of process steps. Of all green energy used, most is used by the climate chamber even though lower temperature is used as input.

Sensitivity analysis

The influence of the most important choices and assumptions made on the final results can be determined using a sensitivity analysis. For the SBK (Stichting Bouwkwiteit) some items are important, of which the results can be found in Table 7.9 below and on the next page.

Table 7.9 - Sensitivity analysis performance mix

Subject of sensitivity analysis	Reasoning
Spread at group-averages	SBK requirement; no sensitivity analysis needed as the concrete is produced at 1 location.
Geographical spread	SBK requirement; no sensitivity analysis needed as the concrete is produced at 1 location.
Technological spread	SBK requirement; no sensitivity analysis needed as the concrete is produced at 1 location.

Spread in composition	SBK requirement; no sensitivity analysis needed as the concrete is produced at 1 location.
Waste scenario's	Not relevant in this research.
Influence of allocation	Performed conform SBK, no sensitivity analysis deemed necessary.

7.4. LCA comparison and conclusion

It is very interesting to see how much the total binder contributes to the total MKI value. For the reference mixture, the CEM III/B has the largest contribution towards the total MKI value. The MKI value of the binder component of the alkali activated concrete mixtures is a lot lower due to the combination of alternative production methods and overall lower transportation distances of the materials. However, calcined clay is still a material in development and even though the environmental impact is lower when compared to cement, if it were used in equal quantities the difference would be negligible (and for alkali activated concrete activator would also need to be added, thus a calcined clay only binder results in higher overall MKI). Table 7.10 below illustrates the reduction in CO₂ and MKI for the alkali activated mix designs compared to the reference mix design.

Table 7.10 - CO₂ and MKI reduction

Mix design name	MKI value per m ³ concrete	Reduction compared to reference	CO ₂ emission per m ³ concrete in kg	Reduction compared to reference
Reference	€ 11,00	-	128	-
Low MKI	€ 5,72	-48%	48	-63%
Performance	€ 7,97	-28%	83	-35%

From the information from Table 7.10, it can clearly be concluded that both alkali activated concrete mix designs succeed in lowering the MKI, but also CO₂ emissions when compared to the reference mix design which contains blast furnace slag cement. The low MKI mix design has a 48 % reduction in MKI whilst lowering CO₂ emissions by 63 % which is significant. The performance mix design is able to reduce the MKI by 28 % whilst lowering the CO₂ emissions by 35 %.

It is clear that these are very large reductions compared to the reference concrete therefore further increasing the potential of alkali activated concrete to be applied in concrete revetment products. However, it is very important that the following aspects are considered to confidently use these results:

- The alkali activated concrete should have at least equal lifespan. In a complete LCA, considering all life stages, lifespan of the material is considered. If the lifespan of the alkali activated concrete is half of its cement counterpart, the profit in environmental footprint evaporates as the concrete is to be produced twice in the same period of time. Therefore, lifespan should be validated.
- Recyclability is considered in the complete LCA. The level in which alkali activated concrete is recyclable should be investigated prior to executing a complete LCA. As of right now, alkali activated concrete does not seem to have any ecological benefits or significant drawbacks for recyclability when compared to cement concrete, as it can be crushed and re-used in other application in the civil engineering field. Researches conclude that there might be possibilities of application of recycled alkaline activated concrete in structural applications, but that there are

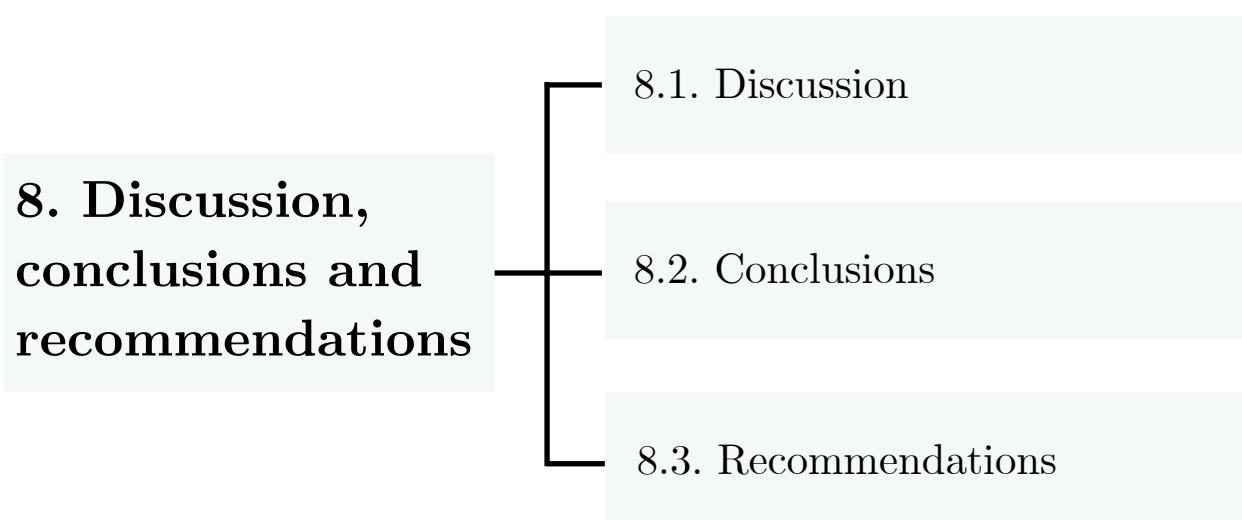
limitations like with recycled cement concrete (e.g. high water absorption, high Los Angeles abrasion) but that strength characteristics are promising (Chaliasou et al., 2018), (Akbarnezhad et al., 2015). Concluding the above: the end-of-life situation of both alkali activated concrete and cement concrete is equal, with the assurance that both concretes also meet the environmental threshold values (e.g. amount of heavy metals in the concrete) (Provis & van Deventer, 2009).

- If the cement production plants are to reduce their emissions, it is very well possible that the environmental impact of cement is lowered, making it more competitive to the ingredients used for alkali activated concrete.
- The MKI input values for GGBFS are based on the value of steel versus the value of GGBFS. If GGBFS is utilized even more in the industry, the increase of value can induce large changes to the environmental emissions allocated to GGBFS in the steel industry, resulting in larger environmental footprint for concrete products that contain GGBFS. This affects both alkali activated concrete and blast furnace slag cement concrete, as both contain GGBFS. This increases both of their environmental footprint, but this increase does not occur for one of the most polluting cement types: Portland cement.

The final aspect that is to be considered is not the environmental impact on product level, but environmental impact on system level. On product level, the incorporation of alkali activated concrete proves to lower the environmental footprint, but what happens when all industries would transition to alkali activated concrete? Replacing cement by GGBFS and CC does lower overall environmental footprint as the environmental footprint of GGBFS is much lower due to its partial allocation to steel production and CC is calcined at half the temperature of cement production, yielding lower emissions. However, such a transition requires that there is sufficient GGBFS available, that its value does not drastically increase (this is uncertain, as higher demand from the concrete industry logically results in higher value, therefore the allocation of environmental footprint of the entire steel industry transitions more to GGBFS and less to steel) and finally, that all GGBFS remains locally available. This also requires the cement industries to not improve on their production process, as that potentially lowers the environmental footprint of cement. These are uncertain factors and dependent on development of lower-emission production facilities for both the steel and cement industries.

Therefore the alkaline activated concrete does lower environmental footprint on product level, but on system level it is strongly dependent on development of lower emission industries which is required for improvements on system level.

Discussion, conclusions and recommendations



This chapter concludes this research. Section 8.1 contains the discussion of the results which is followed by section 8.2, which provides the conclusions. Finally, section 8.3 contains recommendations for future research.

8.1. Discussion

The research results indicate that there is the possibility to utilize alkali activated concrete in concrete revetment products using a combination of the upscaling methodology and Taguchi method. This section discusses the added value of the used methodologies in section 8.1.1. Section 8.1.2. connects the research results towards literature and section 8.1.3. provides a description of the limitations of this research.

8.1.1. Added value of methodology

This research uses two different methodologies, namely a research methodology and a mix design methodology. The added value of each of these methodologies is described separately.

Research methodology - upscaling

Upscaling proves to be an excellent methodology for the development of mature concrete mix designs. By working from small scale towards large scale, differences between each scale are identified more efficiently. When testing freeze-thaw performance at both mortar and concrete level, significant changes in the results are visible. The mortar has great performance, while the upscaled concrete does not always have great performance. This difference is attributed to the inclusion of different aggregates, compaction and scaling effects. If the research would start with concrete mix designs immediately, the different mechanisms involved in upscaling are more complicated to find and explain.

Finding the right correlations between research results and literature is more challenging without an upscaling methodology. Upscaling provides better overview with determination of the right deterioration mechanisms and therefore proves to be a valuable methodology in this research.

Mix design methodology - Taguchi method

The Taguchi method proves to be a great methodology for development of a mix design that needs to fulfill many requirements. It enables to research the effects of different mix design parameters without the need to cast a lot of mix designs. By using a broad range of each mix design parameter, the Taguchi method enables a high success rate for finding the right mix design for the application.

Furthermore, Taguchi method also provides valuable information with regards to the effects of each mix design parameter coupled to performance characteristics. With these so called trends, the optimal mix design direction can be determined. In this research, the final used mix designs are the result of both the upscaling methodology (keep the mix designs equal, but only scale them up) and the Taguchi method. The research results show that Taguchi trends correlate with the mix design parameters of the final concrete mix designs, proving that the trends indeed provide a direction for the optimal mix design composition.

Overall, the methodologies combine well. Upscaling allows for efficient gathering of information with regards to finding reasons why a mix design performs the way it does. In combination with Taguchi, a lot of material is saved in finding the right composition of the concrete and with using Taguchi the effects of each mix design parameter are clearly visualized to provide valuable information with regards to optimal mix design compositions for the application in mind. Based on the above it is clear that if a research like this is repeated, results will be the same. This implies that the research results and used methodologies are valid.

8.1.2. Relation of research results and literature

The relation of the research results and literature is in this case difficult, as no traceable research has been found which focuses on mix design development of alkali activated concrete revetment products or alkali activated earth moist concrete. However, some relations are found for some performance characteristics.

Several mechanisms influence the freeze-thaw performance of alkaline activated concrete. The specimens are not optimally compacted with the small compaction machine from the industry, leading to a porous top half of the specimens. From freeze-thaw visualizations it is clear that the top half of some specimens degrades more than the bottom half, which is better compacted. This is also observed by Fu et al. (2011) where it is concluded that a less permeable shell results in better freeze-thaw performance, but this is only true if there are sufficient voids to relieve the internal stresses caused by crystallization.

Increase of permeability is further enhanced by recycled aggregates, which are used in the concrete specimens. This increases permeability and further lowers freeze-thaw performance. Besides that, recycled aggregates can enhance shrinkage of specimens where shrinkage is not proven in this case, but very much considered as it is widely reported for alkaline activated slag concrete. The decrease in freeze-thaw performance due to shrinkage is reported by Nanayakkara et al. (2020) as well as the increase in shrinkage due to inclusion of recycled aggregates.

Shrinkage and formation of microcracking is highlighted by Collins & Sanjayan (1998) where it is concluded that this causes reduction in freeze-thaw performance of specimens.

The study by Urgulu et al. mentions that the inclusion of recycled aggregates results in worse freeze-thaw performance and that increase of the modulus also reduces freeze-thaw performance. Alanazi et al. also concludes that increase of modulus results in worse freeze-thaw performance and the same trend is observable in this research, even with different modulus values than the other researches.

Both Alanazi et al. and Barges et al. conclude that increase of metakaolin, which is quite similar to calcined clay, reduces freeze-thaw performance whilst increasing porosity in the specimens due to more difficult compaction due to the stickiness of the material. The same is true for this research as clearly proven by the Taguchi trend figures.

Finally Puertas et al. has observations that increase of sodium hydroxide (thus decrease in modulus) results in more ettringite to be formed on the specimens. Ettringite is spotted on nearly every specimen, but does not have a direct link to the findings by Puertas et al.

8.1.3. Limitations of this research

This research has the following limitations:

- Taguchi method has a wide range of mix design parameters, but the range can be further extended to see if there is other possible optimal mix design trends observable. This is realized by repeating the research with identical experiments, but either a different Taguchi array or by expanding the Taguchi from L9 array (4 parameters, 3 levels and 9 mix designs) to a L16 array (5 parameters, 4 levels and 16 mix designs).
- All upscaling phases are executed under curing in a very high humidity (> 95 %) curing room, where leaching is observed and confirmed for paste phase specimens. Still, all specimens have not been covered in foil to prevent leaching. Either a lower humidity curing room (not available) or foil-wrapped specimens should be used throughout the research to maximize the performance potential of the mix designs.
- Sulfate attack at mortar level results in no observable degradation of the specimens except for minor formation of ettringite which indicates the very beginning of degradation. This short testing period is therefore considered a limitation.

- Freeze-thaw at mortar level is tested with an adaption to the norm. After sawing the specimens, they are submerged in potable water for several days and then put in the testing setup with de-icing solution. Two limitations are found: A period of drying before pre-saturation should be included to observe (if any) possible drying shrinkage at mortar level and pre-saturation should be performed with de-icing solution instead of potable water.
- Freeze-thaw at mortar level used an adaption from the norm. Cubes are completely submerged in de-icing solution in a sealed bag. However, this decision had to be made as the availability of the machine was very limited (only 4 trays available) and 7 mix designs were tested. Testing mortar according to CDF test but with 40 x 40 x 160 mm prisms should provide more accurate results, but once again, the machine should be available better to do that.
- At concrete scale, the difference between the two possible aggregate types (TU and industry) should be included to the comparison to prove that recycled aggregates indeed worsen drying shrinkage of the specimens.
- Compaction at concrete scale is performed as well as possible with the small compaction machine provided by the industry. Upon visual inspection it is clear that this compaction still is not optimal for the concrete mix designs due to a lot of visible pores in the top half of each casted specimen. Adjusting frequency of vibration and increasing the amount of pressure can alleviate or solve this problem as it forms a limitation to accurately represent the produced concrete in the factory process.
- With the life cycle analysis, only the product scale has been considered. To accurately determine the full environmental impact of alkali activated concrete, more experiments should be added to accurately determine the lifespan expectation of the material. Also, the end-of-life situation (recyclability) is a very important aspect which should be considered for a best possible comparison to regular cement concrete.
- With the life cycle analysis, only limited information is available for the calcined clay. While it is very likely that the input data is conservative, upon analysis it is clear that the environmental impact of calcined clay may be significant even with accurate data input. This might change the perspective of how much research should be performed on not-so-pure calcined clay as precursor in alkaline activated concrete.

8.2. Conclusions

This thesis focused on the following research question:

“To what extend can dry-cast slag and calcined clay-based alkali activated concrete be utilized in concrete revetment applications with equal performance in freeze-thaw and sulfate attack resistance and lower environmental impact compared to blast-furnace slag cement concrete?”

In order to answer this question, the challenges as stated in chapter 1 are repeated and evaluated. Combined with the performance results and life cycle analysis results, mentioned after the three challenges, the research question is answered.

Challenge 1: In order to lower the environmental impact of the developed concrete, the materials should be locally available.

Both the GGBFS and CC are locally available for the Dutch concrete market, but the extend of availability is questionable. The GGBFS market is nearly saturated and a significant amount is used for blast furnace cement. If a company that currently utilizes blast furnace cement transitions towards alkali activated concrete, the total availability scale does not change.

The research proves that while CC can be applied, the amount to which it can be applied is limited when combined with GGBFS. Based on the research results it is recommended to use no more than a value around 30 % of CC in a blended precursor mix.

Challenge 2: In what way can durability of alkaline activated concrete be improved in freeze-thaw performance and sulfate attack performance and what are deterioration mechanisms for alkali activated concrete subjected to freezing and thawing.

In terms of freeze-thaw performance, it is clear that introduction of calcined clay results in worse performance due to more difficult compaction and because of a more permeable shell, allowing the de-icing solution to penetrate more into the specimen resulting in more scaling. The effect of compaction on concrete scale is clearly proven, as non-optimal compaction results in many pores and a very permeable structure with significant scaling as a result. Scaling also increases with increasing W/B ratio and a lower modulus is also preferred as it allows for better relief of internal stresses caused by crystallization. Freeze-thaw performance of alkali activated concrete has potential on long term as it shows retarding scaling behavior.

Sulfate attack performance is determined to be sufficient as no severe degradation is observed between specimens that are and are not exposed to sulfate attack. However, the duration of sulfate attack exposure is limited and as slight formation of ettringite is found, a first sign of possible degradation. Still, the used MgSO_4 solution is high in concentration, so this still proves that alkali activated concrete is resistant to sulfate attack.

Challenge 3: Earth moist concrete is not used often for alkali activated concrete. To what extend is this a challenge for the specific application.

Confirmation that the mix designs are earth moist proves to result in some valuable observations. Alkali activated concrete responds differently to compaction when in earth moist state compared to cement concrete. While the alkali activated concrete mix designs both have a compaction value on the dry spectrum of the earth moist definition, the actual concrete is rather wet and also near bleeding condition. At the same time, the industry mix is near the wet spectrum of the earth moist definition while visually much drier than the alkali activated concrete mixtures. This indicates that the earth moist workability requirement for concrete does not necessarily apply for alkaline activated concrete.

Compaction is also a challenge for development of representative concrete specimens. The used compaction machine is the best option for this research, providing a combination of pressure

and vibration. Still, the mix designs have to be adjusted such that they are a little more moist. Without this increase in moisture, the mix designs can not be compacted due to crumbling. If the moisture addition is too high, the specimens sag as there is no mold after compaction like wet-cast concrete. In the end, the sweet spot that is found proves to still be insufficient as even the reference specimens do not perform as intended. Therefore, the specimens from the concrete phase are as well compacted within the scope of this research but on the smaller lab scale samples earth moist concrete can be a challenge for good comparison with and simulation of industrial scale products.

To further answer the research question, the performance requirements and LCA results are also concluded:

Performance - Compressive strength

Compressive strength development of the alkali activated concrete mix designs is sufficient. The 24 hour curing 12 MPa target and the final 28 day 37 MPa target is reached by both mix designs. Deviation within the specimens is on the high side, but sufficient for the amount of specimens. Most of the deviation is attributed towards the compaction method, as the top half of the specimens is not optimally compacted which is visually validated.

Performance - Freeze-thaw

While freeze-thaw performance in mortar phase proves to be very good, with levels way below scaling, it is not the case for the concrete scale specimens. The concrete specimens have quite significant scaling, but so does the reference concrete. The level of compaction is expected to be an important contributor to this performance, as the reference also does not perform as intended. One specimen of the C01 mix manages to outperform all other specimens, indicating that with some modifications and better compaction, freeze-thaw performance of alkali activated concrete can be sufficient enough for ensuring long service life in a concrete revetment, especially when combined with the retarding scaling behavior.

Performance - Sulfate attack

Sulfate attack is only tested at mortar level. At mortar level, during the testing period of 14 days, there is no observable damage to the specimens except for some starting formation of ettringite on the surface. As the same test on concrete level experiences the same testing duration, it is decided not to perform sulfate attack at concrete level. Still, the sulfate attack solution is high in concentration and is expected to be no problem for alkali activated concrete in a concrete revetment.

Performance - Water absorption

Water absorption of the concrete specimens is higher than the norm for revetment products allows, but so is water absorption of the reference mix design. The level of compaction is an important contributor to this performance, as there are a lot of visible pores on the top half of each specimen, which is the half that is not optimally compacted. Therefore, as the performance of alkali activated concrete is both above and below the reference concrete, it is expected that water absorption forms no problem on industrial scale after optimization of the mix design.

Life cycle analysis

It is clear that on product level, the alkali activated concrete is a better performer for the milieu kosten indicator and in terms of carbon dioxide emissions. A reduction of 48 % in terms of MKI and 63 % in terms of carbon dioxide is possible, but with some remarks: The concrete should still prove to have equal lifespan to regular cement concrete and should also be recyclable. While both factors are not tested, literature indicates that alkali activated concrete should be as recyclable as cement concrete.

Final conclusion

So to what extent can dry-cast slag and calcined clay-based alkali activated concrete be utilized in concrete revetment applications with equal performance in freeze-thaw and sulfate attack resistance and lower environmental impact compared to blast-furnace slag cement concrete?

From all research results it is clear that alkali activated concrete definitely proves to have great potential for concrete revetment application even though compaction during the concrete phase is not optimal.

The environmental impact of alkali activated concrete shows large reductions for both the MKI and CO₂ emissions over regular cement concrete with equal or better performance.

Calcined clay can be applied, but to limited extend. It is not a very pure calcined clay which is also clear in the Taguchi performance trends, therefore a substitution of no more than a value slightly higher than the medium percentage is recommended. Still, this can slightly alleviate the saturated ground granulated blast furnace slag market, but not in such a great manner that excess ground granulated blast furnace slag becomes available.

This research proves that alkali activated concrete is a very interesting contender to be used in concrete revetment products applications. There are still questions open for research to really validate it can be applied in revetments with full confidence and with the promise of long service life, but with the mix designs developed in this research the environmental impact can be reduced drastically while the alkali activated concrete mostly performs equal or better to its cement counterpart and with the additional benefit that the climate chamber temperature of industries that produce revetment products can be decreased, resulting in large energy savings. By utilizing the alkali activated concrete mix designs developed in this research, the concrete industry has great potential to lower its environmental footprint of concrete revetment products.

8.3. Recommendations

This research answers many questions with regards of application of alkali activated concrete in concrete revetment products. Still, there are recommendations for future research on this topic:

Recommendations - materials

- The influence of the amount of precursor is not considered. Lower precursor amount is preferable as this allows alleviation of the ground granulated blast furnace market. Therefore, a study which focuses on the effects of precursor amount on durability of alkaline activated concrete is recommended.
- Calcined clay is only partially able to replace ground granulated blast furnace slag in the precursor mix. Another possible contender is metakaolin. A research that focuses on its availability in Europe and application possibilities in a precursor mix is recommended, as alleviating the ground granulated blast furnace slag market is a necessity in the Netherlands if alkaline activated concrete is to be widely adopted.

Recommendations - mix designs

- In this research the Taguchi method is used for mix design development and proves to work very well for development of alkali activated concrete mix designs. Still, the range of the mix design parameters is limited in this research. Therefore a different Taguchi array or an expanded Taguchi array is recommended for future research, in combination with a more narrow range of experiments to be conducted such that the amount of work is not out of bounds. A L16 array (5 parameters, 4 levels and 16 mix designs) is a good contender, or another L9 array but with modified parameters. Still, a L16 array also allows for variation of precursor amount.
- Setting time is not tested in this research due to the amount of experiments conducted. Still this is strongly recommended for future research as the concrete should have a sufficient setting time in case of failures in the factory process. Otherwise, the concrete is hardening which results in large and unnecessary logistical operations and delays.

Recommendations - durability

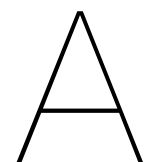
- Freeze-thaw performance can be expanded in several different directions to allow more detailed explanations of deterioration mechanisms.
 - First of all, results of freeze-thaw should be visualized more in terms of CT-scans for internal pore structure and with ESEM to observe the interaction between coarse aggregate and mortar. The latter is difficult, as the pretreatment method might introduce cracking, but still it is seen as good option. MIP can also be used to determine the pore size distribution as this might be a good indication of the room inside the concrete for the water to expand in.
 - Influence of SAP (super absorbent polymers) or AEA (air entraining agents) can be considered in a separate study as these might improve freeze-thaw performance. Furthermore, a research that focuses on development of an anti-shrinkage additive is valuable, as alkali activated concrete is reported to have quite a lot of shrinkage. This shrinkage can induce microstructure cracking and this also has effect on freeze-thaw performance.
 - Different freeze-thaw pre-treatment procedures should also be considered. It is logical to follow the concrete norm, but it can very well be true that alkali activated concrete needs a completely different pretreatment for proper performance in freezing and thawing as the underlying mechanisms are different from cement concrete. Curing time and method (50 % humidity, 100 % humidity, complete submersion in water, 20 or 30 degrees Celsius temperature etc.), pre-saturation drying and pre-saturation period can be varied to study the

effects on freeze-thaw performance. Also the differences between the testing methods (slab test, cube test and CDF test) are important to consider.

- Shrinkage should be considered more in general for alkali activated concrete, especially on higher performing concrete and concrete that includes more sodium silicate in the activator solution. Preventing or at least reducing shrinkage may solve or alleviate a lot of durability properties.
- Sulfate attack should be tested over a longer testing period as there was no real damage observed to the specimens in this research. Also, different sulfate attack solutions should be used, like sodium sulfate. A testing period on concrete scale of several months is recommended. Within this testing period, it is important to be able to couple the influence of deterioration to the service life of the concrete by considering the chemical composition of (in the case of revetment products) seawater.
- A testing program to confidently determine the service life of the alkali activated concrete via accelerated testing is important to mature the material towards industrial level. It is important that for each application, the service life of the alkali activated concrete is identical to its cement concrete counterparts. If the service life is not sufficient, the concrete needs to be replaced more often resulting in no environmental emission gains.
- The effects of incorporating recycled aggregates and also the recyclability of alkali activated concrete is very important, as this also has effects on environmental impact/footprint of the material.

Recommendations - LCA and cost

- The LCA calculations in this report only consider the product level of the materials. However, for full prove that alkali activated concrete has lower environmental product, all environmental impact modules should be included. To make a more detailed comparison with cement concrete, the lifespan of the material should be proven as well as the recyclability of the material. Great care should be taken in formation of the functional unit, as this has influence on how well and good the comparison is. Only comparing mix designs on compressive strength basis is not detailed enough, the full durability of the product should be considered in life cycle analysis for the best possible comparison to cement concrete.
- Pricing of the components used in the mix design is to be considered as this is a very important parameter in industrial application. If the mix designs perform very well in all aspects but the price of the materials is significantly higher, it is very difficult to apply the concrete in industry. Economical optimizations of mix designs is a very important step to further mature alkali activated concrete in applications.
- Availability of the activator components on the long term (> 10 years) is also very important and should be investigated if more industries want to transition towards alkali activated concrete.
- Development of industries that produce basic materials for concrete like cement, GGBFS, CC etc. is important. If it is certain that some industries can convert to production methods with much lower emissions, a shift in the preferred paste composition might occur. This potential of lower emission production should be investigated to further solidify LCA output.



Requirements for concrete revetment application

This appendix provides an overview of requirements that concrete should comply with when applied as revetment product. These requirements are used for the literature review to focus on these specific requirements thus increasing overall efficiency.

The requirements are structured in 3 categories:

1. Requirements from Dutch concrete revetment norms (NEN)
2. Requirements from the industry with regards to production
3. Requirements with regards to availability of materials

1. Requirements from Dutch concrete revetment norms

The norms describing requirements for concrete revetments have been obtained from a document describing the internal quality control from Kiwa, a company responsible for certification and quality control of products from industry. These norms specifically include what the concrete should comply with in order to enable application in the coastal environment. The norms are described in Table A.1 below. Note that these norms can describe the requirements for the product, so not always from the concrete itself.

Table A.1 - Norm overview

Norm	Description
NEN 7024-1+C1	Elements for block revetments - Part 1: General requirements
NEN 7024-2	Elements for block revetments - Part 2: Elements made of cement concrete, without interlocking and without reinforcement
NEN-EN 1338:2003	Concrete paving blocks - Requirements and test methods
NEN-EN 12390-3	Testing hardened concrete - Part 3: Compressive strength of test specimens

For each norm, an overview is provided on the next pages.

NEN-EN 7024-1+C1

General: The products created at industry are category I, meaning that they are applied in revetments as named in the law of water and other applications where specific requirements are stated related to stability under hydraulic loading. The list of requirements contains both product and concrete requirements. As this thesis focuses on concrete, only requirements with regards to concrete will be listed.

1. Structure

There may be no faults in structure. A minimum of 5 specimens are chosen to use as reference when structure has to be tested.

2. Texture

Texture between specimens should be nearly identical. A minimum of 5 specimens are chosen to use as reference when structure has to be tested.

3. Wet density

Has to be determined and should be within -2.2 % and +6.5 % of the declared nominal wet density. For use category I the additional requirement is that the mean minus 1.645 the standard deviation should at least contain the declared nominal value.

4. Freeze-thaw resistance

Resistance should be tested using appendix E of this norm. No block may be damaged and a mass loss ≤ 0.2 % is acceptable. No visible cracks may occur. This test is performed using the *full product* and not a specific concrete specimen.

NEN-EN 7024-2

General: This norm provides additional requirements a block revetment should comply with besides NEN-EN 7024-1.

1. Compressive strength

Compressive strength is normative, however, if the size of the element does not suffice, the splitting strength should be determined. Compressive strength should be at least of class C30/37 as defined in NEN-EN 206:2014, 4.3.1. Dimensions of the specimens are to be determined according to NEN-EN 12350-1.

2. Water absorption

Should be in compliance with class II of 5.3.2.2 of NEN-EN 1338:2003 and determined using 5.2.6.1 of this norm. 5.2.6.1 states that it should be determined using appendix E of NEN-EN 1338:2003.

3. Freeze-thaw resistance

Must comply with class III of 5.3.2.2 of NEN-EN 1338:2003 and is determined using 5.2.6.2 of this norm. 5.2.6.2 states it should be determined with appendix D of NEN-EN 1338:2003.

NEN-EN 1338:2003

General: This norm provides information on execution of certain requirements that are stated in NEN-EN 7024-2.

1. Appendix D, Freeze-thaw

Specimen subjected to 28 freeze-thaw cycles with 3% NaCl solution where mass loss per unit is described as the total mass after 28 cycles in kg divided by the area of the test surface. The CDF test is used, which describes mass loss should be lower than 1.5 kg/m².

2. Appendix E, Water absorption

Specimen soaked to constant mass and oven dried to constant mass. Loss in mass expressed as percentage of the mass of the dry specimen. Total water absorption should be ≤ 6 % by mass as a mean, where the block shall weigh less than 5 kg.

2. Production related requirements from industry

industry provided the additional requirements as stated in Table A.2 below.

Table A.2 - Additional requirements provided by industry

What	Requirement
Concrete strength class	C30/37 minimum
Desired strength after 24h curing	12 MPa
Environmental class	XF4
Consistency class	C1, earth moist
Desired density	min 2300 kg/m ³
Maximum aggregate size	10 mm

Curing room temperature/humidity/time	27-29 °C, humidity 85-92 %, time: 24h. Lower temperature possible.
Current ECI/CO ₂	< € 11,-/m ³ and < 128 kg CO ₂ /m ³

As can be seen, industry desires 12MPa strength after 24h curing at 27-29 °C with a humidity of 85-92 % as that corresponds with their current production process. This initial strength is necessary due to outside stacking the products on top of each other after 24h curing. Final strength should comply with at least C30/37 with a density of at least 2300 kg/m³. The density requirement is important, as a more dense concrete results in a more stable revetment product.

Another important requirement is the use of a maximum aggregate size of 10mm due to the size of the products, combined with a C1 consistency (earth moist concrete) to enable the dry-cast production process to function well. Finally, the product is situated in an environment consisting of salt water, de-icing salt probabilities and therefore environmental class XF4.

As for a reference, the current mixture has an environmental impact cost indicator of € 11,-/m³ and currently they emit approximately 128 kg CO₂/m³ concrete and the final alkali activated concrete should provide lower environmental cost indicator and lower CO₂ emissions.

3. Material availability requirements

Alkali activated concrete can consist of many materials, however not every material is available in the same proportion or composition. Each country will have its own preference in terms of suitable materials for alkali activated production.

Material availability has already shortly been addressed and therefore only a brief overview of the materials that are used for mix design are given below:

Precursors

- Ground granulated blast furnace slag (main component)
- Calcined clay (as partial replacement of ground granulated blast furnace slag)

Activators

- Sodium hydroxide
- Sodium silicate

Aggregates

- All aggregates are fine as long as size is < 10mm and particle size distribution should follow earth moist concrete designs to enable a proper mixture. Another option is to use exact aggregate composition as provided by the industry for the most apples-to-apples comparison in the concrete phase.

4. Overview of requirements

Table A.3 and A.4 below summarizes all requirements that should be met for the alkali activated concrete.

Table A.3 - Overview of material selection

Category	Selection type	Value
Materials	Maximum aggregate size	10 mm
	Precursor types	GGBFS & CC
	Activator types	Sodium hydroxide & sodium silicate

Table A.4 - Overview of requirements

Category	Requirement	Value
Production	Curing temperature	20 - 30 °C
	Curing duration	24h, then outside
	Curing humidity	80 - 95 %
	Consistency class (EN 12350-4)	C1 (earth moist concrete)
	Environmental class	XF4
	Density (For stability of products on dike)	> 2300 kg/m ³
	Compressive strength after 24h (Packaging of products after curing)	≥ 12 MPa
Performance	Compressive strength after 28d (NEN-EN 12390-3:2019(E))	≥ 37 MPa
	Freeze-thaw resistance (NVN-CEN/TS 12390-9:2016, CDF test)	Mass loss ≤ 1.5 kg/m ² after 28 FT cycles.
	Water absorption (NEN-EN1338:2003, appendix E)	Total water absorption ≤ 6 % by mass
	Environmental cost indicator	< € 11,-/m ³ and < 128kg CO ₂ /m ³

Alkali activated paste trials

The very first practical step in this thesis is the alkali activated paste trials. In the alkali activated paste trials, basic knowledge about working with equipment and the materials are the main learning objectives. Besides that, it also aims to determine some basic properties of preliminary mix designs to observe if the literature review results in proper mix designs.

This appendix describes the full procedure that has been followed during the paste trials and is structured as follows:

- Mix design of alkali activated paste trials
- Experiments performed during paste trials
- Results of paste trials
- Conclusion of paste trials

Mix design of alkali activated paste trials

The mix design of the alkali activated paste trials have been formed with a combination of literature review that focused on ground granulated blast furnace slag based alkali activated paste and recommendations of my supervisors. The goal within the alkali activated paste trials mix designs is to observe the influence of the sodium oxide content percentage relative to the dry binder. Expectations are that this parameter is directly linked to compressive strength performance, where a higher sodium oxide content is expected to result in higher compressive strength.

In total, three mix designs have been created. The following input parameters were kept at constant value:

- Water-to-binder ratio (W/B), 0.35.
- Modulus of water glass (Ms), 0.7
- Amount of granulated slag relative to calcined clay ($\text{GGBFS}/(\text{GGBFS}+\text{CC})$), 1

The sodium oxide content has been varied in such a way that three mix designs are obtained. The sodium oxide content was put at 2, 4, 6 % relative to the dry binder amount.

This results in the mix designs, expressed in kg/m^3 and per 1 prism or 1 liter (sufficient for 3 prisms), presented in Table B.1 - B.3 on the next page.

Table B.1 - Paste trial mix design 1

Mix design 1: 100% GGBFS, 2% Na₂O = M1				
Input				
W/B	Ms	GGBFS/(GGBFS+CC)	Precursor/activator	Total precursor
0,35	0,7	1	26	400
Mix design composition				
	In kg/m ³	Per prism (g)	1 Liter mix (g)	
Calcined clay	0,00	0,00	0,00	
GGBFS	1171,74	299,97	1171,74	
SS solution	55,74	14,27	55,74	
SH solution	41,09	10,52	41,09	
SH extra water	374,12	95,77	374,12	
Sand	0,00	0,00	0,00	

Table B.2 - Paste trial mix design 2

Mix design 2: 100% GGBFS, 4% Na₂O = M2				
Input				
W/B	Ms	GGBFS/(GGBFS+CC)	Precursor/activator	Total precursor
0,35	0,7	1	13	400
Mix design composition				
	In kg/m ³	Per prism (g)	1 Liter mix (g)	
Calcined clay	0,00	0,00	0,00	
GGBFS	1141,44	292,21	1141,44	
SS solution	108,60	27,80	108,60	
SH solution	80,06	20,50	80,06	
SH extra water	329,39	84,32	329,39	
Sand	0,00	0,00	0,00	

Table B.3 - Paste trial mix design 3

Mix design 3: 100% GGBFS, 6% Na₂O = M3				
Input				
W/B	Ms	GGBFS/(GGBFS+CC)	Precursor/activator	Total precursor
0,35	0,7	1	9	400
Mix design composition				
	In kg/m ³	Per prism (g)	1 Liter mix (g)	
Calcined clay	0,00	0,00	0,00	
GGBFS	1115,79	285,64	1115,79	
SS solution	153,34	39,26	153,34	
SH solution	113,04	28,94	113,04	
SH extra water	291,51	74,63	291,51	
Sand	0,00	0,00	0,00	

Experiments performed during paste trials

The following experiments have been performed during the paste trials:

- Workability
- Flexural strength
- Compressive strength

Workability has been determined after mixing. Both flexural strength and compressive strength have been performed after curing the specimens for 1 day and 7 days.

Results of the paste trials

The paste trials started with preparation of the activator. The sodium silicate, sodium hydroxide and water are mixed according to the in Table B.1 - B.3 provided mix designs and stirred. After stirring, the buckets are sealed to prevent evaporation and stored for approximately 24 hours.

After 24 hours, the paste can be prepared. First, the ground granulated blast furnace slag is weighed according to the mix designs, for a batch of 1 liter. The ground granulated blast furnace slag is then mixed for 1 minute. After 1 minute, the activator solution is slowly added in the mix and is mixed for an additional minute. Then the mixture is hand-mixed with a scoop as the mixer does not mix all the way to the bottom of the bowl. This way, all ground granulated blast furnace slag is mixed in the paste. After hand mixing, the mix is mixed for 1 more minute at high speed.

Workability

After mixing, the first step was to perform a workability test. It has to be noted, that throughout the entire paste trial phase, only mixes 1 and 2 are used after all, since mix 3 settled extremely fast and is therefore determined “failed”.

The workability test contains of a small cone, which is filled in 2 steps with the paste. After filling, the cone is lifted and the machine provides 15 ticks to the mixture. After the 15 ticks, the spread is measured, providing information about the workability.

Results can be seen in Figure B.1 below:



Figure B.1 - Workability results of mix 1 and 2

As can be seen, both mixture 1 and 2 have similar results. With mix 1 having 230mm average spread and mixture 2 230mm spread. This indicates good workability. However, as mentioned, mix 3 can not be measured due to a very fast setting time.

Specimen preparation

After the workability test, the paste is poured in 40 x 40 x 160 mm prism molds made from styrofoam after the styrofoam was coated with demolding oil. The prisms are vibrated for 15 seconds and then covered with foil to prevent evaporation. After 1 day, the molds are removed and specimens are cured in the curing room at 20 °C and 95 % RH. In Figure B.2 below this process is visualized.



Figure B.2 - Preparation of paste in molds (left) and specimens in curing room (right)

Flexural strength

The trial stage consists of two mechanical property tests that are performed right after the other. These tests are flexural strength and compressive strength, of which the first is elaborated.

The test setup consists of a small-scale flexural strength machine. The specimens are labelled and put in the testing machine with the upper cast side facing towards the right. This way, the testing surface is as even as possible resulting in more accurate results. The specimens are tested at a starting load of 0.1kN, increasing with 0.1kN per second with a stop load of 20 %. The specimens, testing setup and results can be seen on the Figures B.3 and B.4 on the next page



Figure B.3 - Flexural strength test setup

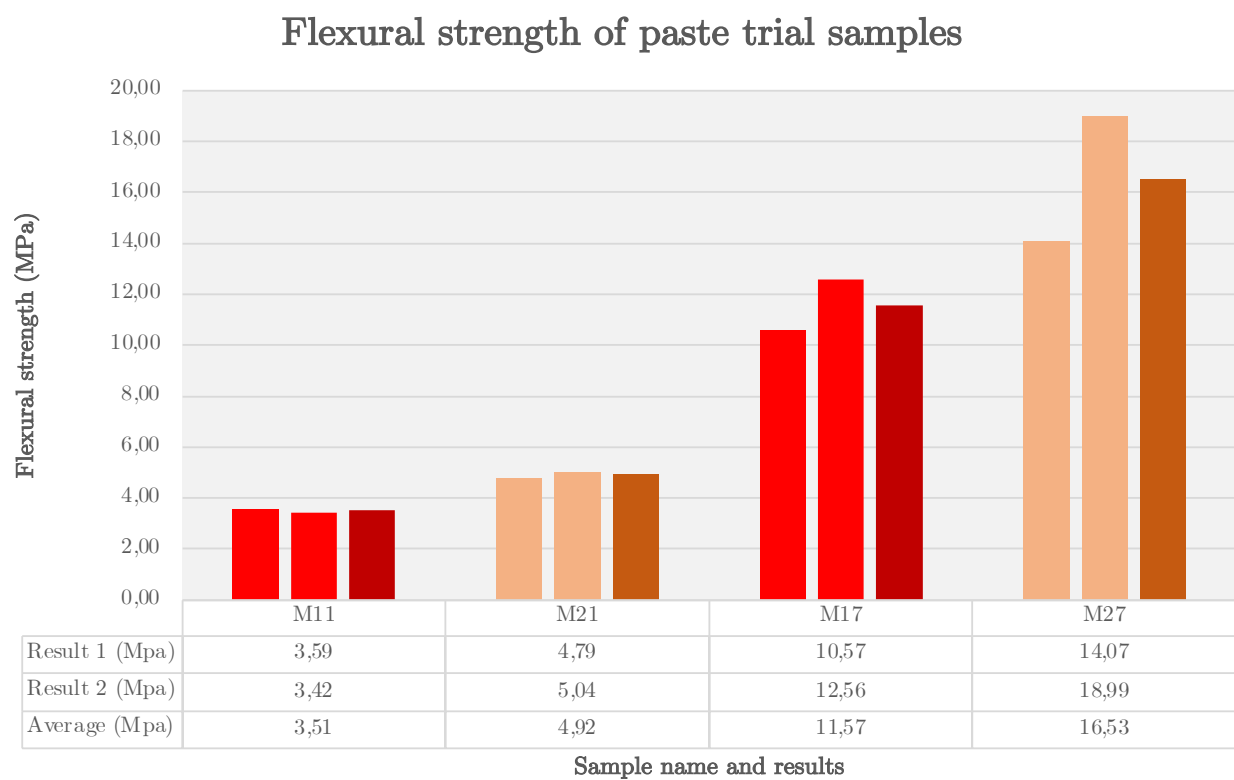


Figure B.4 - Flexural strength results

The mix design are labelled as follows:

M11 = Mix 1, curing for 1 day

M21 = Mix 2, curing for 1 day

M17 = Mix 1, curing for 7 days

M27 = Mix 2, curing for 7 days

Results for flexural strength are largely as expected. The second mixture performs better due to the higher sodium oxide content in the mixture. The difference between the two mixes is even more pronounced at the 7 day specimen results, albeit with large differences between the final results, varying from 14 to 19MPa for the second mix. This variation is possibly caused by irregularities in the specimens, as the styrofoam can introduce some concentrated load spikes causing faster failure of the specimens.

Compressive strength

For compressive strength testing, the same test setup is used, but the flexural strength module is replaced with the compressive strength module. As the specimens are broken in half, this enables compressive strength testing of an effective 40 x 40 mm surface area. Compressive strength is tested with a 1 kN starting load, with 2.4 kN/s increase of load with a 20 % stop load. The specimens and test setup can be seen on Figure B.5 on this page. Results are shown in Figure B.6 on the next page.



Figure B.5 - Compressive strength testing setup (left) and failed specimens (right)

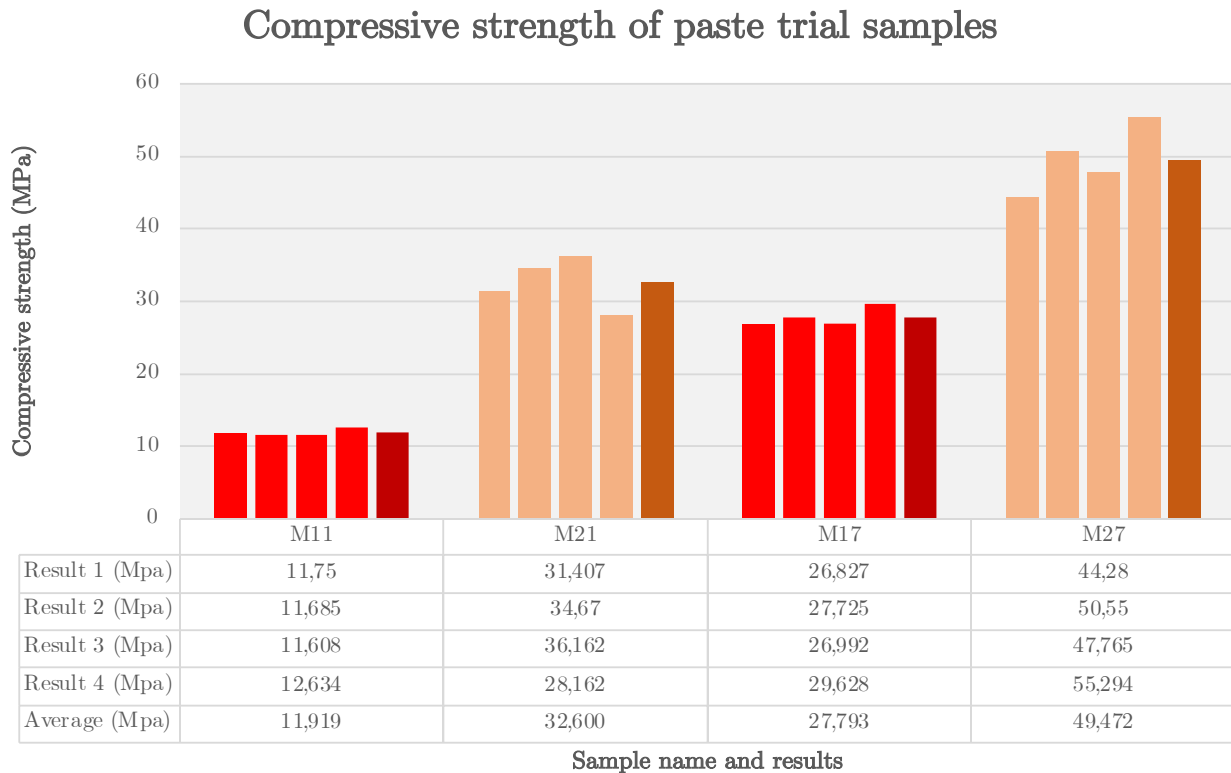


Figure B.6 - Compressive strength results

The results for compressive strength is as expected as well. Higher sodium oxide content results in higher early strength and higher 7 day compressive strength. It is especially obvious how much influence the sodium oxide content has looking at mix 2 after 1 day and mix 1 after 7 days of curing. Mix 2 is already stronger after 1 day than mix 1 after 7 days.

However, a mix might be workable and strong when making a paste, but its properties and behavior can change significantly when incorporating sand or sand with aggregates. Before composition of the final Taguchi array for the paste phase, another small trial has been executed in order to observe the effect of incorporating sand in the mixture to create a mortar as this study does not only focus on paste scale, but also mortar and concrete scale. This way, the water to binder ratio can be determined in such a way that the mixture compositions in terms of the precursor and activator can be kept as constant as possible throughout the study.

In order to determine this, a high strength mixture has been mixed with water to binder ratio of 0.35 (Mix 4) and 0.32 (Mix 5), of which the first water to binder ratio is the initial proposed value for the paste phase. Another mix design focussed on the effects on the workability when including calcined clay in the mixture (Mix 6). Mix 6 can be directly compared to Mix 1, as both have the same contents, but mix 6 has 50 % calcined clay and 50 % ground granulated blast furnace slag, whereas mix 1 has 100 % ground granulated blast furnace slag. Mix designs 4-6 can be seen in tables B.4 - B.6 on the next page.

Table B.4 - Paste trial mix design 4

Mix design 4: 100% GGBFS, 5% Na₂O, sand = M4				
Input				
W/B	Ms	GGBFS/(GGBFS+CC)	Precursor/activator	Total precursor
0,35	0,7	1	15	400
Mix design composition				
	In kg/m ³	Per prism (g)	1 Liter mix (g)	
Calcined clay	0,00	0,00	0,00	
GGBFS	400,00	102,40	400,00	
SS solution	47,12	12,06	47,12	
SH solution	34,74	8,89	34,74	
SH extra water	109,58	28,05	109,58	
		0,00		
Sand	1633,88	418,27	1633,88	

Table B.5 - Paste trial mix design 5

Mix design 5: 100% GGBFS, 5% Na₂O = M5				
Input				
W/B	Ms	GGBFS/(GGBFS+CC)	Precursor/activator	Total precursor
0,45	0,7	1	10,5	400
Mix design composition				
	In kg/m ³	Per prism (g)	1 Liter mix (g)	
Calcined clay	0,00	0,00	0,00	
GGBFS	400,00	102,40	400,00	
SS solution	47,12	12,06	47,12	
SH solution	34,74	8,89	34,74	
SH extra water	153,39	39,27	153,39	
Sand	1547,79	396,23	1547,79	

Table B.6 - Paste trial mix design 6

Mix design 6: 50% GGBFS, 2% Na₂O, sand, normal w = M6				
Input				
W/B	Ms	GGBFS/(GGBFS+CC)	Precursor/activator	Total precursor
0,35	0,7	0,5	26	400
Mix design composition				
	In kg/m ³	Per prism (g)	1 Liter mix (g)	
Calcined clay	608,73	155,83	608,73	
GGBFS	608,73	155,83	608,73	
SS solution	57,92	14,83	57,92	
SH solution	42,70	10,93	42,70	
SH extra water	388,72	99,51	388,72	
		0,00		
Sand	0,00	0,00	0,00	

Results

The paste and mortar trial phase had some very interesting results. The mixes are discussed one by one.

Paste trial mix 4

This mixture contains the same 0.35 water-to-binder ratio as the paste phase recipes. This recipe is specifically chosen to see the effects of including sand to create a mortar to the workability of the mixture. By determination of the workability, a good range for the water-to-binder ratio for the actual paste phase can be determined. This way, the parameters in this study do not need to drastically change in each new phase. To decrease workability, a high sodium oxide content was chosen as high sodium oxide content causes a failed mixture in the paste trials (mix 3, 6 % sodium oxide).

Mixing went fine, but it was immediately clear that the amount of water is too little. This causes the mixture to feel like moist sand, but crumbly moist sand. Testing workability resulted in that as well, see Figure B.7 below.

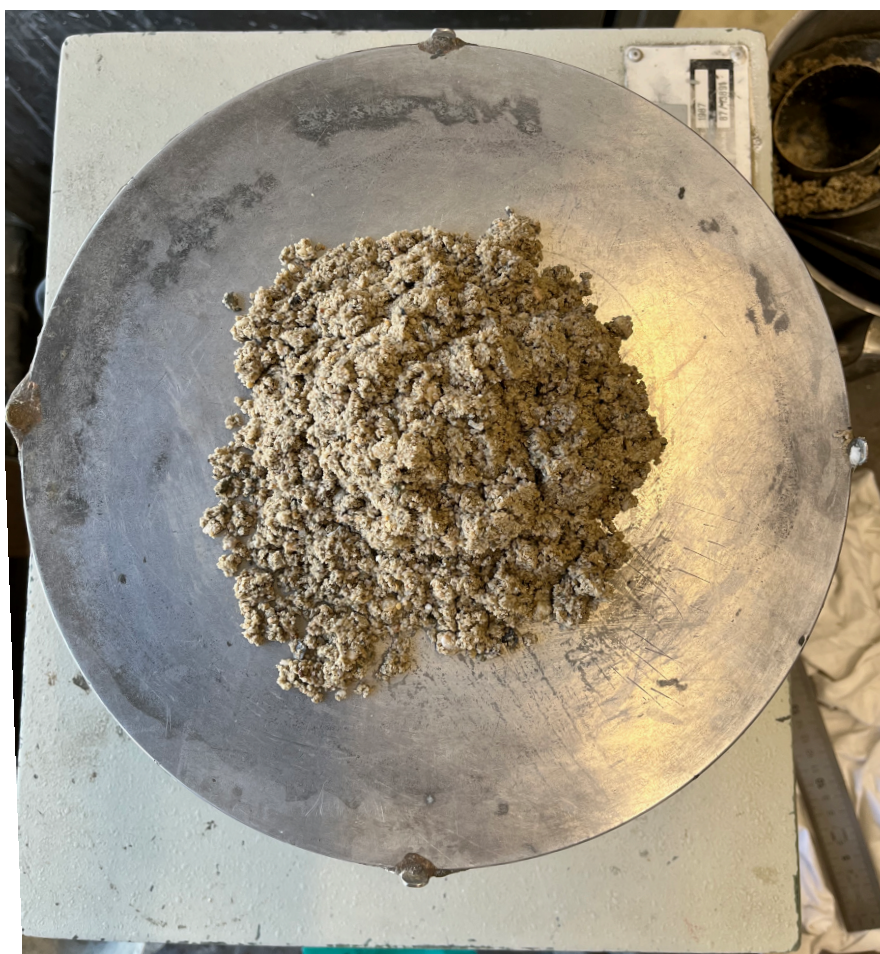


Figure B.7 - Workability test of Paste trial mix 4

It is clear that this is a non-suitable mix for actual production. Therefore, the water-to-binder ratio is increased by 0.1 (from 0.35 to 0.45) to clearly form a range of water-to-binder ratio.

Paste trial mix 5

As mentioned, this trial mix contains the same ingredients as paste trial mix 4, but the water-to-binder ratio is increased from 0.35 to 0.45. During mixing, the mixture is way more moist and quite good in terms of how to handle with the mix. This mixture's workability can be observed in Figure B.8 on the next page.

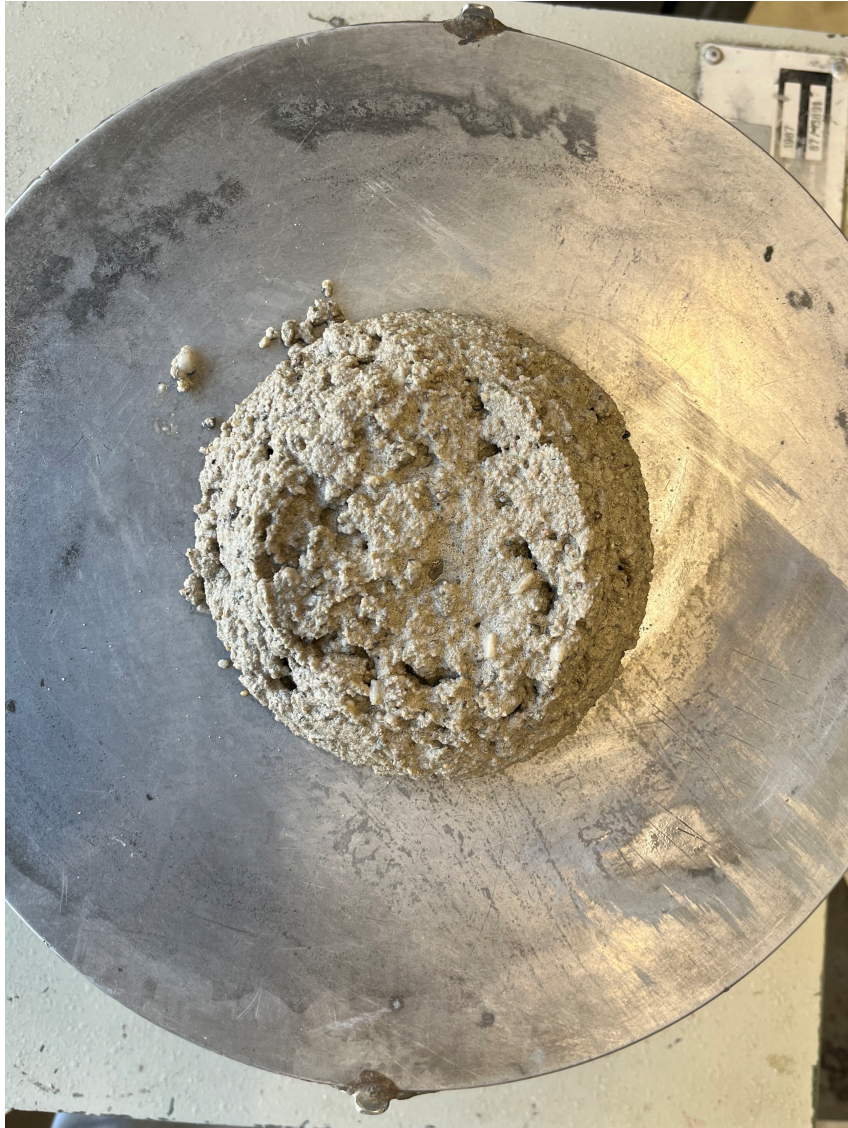


Figure B.8 - Workability test of Paste trial mix 5

Clearly, this is a better mix. However, it is near the limit in terms of workability, since it is on the wet side for an earth moist concrete, which is the final goal of workability in this research. Therefore, a water-to-binder ratio of 0.45 is chosen as the maximum value for the paste phase. 0.35 is still used for the paste phase, as a clear lower limit should be used as a lower water-to-binder ratio generally increases compressive strength.

Paste trial mix 6

Finally, it is time to validate the read information concerning calcined clay. Calcined clay can have great influence on workability due to its particle shape, specific surface area and particle size distribution. To observe this influence, the most workable mixture of the paste phase trials mixes 1 - 3 was chosen, where 50 % of the ground granulated blast furnace slag is replaced with calcined clay. Mixture 1 has the highest workability and is therefore used. The result of the workability can be observed in Figure B.9 on the next page.



Figure B.9 - Workability test of Paste trial mix 6

The workability is measured to be 150mm. This is quite a large difference compared to 230mm workability from mix M1. This also further supports the decision to use a water-to-binder ratio variation of 0.35-0.45 in the paste phase.

C_I

Alkali activated paste mix designs

P01 - Paste phase mix 1					
Input					
W/B	Ms	GGBFS/(GGBFS+CC)	Na2O %	Precursor/activator	Total precursor
0,35	0,4	1	3	20,5	400
Mix design composition					
	In kg/m ³	Per prism (g)	6 prisms = 2,5L needed (g)		
Calcined clay	0,00	0,00	0,00		
GGBFS	1161,73	297,40	2904,33		
Total precursor	1161,73	297,40	2904,33		
SS Solids	20,32	5,20	50,81		
SS Solution	46,19	11,82	115,48		
SH Solids	72,70	18,61	181,75		
Added Water to SH	364,22	93,24	910,56		
SH Total	436,93	111,85	1092,31		
Total activator	483,12	123,68	1207,79		
Total density	1644,85				

Figure C.1 - Paste phase mix 1

P02 - Paste phase mix 2					
Input					
W/B	Ms	GGBFS/(GGBFS+CC)	Na2O %	Precursor/activator	Total precursor
0,35	0,7	0,75	4	13,5	400
Mix design composition					
	In kg/m ³	Per prism (g)	6 prisms = 2,5L needed (g)		
Calcined clay	291,25	74,56	728,11		
GGBFS	873,74	223,68	2184,34		
Total precursor	1164,98	298,24	2912,45		
SS Solids	46,96	12,02	117,41		
SS Solution	106,73	27,32	266,83		
SH Solids	78,69	20,14	196,71		
Added Water to SH	338,83	86,74	847,08		
SH Total	417,52	106,88	1043,80		
Total activator	524,25	134,21	1310,63		
Total density	1689,23				

Figure C.2 - Paste phase mix 2

P03 - Paste phase mix 3					
Input					
W/B	Ms	GGBFS/(GGBFS+CC)	Na2O %	Precursor/activator	Total precursor
0,35	1	0,5	5	9,5	400
Mix design composition					
	In kg/m ³	Per prism (g)	6 prisms = 2,5L needed (g)		
Calcined clay	582,33	149,08	1455,81		
GGBFS	582,33	149,08	1455,81		
Total precursor	1164,65	298,15	2911,63		
SS Solids	84,13	21,54	210,33		
SS Solution	191,21	48,95	478,02		
SH Solids	76,96	19,70	192,41		
Added Water to SH	304,98	78,07	762,44		
SH Total	381,94	97,78	954,85		
Total activator	573,15	146,73	1432,87		
Total density	1737,80				

Figure C.3 - Paste phase mix 3

P04 - Paste phase mix 4					
Input					
W/B	Ms	GGBFS/(GGBFS+CC)	Na2O %	Precursor/activator	Total precursor
0,4	1	1	4	12	400
Mix design composition					
	In kg/m ³	Per prism (g)	6 prisms = 2,5L needed (g)		
Calcined clay	0,00	0,00	0,00		
GGBFS	1072,54	274,57	2681,36		
Total precursor	1072,54	274,57	2681,36		
SS Solids	61,34	15,70	153,34		
SS Solution	139,40	35,69	348,50		
SH Solids	56,11	14,36	140,28		
Added Water to SH	358,65	91,81	896,62		
SH Total	414,76	106,18	1036,90		
Total activator	554,16	141,87	1385,40		
Total density	1626,70				

Figure C.4 - Paste phase mix 4

P05 - Paste phase mix 5					
Input					
W/B	Ms	GGBFS/(GGBFS+CC)	Na2O %	Precursor/activator	Total precursor
0,4	0,4	0,75	5	12,5	400
Mix design composition					
	In kg/m ³	Per prism (g)	6 prisms = 2,5L needed (g)		
Calcined clay	272,34	69,72	680,85		
GGBFS	817,02	209,16	2042,56		
Total precursor	1089,37	278,88	2723,41		
SS Solids	31,25	8,00	78,14		
SS Solution	71,03	18,18	177,58		
SH Solids	111,80	28,62	279,51		
Added Water to SH	374,93	95,98	937,31		
SH Total	486,73	124,60	1216,82		
Total activator	557,76	142,79	1394,41		
Total density	1647,13				

Figure C.5 - Paste phase mix 5

P06 - Paste phase mix 6					
Input					
W/B	Ms	GGBFS/(GGBFS+CC)	Na2O %	Precursor/activator	Total precursor
0,4	0,7	0,5	3	18	400
Mix design composition					
	In kg/m ³	Per prism (g)	6 prisms = 2,5L needed (g)		
Calcined clay	565,47	144,76	1413,67		
GGBFS	565,47	144,76	1413,67		
Total precursor	1130,93	289,52	2827,33		
SS Solids	34,19	8,75	85,48		
SS Solution	77,71	19,89	194,28		
SH Solids	57,29	14,67	143,22		
Added Water to SH	405,34	103,77	1013,36		
SH Total	462,63	118,43	1156,58		
Total activator	540,34	138,33	1350,86		
Total density	1671,28	427,85	4178,19		

Figure C.6 - Paste phase mix 6

P07 - Paste phase mix 7					
Input					
W/B	Ms	GGBFS/(GGBFS+CC)	Na2O %	Precursor/activator	Total precursor
0,45	0,7	1	5	10,5	400
Mix design composition					
	In kg/m ³	Per prism (g)	6 prisms = 2,5L needed (g)		
Calcined clay	0,00	0,00	0,00		
GGBFS	1003,61	256,92	2509,03		
Total precursor	1003,61	256,92	2509,03		
SS Solids	52,02	13,32	130,04		
SS Solution	118,22	30,26	295,55		
SH Solids	87,15	22,31	217,88		
Added Water to SH	384,86	98,52	962,14		
SH Total	472,01	120,83	1180,03		
Total activator	590,23	151,10	1475,58		
Total density	1593,84				

Figure C.7 - Paste phase mix 7

P08 - Paste phase mix 8					
Input					
W/B	Ms	GGBFS/(GGBFS+CC)	Na2O %	Precursor/activator	Total precursor
0,45	1	0,75	3	16	400
Mix design composition					
	In kg/m ³	Per prism (g)	6 prisms = 2,5L needed (g)		
Calcined clay	261,35	66,91	653,38		
GGBFS	784,06	200,72	1960,14		
Total precursor	1045,41	267,62	2613,52		
SS Solids	44,84	11,48	112,10		
SS Solution	101,91	26,09	254,77		
SH Solids	41,02	10,50	102,55		
Added Water to SH	422,26	108,10	1055,65		
SH Total	463,28	118,60	1158,19		
Total activator	565,18	144,69	1412,96		
Total density	1610,59	412,31	4026,48		

Figure C.8 - Paste phase mix 8

P09 - Paste phase mix 9					
Input					
W/B	Ms	GGBFS/(GGBFS+CC)	Na2O %	Precursor/activator	Total precursor
0,45	0,4	0,5	4	15,5	400
Mix design composition					
	In kg/m ³	Per prism (g)	6 prisms = 2,5L needed (g)		
Calcined clay	529,18	135,47	1322,94		
GGBFS	529,18	135,47	1322,94		
Total precursor	1058,35	270,94	2645,88		
SS Solids	24,49	6,27	61,22		
SS Solution	55,65	14,25	139,14		
SH Solids	87,60	22,42	218,99		
Added Water to SH	432,02	110,60	1080,05		
SH Total	519,62	133,02	1299,04		
Total activator	575,27	147,27	1438,18		
Total density	1633,62				

Figure C.9 - Paste phase mix 9

D_i

Alkali activated mortar mix designs

M01 - Mortar phase mix 1					
Input					
W/B	Ms	GGBFS/(GGBFS+CC)	Na2O %	Precursor/activator	Total precursor
0,35	0,4	1	3	20,5	400
Mix design composition					
	In kg/m ³	Per prism (g)	6 prisms = 2,5L needed (g)		
Calcined clay	0,00	0,00	0,00		
GGBFS	625,15	160,04	1562,87		
Total precursor	625,15	160,04	1562,87		
SS Solids	10,94	2,80	27,34		
SS Solution	24,86	6,36	62,14		
SH Solution	39,12	10,02	97,80		
Added Water to SH	195,99	50,17	489,99		
SH Total	235,12	60,19	587,79		
Total activator	259,97	66,55	649,93		
Sand	1172,15	300,07	2930,38		
Total density	2057,27				

Figure D.1 - Mortar phase mix 1

M02 - Mortar phase mix 2					
Input					
W/B	Ms	GGBFS/(GGBFS+CC)	Na2O %	Precursor/activator	Total precursor
0,35	0,7	0,75	4	13,5	400
Mix design composition					
	In kg/m ³	Per prism (g)	6 prisms = 2,5L needed (g)		
Calcined clay	156,28	40,01	390,70		
GGBFS	468,84	120,02	1172,09		
Total precursor	625,12	160,03	1562,79		
SS Solids	25,20	6,45	63,00		
SS Solution	57,27	14,66	143,18		
SH Solution	42,22	10,81	105,55		
Added Water to SH	181,81	46,54	454,54		
SH Total	224,04	57,35	560,09		
Total activator	281,31	72,01	703,27		
Sand	1172,09	300,06	2930,23		
Total density	2078,52				

Figure D.2 - Mortar phase mix 2

M03 - Mortar phase mix 3					
Input					
W/B	Ms	GGBFS/(GGBFS+CC)	Na2O %	Precursor/activator	Total precursor
0,35	1	0,5	5	9,5	400
Mix design composition					
	In kg/m³	Per prism (g)	6 prisms = 2,5L needed (g)		
Calcined clay	312,65	80,04	781,63		
GGBFS	312,65	80,04	781,63		
Total precursor	625,31	160,08	1563,27		
SS Solids	45,17	11,56	112,93		
SS Solution	102,66	26,28	256,65		
SH Solution	41,32	10,58	103,31		
Added Water to SH	163,74	41,92	409,36		
SH Total	205,07	52,50	512,67		
Total activator	307,73	78,78	769,32		
Sand	1172,45	300,15	2931,12		
Total density	2105,48				

Figure D.3 - Mortar phase mix 3

M04 - Mortar phase mix 4					
Input					
W/B	Ms	GGBFS/(GGBFS+CC)	Na2O %	Precursor/activator	Total precursor
0,4	1	1	4	12	400
Mix design composition					
	In kg/m ³	Per prism (g)	6 prisms = 2,5L needed (g)		
Calcined clay	0,00	0,00	0,00		
GGBFS	600,22	153,66	1500,54		
Total precursor	600,22	153,66	1500,54		
SS Solids	34,33	8,79	85,81		
SS Solution	78,01	19,97	195,03		
SH Solution	31,40	8,04	78,50		
Added Water to SH	200,71	51,38	501,77		
SH Total	232,11	59,42	580,27		
Total activator	310,12	79,39	775,30		
Sand	1125,40	288,10	2813,51		
Total density	2035,74				

Figure D.4 - Mortar phase mix 4

M05 - Mortar phase mix 5					
Input					
W/B	Ms	GGBFS/(GGBFS+CC)	Na2O %	Precursor/activator	Total precursor
0,4	0,4	0,75	5	12,5	400
Mix design composition					
	In kg/m³	Per prism (g)	6 prisms = 2,5L needed (g)		
Calcined clay	149,09	38,17	372,73		
GGBFS	447,27	114,50	1118,18		
Total precursor	596,36	152,67	1490,91		
SS Solids	17,11	4,38	42,78		
SS Solution	38,89	9,95	97,22		
SH Solution	61,21	15,67	153,01		
Added Water to SH	205,25	52,54	513,12		
SH Total	266,46	68,21	666,14		
Total activator	305,34	78,17	763,36		
Sand	1118,18	286,25	2795,46		
Total density	2019,89				

Figure D.5 - Mortar phase mix 5

M06 - Mortar phase mix 6					
Input					
W/B	Ms	GGBFS/(GGBFS+CC)	Na2O %	Precursor/activator	Total precursor
0,4	0,7	0,5	3	18	400
Mix design composition					
	In kg/m ³	Per prism (g)	6 prisms = 2,5L needed (g)		
Calcined clay	309,20	79,15	772,99		
GGBFS	309,20	79,15	772,99		
Total precursor	618,39	158,31	1545,98		
SS Solids	18,70	4,79	46,74		
SS Solution	42,49	10,88	106,23		
SH Solution	31,33	8,02	78,31		
Added Water to SH	221,64	56,74	554,10		
SH Total	252,97	64,76	632,41		
Total activator	295,46	75,64	738,64		
Sand	1159,48	296,83	2898,71		
Total density	2073,33	530,77	5183,33		

Figure D.6 - Mortar phase mix 6

M07 - Mortar phase mix 7					
Input					
W/B	Ms	GGBFS/(GGBFS+CC)	Na2O %	Precursor/activator	Total precursor
0,45	0,7	1	5	10,5	400
Mix design composition					
	In kg/m³	Per prism (g)	6 prisms = 2,5L needed (g)		
Calcined clay	0,00	0,00	0,00		
GGBFS	572,29	146,51	1430,72		
Total precursor	572,29	146,51	1430,72		
SS Solids	29,66	7,59	74,15		
SS Solution	67,41	17,26	168,53		
SH Solution	49,70	12,72	124,24		
Added Water to SH	219,46	56,18	548,64		
SH Total	269,15	68,90	672,89		
Total activator	336,57	86,16	841,42		
Sand	1073,04	274,70	2682,60		
Total density	1981,90				

Figure D.7 - Mortar phase mix 7

M08 - Mortar phase mix 8					
Input					
W/B	Ms	GGBFS/(GGBFS+CC)	Na2O %	Precursor/activator	Total precursor
0,45	1	0,75	3	16	400
Mix design composition					
	In kg/m ³	Per prism (g)	6 prisms = 2,5L needed (g)		
Calcined clay	148,48	38,01	371,20		
GGBFS	445,44	114,03	1113,60		
Total precursor	593,92	152,04	1484,80		
SS Solids	25,47	6,52	63,68		
SS Solution	57,90	14,82	144,74		
SH Solution	23,30	5,97	58,26		
Added Water to SH	239,89	61,41	599,74		
SH Total	263,20	67,38	658,00		
Total activator	321,09	82,20	802,73		
Sand	1113,60	285,08	2784,00		
Total density	2028,61	519,32	5071,53		

Figure D.8 - Mortar phase mix 8

M09 - Mortar phase mix 9					
Input					
W/B	Ms	GGBFS/(GGBFS+CC)	Na2O %	Precursor/activator	Total precursor
0,45	0,4	0,5	4	15,5	400
Mix design composition					
	In kg/m ³	Per prism (g)	6 prisms = 2,5L needed (g)		
Calcined clay	295,19	75,57	737,99		
GGBFS	295,19	75,57	737,99		
Total precursor	590,39	151,14	1475,97		
SS Solids	13,66	3,50	34,15		
SS Solution	31,05	7,95	77,61		
SH Solution	48,87	12,51	122,16		
Added Water to SH	241,00	61,70	602,49		
SH Total	289,86	74,20	724,66		
Total activator	320,91	82,15	802,27		
Sand	1106,98	283,39	2767,45		
Total density	2018,28				

Figure D.9 - Mortar phase mix 9

Alkali activated concrete phase trials

In previous phases, the paste phase and the mortar phase, mix designs are rather fluid and can easily be compacted. However, this research focuses on development of alkali activated concrete with earth moist concrete workability requirement. Some trials have been performed prior to execution of the concrete phase to determine the optimal method for compaction and to fine tune the upscaled mixes to be categorized as earth moist concrete.

Besides the earth moist concrete requirement, the type of aggregates is also changed in the concrete phase. TU Delft aggregates are no longer used and aggregates provided by industry are substituted. During this trial, the difference between using the two aggregates is evaluated as well, since aggregates of industry have higher water absorption which can significantly change workability properties. To ensure that the upscaling from mortar towards concrete is not too large of a step, the particle size distributions of both aggregate types is matched based on a mix design delivered by industry. Particle size distributions have been obtained by sieving all aggregate types used in this research, see Table E.1 below.

Table E.1 - Aggregates used in research

Aggregate from TU Delft	Aggregate from industry
Standard sand (0-4 fraction)	Concrete sand (0-2 fraction)
Sand (0.125-0.25 fraction)	Blast furnace slag (2-8 fraction)
Sand (0.5-1 fraction)	Copper slag (0-4 fraction)
Sand (1-2 fraction)	Secondary aggregates (0-10 fraction)
Sand (2-4 fraction)	
Sand (4-8 fraction)	

By measuring the particle size distribution of the industry aggregates first, the particle size distribution of the four aggregate types can be combined in the right proportions according to the delivered mix design. To match TU Delft aggregates to this particle size distribution, the standard sand was first measured and compared to the combined graph from industry. Based on that result, the difference is accounted for by the other 5 fractions shown in Table E.1. Finally, by proportioning the aggregates from TU Delft, the combined particle size distribution of both industry and TU Delft aggregates is obtained and is shown in Figure E.1 on the next page.

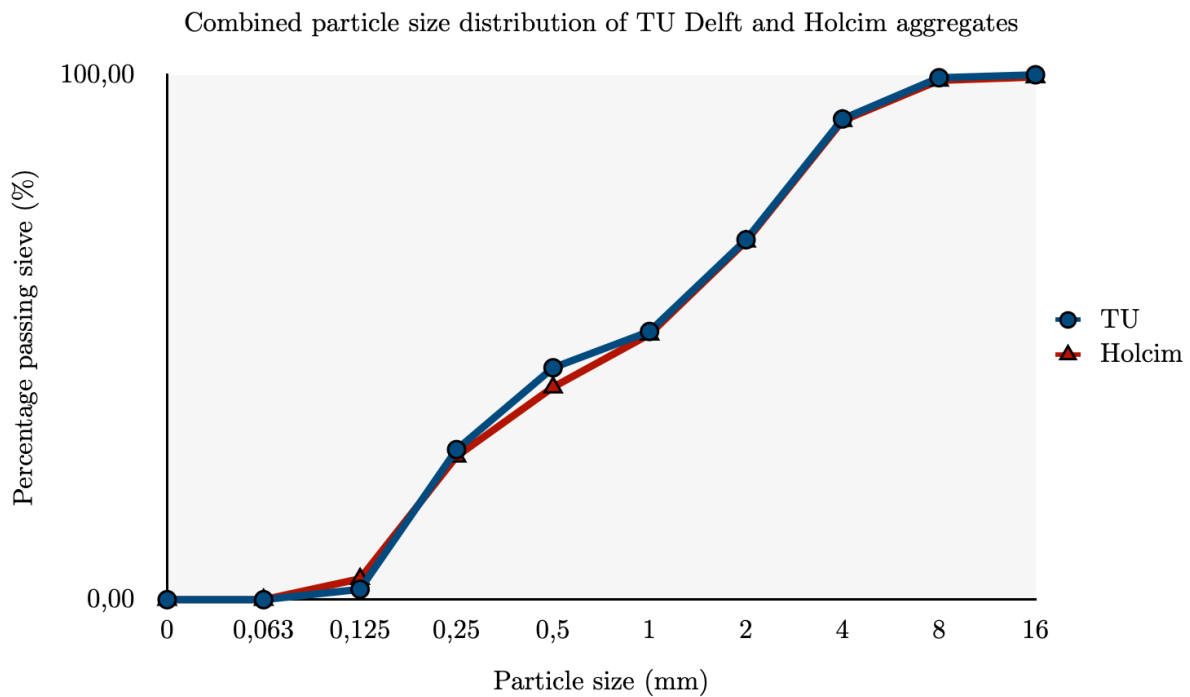


Figure E.1 - Combined particle size distribution

After the mortar phase, it is concluded that M01 and M05 are the mix designs that are upscaled towards concrete scale. These mix designs are directly upscaled, where paste composition is used as a base line and the aggregate composition is based on both specific gravity of the aggregate groups and total available volume of the concrete (as the paste has known volume). To efficiently prepare for the concrete phase, the mix designs as described in Table E.2 are tested on workability to test if the mix designs comply with earth moist concrete or if modifications are needed.

Table E.2 - Mix description of concrete trial mix designs

Mix name	Modifications
CT01	Direct upscaling of M01, TU Delft aggregates
CT02	Direct upscaling of M01, industry aggregates
CT03	Direct upscaling of M05, TU Delft aggregates
CT04	Upscaling of M05 with slight modification, industry aggregates
CT05	Testing of industry concrete mix design

The procedure for workability is described using Figure E.2 on the next page.



Figure E.2 - Procedure for workability measurement

Going from left to right, the following steps are performed:

- Gathering of all materials.
- Mixing all materials:
 - Moisten the concrete mixer
 - First add coarse aggregates, mix for 1 minute
 - Add fine aggregates, mix for 1 minute
 - Add precursor components or cement, mix for 1 minute
 - Slowly water or activator, mix for 3 minutes
 - Pour materials into wheelbarrow
- Fill “Vat van Waltz” from four sides according to standard.
- Vibrate until no further compaction is visible. Measure compaction distance from all four sides as indicated by red arrows.

From the standard, the compaction range should be between 1.26 and 1.45 for earth moist concrete. This translates to a compaction distance of 8.1 cm to 12.4 cm. Results for workability are shown in Table E.3 below.

Table E.3 - Workability results concrete trial

Mix design name	Workability result (cm)	Compaction value	Complies with EMC
CT01	11,35	1,40	✓
CT02	12,30	1,44	✓
CT03	10,90	1,37	✓
CT04	13,30	1,50	x
CT05	9,20	1,30	✓

All mix designs but CT04 (M05 upscaled with industry aggregates, no modifications) comply with the earth moist concrete workability requirement, but the concrete mixtures had quite different behavior which is mandatory to elaborate. First of all, all 5 trial mix designs had formation of chunks during mixing, this is clearly visible in Figure E.2. These chunks, especially for the alkali activated concretes, were difficult to remove during vibration which can influence the workability results. Actual workability would be towards the lower side of the compaction value, as less of these large chunks equals more material in the vat van Waltz, thus less compactability. Another very important observation is the amount of fluids observed after vibration of the mix designs. For example mix design CT04. The compaction value does not comply with the earth moist concrete

requirement. Being on the high side of the range, this translates towards a too dry concrete. However, when visually observing the state of the concrete after vibration, some small amount of moisture was visible on both the top of the vat van Waltz, but also on the sides of the concrete after removing the vat of Waltz, see Figure E.3 below.



Figure E.3 - Results of workability of CT04

According to the workability standard, the concrete is too dry, but visually it is more moist than the reference concrete of industry (CT05). When compacting the concrete in the pressing and vibration combination machine, it is important that the concrete is not too wet because that introduces friction on the side walls of the mould, which can deform the shape of the specimen or even cause removal of some of the material (as is also visible on Figure E.3, right figure, top of the concrete). So even though CT04 does not comply with the earth moist concrete workability according to the standard, it does seem suitable for the compaction method that is applied for creating the concrete specimens (vibration + pressure). If CT04 were to be modified in such a way that it complies with the workability requirement, more fluids (both activator and water) needs to be introduced to lower the compactability, but that would mean that the final concrete is too wet for the machine in which it is compacted.

To verify the workability of the most promising trial mixtures (CT02, CT04 and CT05 or industry reference) the concrete has been put in the pressing and vibration combination machine which is property of industry. Figure E.4 on the next page shows some examples which are elaborated.



Figure E.4 - Workability results from small compaction machine

A lot of trials are performed with the small compaction machine from industry, especially with their in-house mix design which is referred to as the reference mixture in this research. Figure E.4 shows the evolution of moisture and the resulting product very nicely:

1. Concrete is too dry, top side crumbles away very easily.
2. Concrete still too dry, but less crumbling.
3. Concrete is almost in good state, just a little too dry due to little crumbling near the edges.
4. Concrete is just right. A little bit of moisture leaks from the outer shell due to a little too long vibration time, but the corners are just enough compacted.

The final mix designs have been tested until good cubes are formed. It turns out that the directly upscaled M01 with industry aggregates needed no modifications. Upscaled M05 with industry aggregates had less need for moisture. The total activator solution was therefore reduced a little until the cubes had good shape as number 4 in Figure E.4. The industry mixture needed little more moisture than the recipe as the big compaction machine in the factory requires less moisture than the smaller compaction machine.

Please note that the compaction machine from industry does not completely result in optimal compaction of the specimens. To illustrate this, Figure E.5.

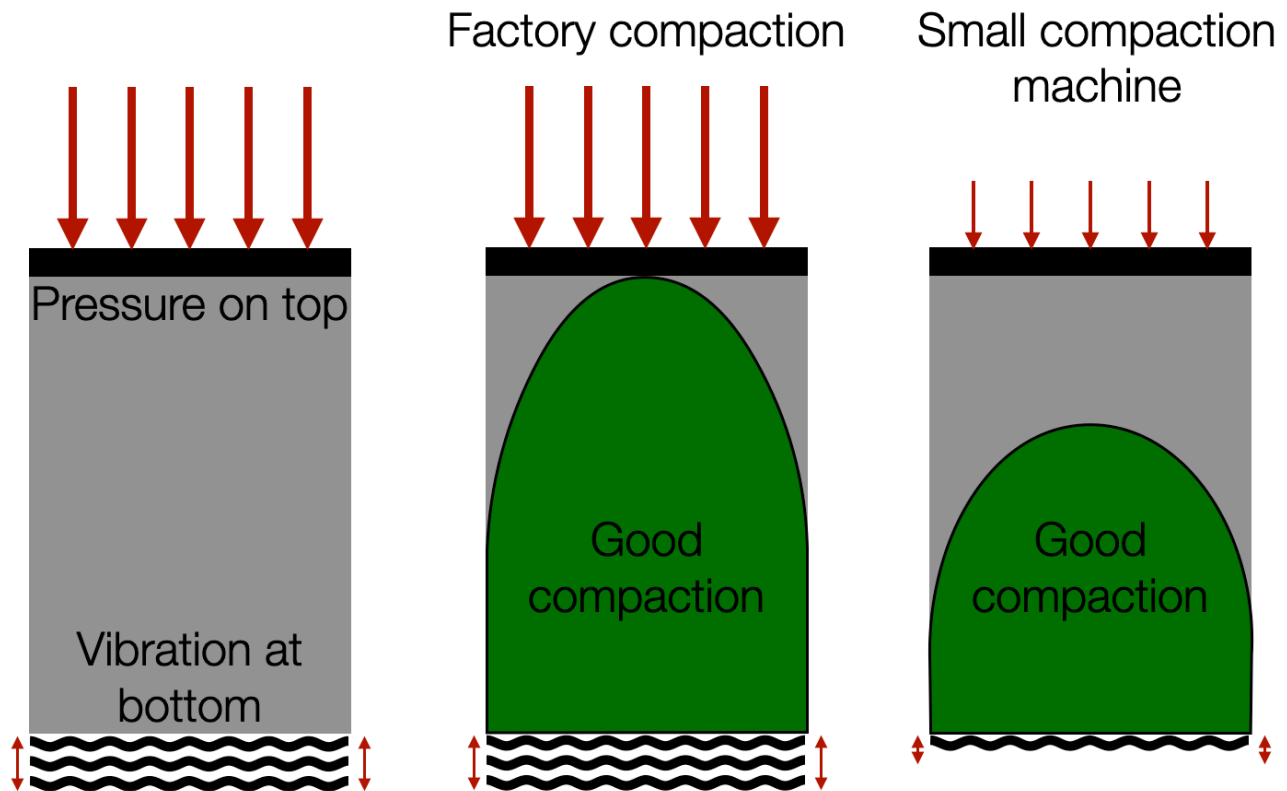


Figure E.5 - Compaction differences between factory and small compaction machine visualized

As can be seen in Figure E.5, the compaction method for this research makes use of a vibrating motor at the bottom of the specimens, whilst even pressure is applied to the top of the specimens. Due to this combination and the behavior of the concrete, there is always a region within the specimen which is optimally compacted (in green) and a region which is weaker (in grey). Therefore, mix designs need to be adjusted such that the weaker parts also have sufficient resistance and durability.

However, this research uses the small compaction machine from industry and not the larger machine in the factory. As this machine has less vibration energy and also can not apply as much pressure as the machine in the factory, the region of good compaction becomes smaller. This results in formation of a more porous structure in the top half of the specimens. This explains why performance results are not always met for the specimens, both in case of the alkali activated concrete and the industry concrete, as worse compaction degrades the performance.

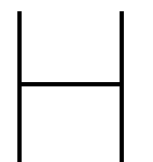
Alkali activated concrete mix designs

C01 - Concrete mix 1, upscaled from P01 and M01					
Input					
W/B	Ms	GGBFS/(GGBFS+CC)	Na2O %	Precursor/activator	Total precursor
0,35	0,4	1	3%	20,5	400
Mix design composition					
	In kg/m ³	Volume (m ³)	For 5 cubes (19L, in kg)	For 1 cube (3,75L, in kg)	
Calcined clay	0,00	0,00	0,00	0,000	
GGBFS	400,00	0,14	7,60	1,520	
SS solution	15,90	0,01	0,30	0,060	
SH solution	25,03	0,02	0,48	0,095	
SH extra water	125,41	0,13	2,38	0,477	
Aggregates > 4mm	535,98	0,21	10,18	2,037	
Aggregates < 4mm	1294,02	0,48	24,59	4,917	
Total	2396,34	1	45,53	9,106	

Figure F.1 - Concrete phase mix 1

C05 - Concrete mix 2, upscaled from P05 and M05					
Input					
W/B	Ms	GGBFS/(GGBFS+CC)	Na2O %	Precursor/activator	Total precursor
0,4	0,4	0,75	4%	14,9	400
Mix design composition					
	In kg/m ³	Volume (m ³)	For 5 cubes (19L, in kg)	For 1 cube (3,75L, in kg)	
Calcined clay	100,00	0,04	1,90	0,380	
GGBFS	300,00	0,11	5,70	1,140	
SS solution	21,90	0,01	0,42	0,083	
SH solution	34,50	0,02	0,66	0,131	
SH extra water	115,60	0,12	2,20	0,439	
Aggregates > 4mm	503,36	0,20	9,56	1,913	
Aggregates < 4mm	1214,64	0,45	23,08	4,616	
Total	2290,00	1	43,51	8,702	

Figure F.2 - Concrete phase mix 2



Full experimental setup

Basic material properties

Aggregate shape

The used method

When working with unknown aggregates it is important to determine their shape in order to know their external characteristics which influence the behavior in a mix design. For example, particles with a lot of visible pores are more likely to absorb more water and flaky particles tend to be orientated in one plane with greater possibility of bleeding as a result. This research uses the flakiness and shape index with help from particle classifications of the British standard in order to determine the shape of the aggregates, BS812: Part 1: 1975 .






Scope

Determine the shape and flakiness index of the coarse aggregates groups

Process

As the proper equipment to test this using the dutch NEN-EN 933-3:2012 is not available, it is decided to determine the shape and flakiness index by separating a 100 particle group from the coarse aggregate groups. These aggregates will be separated by shape as described in table H.1 below. Note that the drawings of the shape is indicative as it is only 2D instead of 3D.

Table H.1 - Particle shape classification according to BS812: Part 1: 1975.

Classification		Description	Example
Rounded		Fully water-worn or shaped by attrition	River or seashore gravel. Desert or wind-blown sand.
Irregular		Naturally irregular or partly shaped by attrition. Edges may be rounded.	Other gravels, land or dug flint.
Flaky		Particles where the thickness is small relative to the other two dimensions.	Laminated rock
Angular		Well-defined edges formed at intersection of roughly planar faces.	Crushed rocks of all types.
Elongated		Usually angular, where the length is considerably larger than the other two dimensions	
Flaky and elongated		Length >> width and width >> thickness	

After separation, the following steps are executed:

1. Take a photo of the sorted aggregates by shape
2. Count the number of aggregates per shape and add notes on their structures. Put results in a table.
3. Interpretation of results and conclusion

Aggregate particle size distribution

The used method

Knowing the particle size distributions (PSD) of each aggregate heap used in a concrete mixture is very important as it influences the particle composition within the concrete mix. The particle size distribution is performed in accordance with NEN-EN 933-1:2012(E)

Scope

Determine the efficiency of the current particle size distribution of industry aggregates by measuring and combining results from the most used aggregates and the amount that is currently used in each cubic meter of concrete.

Test setup figures

The test setup is shown in Figure H.1 below.

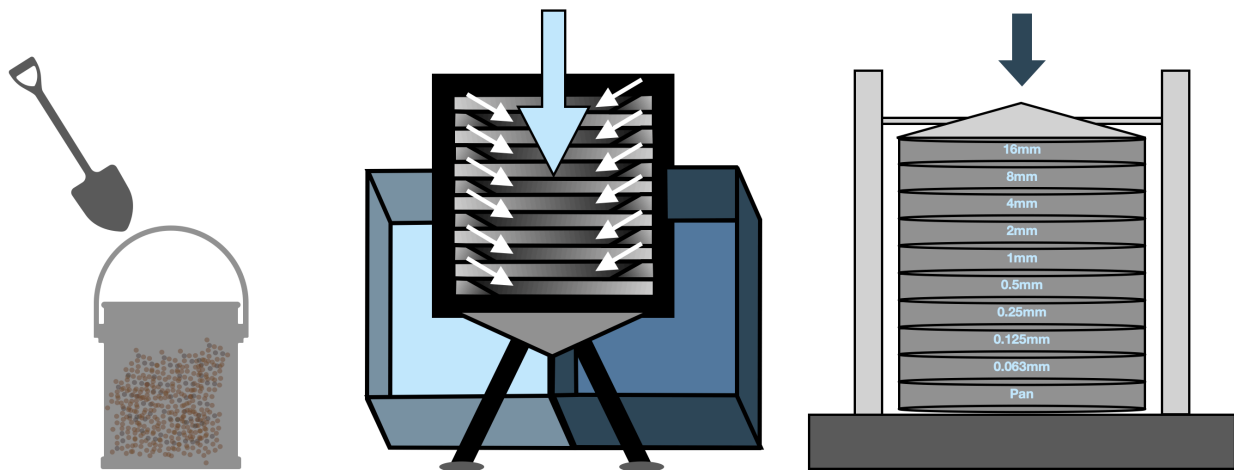


Figure H.1 - Testing setup of particle size distribution of aggregates

The testing setup consists of gathering of material (left). This material is put inside a splitter (middle) to create a representative specimen: a specimen that is a confident representation of the entire heaps of material. This representative specimen is sieved (right) after which the particle size distribution is determined.

Process

The following steps are to be executed for this test:

1. Create a representative specimen by splitting aggregates of the aggregate group concerned. Mass of the specimen should at least 1kg for the aggregates with maximum size 10mm. For the concrete sand ($<4\text{mm}$) this specimen may be 0.2kg. The aggregates should be dried before splitting.
2. Stack sieves from small (0mm, pan) to large (16mm) and put the specimen in the highest sieve.
3. Sieve for 7 minutes with 1mm amplitude.
4. Weigh the mass of the material that is retained on each sieve.
5. Make a table of the mass retained and cumulative percentage passed.
6. Create a PSD graph of the aggregate group.
7. Repeat 1-6 for the other aggregate groups, then create 1 cumulative PSD graph according to mix design.
8. Interpretation of results and conclusion.

Aggregate water absorption and specific gravity

The used method

The amount of water that should be used in any type of concrete mixture is very important as it directly affects its strength and resistance to durability affecting parameters. Therefore a measurement of water absorption and specific gravity is vital. This experiment is in accordance with NEN-EN1097-6:2013, where a modified pycnometer method is used.

Scope

Determination and comparison of water absorption and specific gravity of all used aggregate groups.

Process

The experiment has both water absorption and specific gravity as output, however, the norm states two different experiments depending on the class of aggregate: coarse aggregates (>4mm) or fine aggregates (<4mm).

The steps are described in table H.2 below.

Table H.2 - Steps of water absorption and specific gravity test

Processing steps for water absorption and specific gravity test	
Coarse aggregates	Fine aggregates
1. Weigh 1kg of >4mm aggregates. Sieve if smaller particles are present.	1. Take 2kg of fine aggregates in surface saturated dry condition and put 500g (C) in pycnometer
2. Place sieved specimen in vessel and partially fill with distilled water.	2. Partially fill vessel with distilled water and shake to remove air bubbles. Then completely fill vessel.
3. Keep aggregates immersed for 24h, remove air bubbles by agitation and overfill vessel with distilled water. Weigh vessel (A).	3. Weigh pycnometer and contents (A)
4. Drain vessel, dry surface of aggregates on dry cloths.	4. Pour contents into a tray.
5. Refill vessel and weigh (B).	5. Fill pycnometer with water and weigh (B)
6. Weigh surface saturated dry coarse aggregates (C)	6. Decant water from tray in beaker with a filter. The filter can be put in the tray with its contents.
7. Place aggregates in oven of 105°C for 24h.	7. Place tray in oven of 105°C for 24h.
8. Cool aggregates and weigh them (D)	8. Cool aggregates, remove aggregate from filter and weigh them (D)

After these steps, both experiments can be completed with the following steps:

1. Determine specific gravity with $SG = \frac{D}{C - (A - B)}$
2. Determine water absorption with: $WA = \frac{100 \cdot (C - D)}{D}$
3. Put the results in a table.
4. Interpretation of results and discussion.

GGBFS, CC, SS and SH composition

The used method

Determination of the chemical composition of GGBFS, CC, SS and SH is very important as the four components combined form the binder of the alkali activated concrete. The chemical composition significantly influences the mixture composition that should be used.

For SS and SH, only the information provided by the manufacturer is needed to determine its chemical composition. For GGBFS and CC X-ray Fluorescence or XRF needs to be used. XRF is an analytical method to determine elemental composition of materials and phase composition (Masone, 2015). XRF needs only small specimens and is non-destructive. However, this data is available for both materials and will therefore not be repeated specifically for this research.

Scope

Determine the chemical composition of the precursors and activators used in this research

Process

Manufacturer data for SS, SH, GGBFS and CC

1. Look up what the molarity of SH is, this describes #mol Sodium hydroxide per liter of water.
2. Determine chemical composition based on molarity.
3. Look up what the composition of SS is, this describes % Na₂O, % SiO₂ and % H₂O.
4. Put results in table.
5. Find particle size distribution of GGBFS and CC and put results in graph.
6. Find specific surface area of GGBFS and CC and put results in table.
7. Determine amorphous content of CC from data from manufacture report.
8. Discussion.

Paste phase experiments

Workability

The used method

Workability is very important when it comes to earth moist concrete. In the end, the mixture should have workability which complies with earth moist concrete, namely consistency class C1. To determine the workability for the alkaline activated paste, the mortar procedure as described in NEN-EN 1015-3 is used.

Scope

Determination of the workability of alkali activated paste mix designs

Test setup figures

Figure H.2 shows the testing setup used for this experiment.

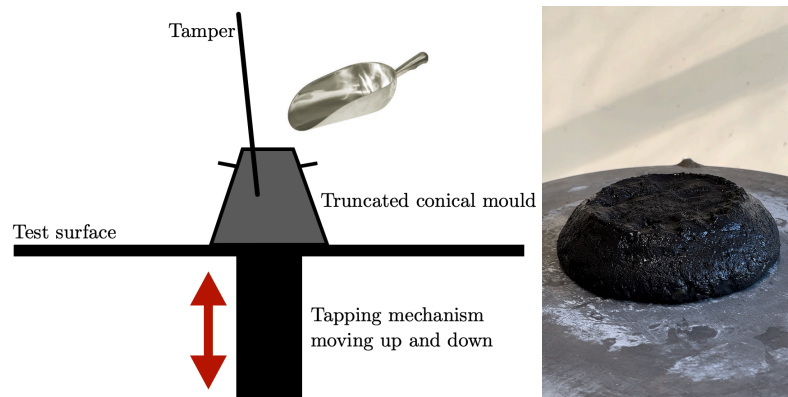


Figure H.2 - Testing setup of workability test of alkali activated paste

The testing setup consists of a test surface with truncated conical mould on top. The test surface is connected to a tapping mechanism, which moves the test surface and anything on top up and down. An example of the end result after tapping can be seen on the right of Figure 3.x.

Process

This test requires a ready mixed alkali activated paste, around 1.5 liter is sufficient. In order to determine the workability of the alkali activated paste, this experiment is conducted. The test consists of a small inverted cone and a table with dropping function. The workability is measured in terms of the spread of the mix after 15 drops of the table.

The following steps are conducted:

1. Preparation of the alkali activated paste mix.
2. Make the flat surface of the test bench moist, as well as the inside of the truncated conical mould.
3. Pour the paste until halfway in the cone and tap it with a tamper so it spreads evenly.
4. Fill the cone completely and tap it with the tamper again. Remove excess material by moving the tamper in a sawing, horizontal movement at the top of the truncated conical mould.
5. After 15s, remove the truncated conical mould vertically in an even manner.
6. Let the table drop for 15 times at an approximate rate of 1 drop/s.
7. Measure the diameter of the spread-out paste in two perpendicular directions.
8. Determine the workability by calculating the mean value of the two measurements.
9. Put results in table.
10. Interpretation of results and discussion.

Flexural strength

The used method

Flexural strength of concrete is important as this introduces tensile stress in the concrete. Concrete generally has a flexural strength capacity which is only 10% of its compressive strength capacity. To determine flexural strength, the mortar norm is followed. Thus, flexural strength of paste specimens is tested using NEN-EN 1015-11:2019.

Scope

Determination of the flexural strength of alkali activated paste mix designs

Test setup figures

Figure H.3 shows the testing setup used for this experiment.

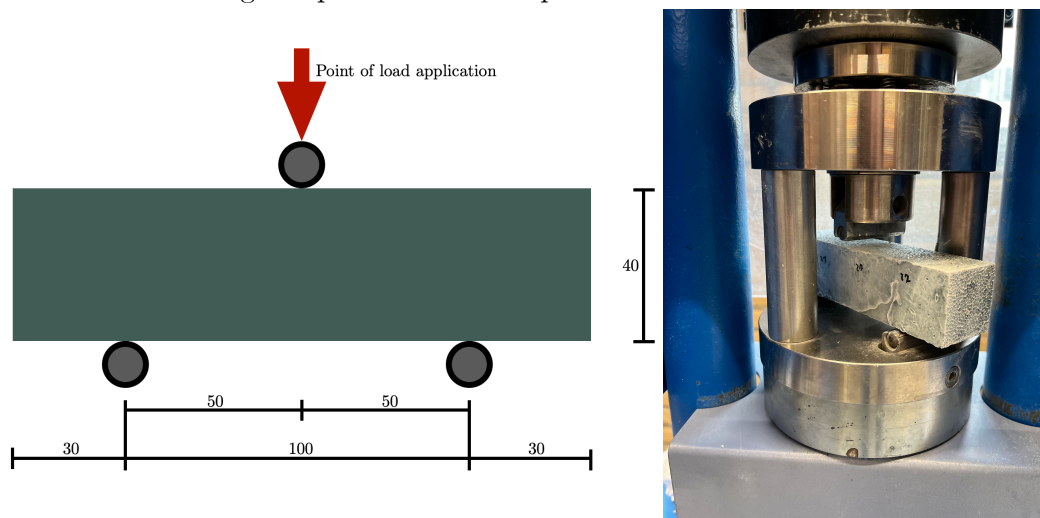


Figure H.3 - Testing setup for flexural strength of alkali activated paste

As can be seen, the testing setup consists of two supporting steel rollers which are spaced 100mm apart. A third steel roller is located at the top with identical dimensions as the bottom two rollers. The vertical plane through the axes of the three rollers are identical. The top roller is capable of slight tilting so the load is distributed evenly, preventing introduction of torsional stress.

Process

This test requires cured alkali activated paste prisms, with dimensions 40x40x160mm. In this phase, specimens which are cured for 1, 7 and 28 days are used, 2 per mix design. The specimens are subjected to a starting load of 0.1kN with an increase of 0.1kN/s. The stopping load range is set at 20%. The following steps are performed:

1. Gather all specimens that should be tested from the curing room.
2. Wipe the bearing surfaces of the rollers and sides of the specimen to remove any loose material.
3. Place the specimen with the casting face sideways
4. Apply loading until failure
5. Determine flexural strength in kN and MPa by readout from the machine. Dimensions of the specimens are already standard in the machine, so the machine automatically determines flexural strength in MPa.
6. Put results in table and graph.
7. Interpretation of results and discussion.

Compressive strength

The used method

Compressive strength is a great parameter to describe the strength of the concrete and it is used through this whole research. As for paste compressive strength, the method as described in NEN-EN 1015-11:2019 is used, but adapted to paste as it originally is a method for mortar.

Scope

Determination of strength of alkali activated paste after 1, 7 and 28 days of curing.

Test setup figures

Figure H.4 shows the testing setup used for this experiment.

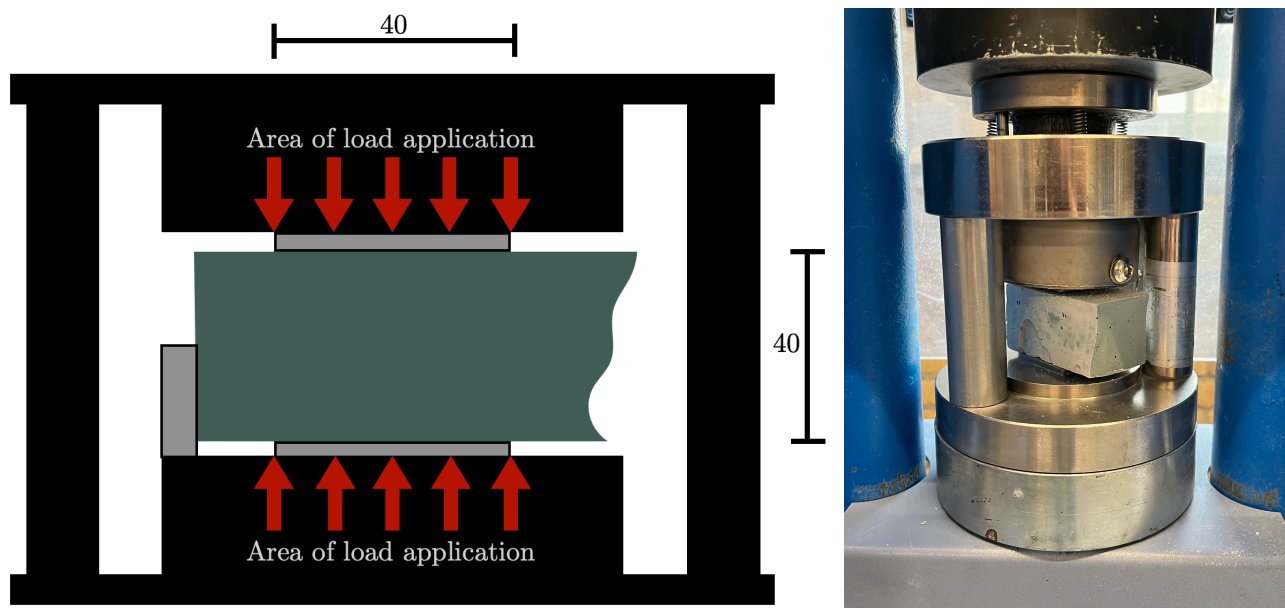


Figure H.4 - Testing setup for compressive strength of alkali activated paste

As can be seen in Figure H.4 the testing setup consists of two plates in between which the specimen is placed. To properly position the specimen, some small steel cylinders are located around the two plates. The load is applied as an area load on both plates. The upper plate is the only moving part, but the load is distributed over the two plates.

Process

The test specimens that are used are 80x40x40mm in size as they are re-used from the flexural strength test, which splits specimens in half. However, the effective tested area of the paste is 40x40mm. The following steps should be performed for measuring compressive strength:

1. Specimens should be tested within 10h from removal from curing room and testing facility should be between 15-25 degrees Celsius. First test flexural strength, re-use those specimens for this compressive strength test.
2. Put specimen in testing machine.
3. Turn on machine and test with constant loading rate of 2.4 kN/s and starting load of 1.0kN. Apply load without shock and increase until failure.
4. Write down failure load (F, in kN).
5. Determine compressive strength using: $f_c = \frac{F}{A_c}$ where A_c is the area that is exposed to compressing (40x40mm).
6. Put results in table and graph.
7. Interpretation of results and discussion.

Mortar phase experiments

Workability

The used method

Workability is very important when it comes to earth moist concrete. In the end, the mixture should have workability which complies with earth moist concrete, namely consistency class C1. To determine the workability for the alkaline activated mortar, the mortar procedure as described in NEN-EN 1015-3 is used.

Scope

Determination of the workability of alkali activated mortar mix designs

Test setup figures

Figure H.5 shows the testing setup used for this experiment.

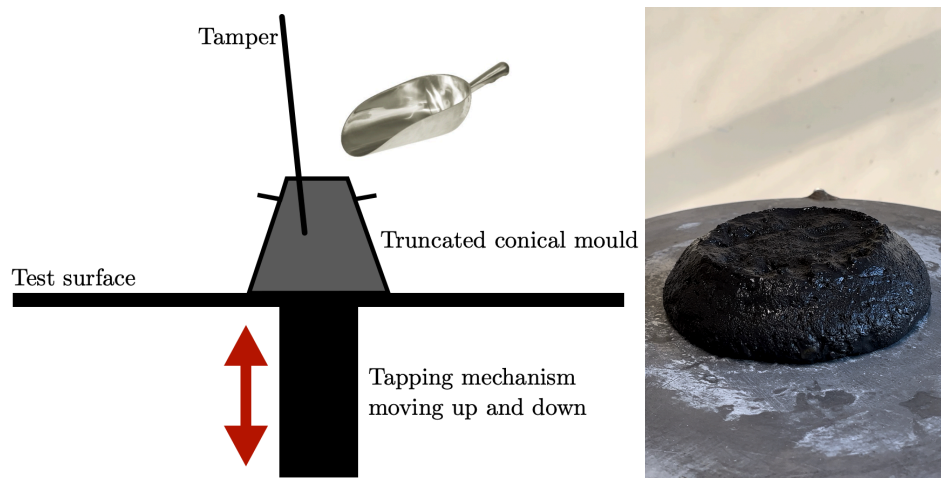


Figure H.5 - Testing setup of workability test of alkali activated mortar

The testing setup consists of a test surface with truncated conical mould on top. The test surface is connected to a tapping mechanism, which moves the test surface and anything on top up and down. An example of the end result after tapping can be seen on the right of Figure H.5.

Process

This test requires a ready mixed alkali activated mortar, around 1.5 liter is sufficient. In order to determine the workability of the alkali activated mortar, this experiment is conducted. The test consists of a small inverted cone and a table with dropping function. The workability is measured in terms of the spread of the mix after 15 drops of the table. The following steps are conducted:

1. Preparation of the alkali activated mortar mix.
2. Make the flat surface of the test bench moist, as well as the inside of the truncated conical mould.
3. Pour the mortar until halfway in the cone and tap it with a tamper so it spreads evenly.
4. Fill the cone completely and tap it with the tamper again. Remove excess material by moving the tamper in a sawing, horizontal movement at the top of the truncated conical mould.
5. After 15s, remove the truncated conical mould vertically in an even manner.
6. Let the table drop for 15 times at an approximate rate of 1 drop/s.
7. Measure the diameter of the spread-out mortar in two perpendicular directions.
8. Determine the workability by calculating the mean value of the two measurements.
9. Put results in table.
10. Interpretation of results and discussion.

Flexural strength

The used method

Flexural strength of concrete is important as this introduces tensile stress in the concrete. Concrete generally has a flexural strength capacity which is only 10% of its compressive strength capacity. To determine flexural strength, the mortar norm is followed. Thus, flexural strength of mortar specimens is tested using NEN-EN 1015-11:2019.

Scope

Determination of the flexural strength of alkali activated mortar mix designs and the trend of mix design parameters to flexural strength development.

Test setup figures

Figure H.6 shows the testing setup used for this experiment.

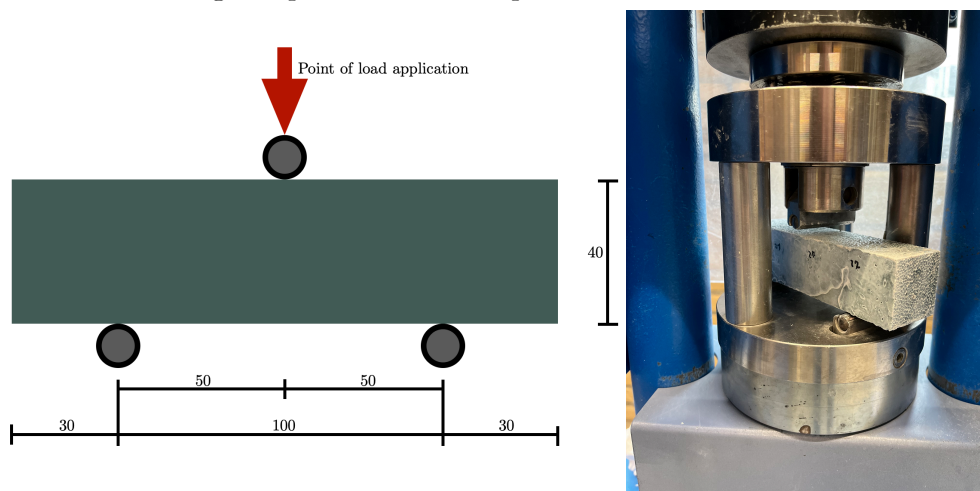


Figure H.6 - Testing setup for flexural strength of alkali activated mortar

As can be seen, the testing setup consists of two supporting steel rollers which are spaced 100mm apart. A third steel roller is located at the top with identical dimensions as the bottom two rollers. The vertical plane through the axes of the three rollers are identical. The top roller is capable of slight tilting so the load is distributed evenly, preventing introduction of torsional stress.

Process

This test requires cured alkali activated mortar prisms, with dimensions 40x40x160mm. In this phase, specimens which are cured for 1, 7 and 28 days are used, 2 per mix design. The specimens are subjected to a starting load of 0.1kN with an increase of 0.1kN/s. The stopping load range is set at 20%. The following steps are performed:

1. Gather all specimens that should be tested from the curing room.
2. Wipe the bearing surfaces of the rollers and sides of the specimen to remove any loose material.
3. Place the specimen with the casting face sideways
4. Apply loading until failure
5. Determine flexural strength in kN and MPa by readout from the machine. Dimensions of the specimens are already standard in the machine, so the machine automatically determines flexural strength in MPa.
6. Put results in table and graph.
7. Interpretation of results and discussion.

Compressive strength

The used method

Compressive strength is a great parameter to describe the strength of the concrete and it is used through this whole research. As for mortar compressive strength, the method as described in NEN-EN 1015-11:2019 is used.

Scope

Determination of strength of alkali activated mortar after 1, 7 and 28 days of curing and the trend of mix design parameters to compressive strength development.

Test setup figures

Figure H.7 shows the testing setup used for this experiment.

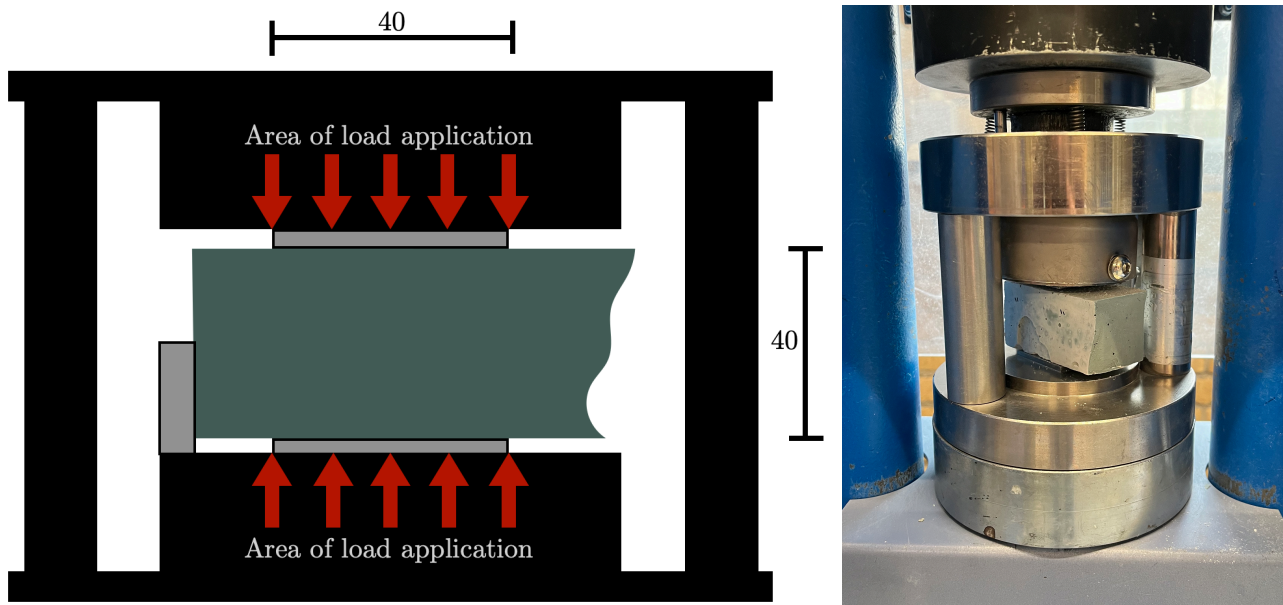


Figure H.7 - Testing setup for compressive strength of alkali activated mortar

As can be seen in Figure H.7 the testing setup consists of two plates in between which the specimen is placed. To properly position the specimen, some small steel cylinders are located around the two plates. The load is applied as an area load on both plates. The upper plate is the only moving part, but the load is distributed over the two plates.

Process

The test specimens that are used are 80x40x40mm in size as they are re-used from the flexural strength test, which splits specimens in half. However, the effective tested area of the concrete is 40x40mm. The following steps should be performed for measuring compressive strength:

1. Specimens should be tested within 10h from removal from curing room and testing facility should be between 15-25 degrees Celsius. First test flexural strength, re-use those specimens for this compressive strength test.
2. Put specimen in testing machine.
3. Turn on machine and test with constant loading rate of 2.4 kN/s and starting load of 1.0kN. Apply load without shock and increase until failure.
4. Write down failure load (F, in kN).
5. Determine compressive strength using: $f_c = \frac{F}{A_c}$ where A_c is the area that is exposed to compressing (40x40mm).
6. Put results in table and graph.
7. Interpretation of results and discussion.

Water absorption

The used method

Water absorption can be used to explain the deterioration mechanisms due to freezing and thawing. It also allows to couple the theory of microstructure formation and reaction kinetics towards the trends of mix design parameters, resulting in finding the origin of why certain mix designs or combinations of parameters result in poor or great freezing and thawing resistance for alkaline activated mortar.

Water absorption is determined using ASTM-C642.

Scope

Determination of water absorption and voids content for coupling theory to deterioration mechanisms of alkali activated mortar.

Testing setup figure

Figure H.8 below shows the testing setup used for this experiment.

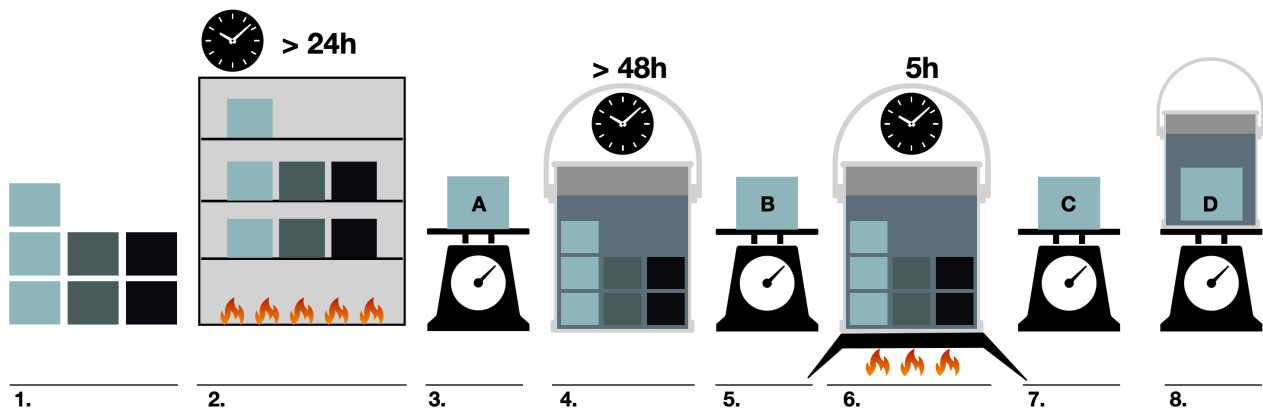


Figure H.8 - Testing setup for water absorption test

As can be seen in Figure H.8, the testing setup has a large amount of different steps, but are relatively simple to execute as described in the process steps below.

Process

1. Prepare specimens with a volume of at least 350 cm^3 . After casting, put them in the curing room for 7 days.
2. Determine the mass of the specimens and put them in an oven with temperature of $100\text{-}110^\circ\text{C}$ for not less than 24h. After this period of time, remove specimens from oven and allow specimens to cool in air with a temperature of $20\text{-}25^\circ\text{C}$.
3. Weigh the specimens. If mass difference is $<0.5\%$ between previous measurement, note the weight as (A). Otherwise, put in the oven once again for 24h.
4. Immerse specimens in water at approximately 21°C for at least 48 hours.
5. Surface dry the specimens with a moist towel and weigh the specimens. If mass difference is $<0.5\%$ between previous measurement, note the weight as (B). Otherwise, put in the water once again for 24h.
6. Place the immersed specimens on a stove and boil for 5 hours. Allow the specimens to cool for at least 14 hours afterwards.
7. After cooling, remove surface moisture with a moist towel and determine mass of specimen. Note the weight as (C).
8. Suspend the specimen by a wire and determine apparent mass in water, note the weight as (D).
9. Determine the following values after testing:

$$\text{Absorption after immersion: } \frac{B - A}{A} \cdot 100 (\%)$$

$$\text{Absorption after immersion and boiling: } \frac{C - A}{A} \cdot 100 (\%)$$

$$\text{Volume of permeable pore space (voids): } \frac{g_2 - g_1}{g_2} \cdot 100 (\%) \text{ with:}$$

$$g_1 = \frac{A}{C - D} \cdot \rho_w, \quad g_2 = \frac{A}{A - D} \cdot \rho_w$$

10. Put results in table or graph
11. Interpretation of results and discussion.

Surface water absorption

The used method

Besides the total water absorption, the surface water absorption is of importance for freezing and thawing resistance as well. If surface sorption is high, this means specimens are likely to absorb fluids quickly and that the surface is permeable. When the fluid freezes, it expands and this is one of the possible mechanisms that explains scaling of specimens. For this experiment, no specific standard is followed as it is quite straight forward.

Scope

Determine the surface sorptivity of mortar specimens.

Test setup figure

Figure H.9 below provides an overview of the testing setup used for this experiment.

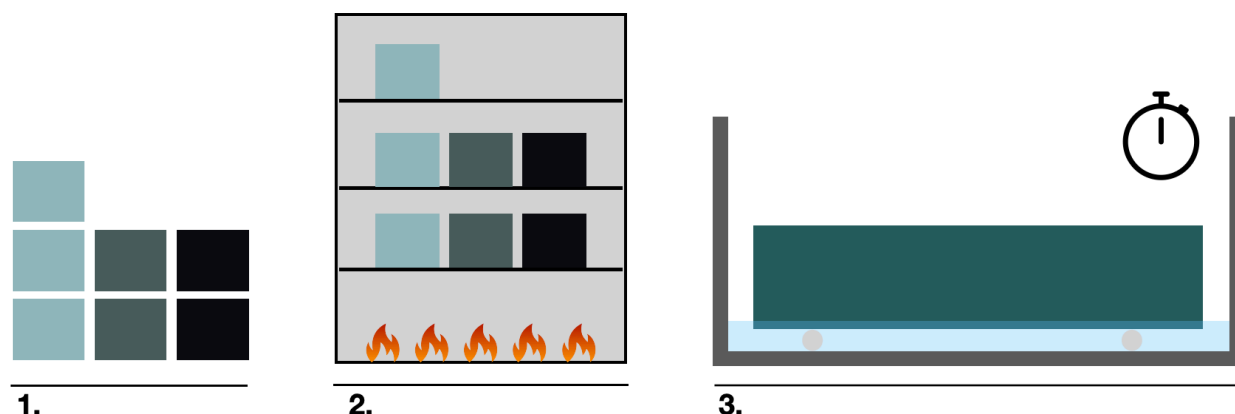


Figure H.9 - Surface absorption testing setup

The test setup consists of casting, drying stage and a measurement stage. The procedural steps are elaborated on the next page.

Process

1. Cast specimens and cure for 7 days.
2. Put specimens in oven at a temperature of 100-110 degrees Celsius until the difference in weight between two 24h intervals is less than 0.5%. Allow the specimens to cool to room temperature and then note the weight.
3. Prepare a bucket with two spacers on top of which the specimen is placed. Carefully add water until the specimen is situated in 5mm water. Start the timer. After 5, 10, 15, 20, 30, 40, 50 and 60 minutes, get specimen out of bucket, surface dry with a moist towel and note the weight. Afterwards place the specimen back in the bucket and check if the amount of water is still sufficient. Repeat until the last measurement point.
4. Put results in table and graph.
5. Interpretation of results

Freeze-thaw resistance

The used method

Concrete exposed to freezing and thawing needs to be durable in order to have sufficient resistance, especially in a sea-environment where concrete revetment products are often situated in. To examine the trend in degradation of concrete due to freeze-thaw cycles, a combination of mass loss and compressive strength is used. The freeze-thaw procedure followed is a modified version of the standardized CDF test and the cube test as described in NVN-CEN/TS 12390-9:2016, as no specific norm applies for mortars.

Scope

Determination of effects of mix design parameters on freeze-thaw resistance of alkali activated mortar in order to evaluate trends in freeze-thaw resistance so that final concrete-scale mix designs can be created from these trends.

Test setup figure

Figure H.10 below shows the testing setup used for this experiment.

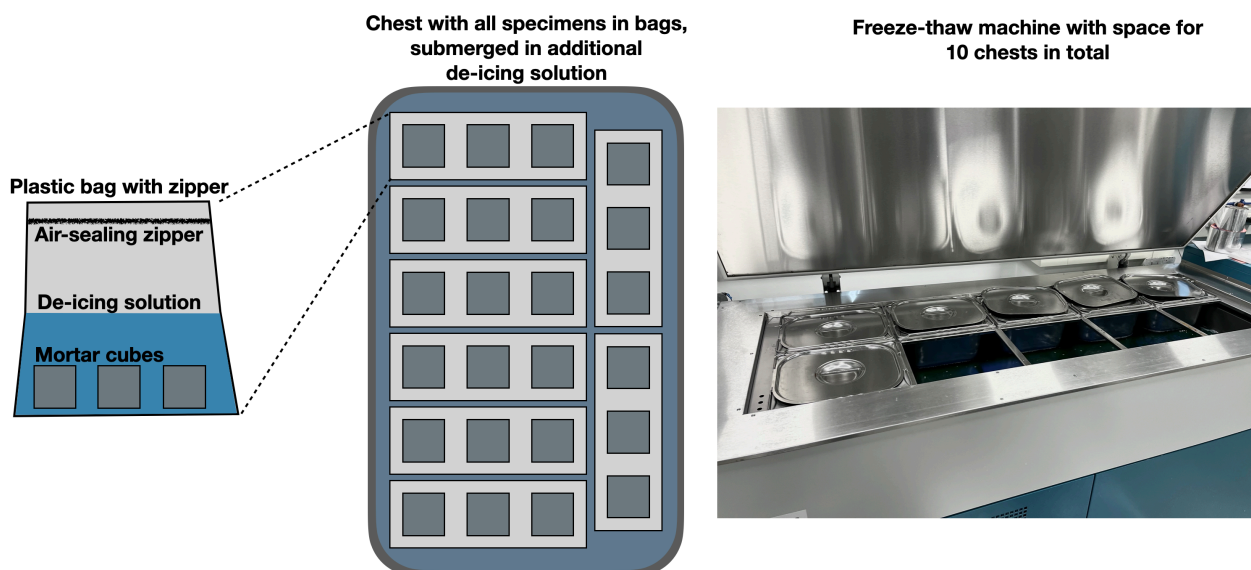


Figure H.10 - Testing setup for Freeze-thaw resistance of alkali activated mortar

As can be seen in Figure H.10, the testing setup consists of three parts:

1. The plastic bag which is freeze-resistant and includes a zipper for sealing the bag, preventing evaporation. This plastic bag contains three mortar cubes which are submerged in de-icing solution.
2. The freeze-thaw chest in accordance with the CDF test as described in NVN-CEN/TS 12390-9:2016, containing the 8 mixtures that are subjected to freezing and thawing. Around the bags, additional de-icing solution is added for better heat conduction.
3. The total freeze-thaw machine, containing 10 chests in total.

Process

The regular CDF test requires 150mm cubes of concrete to be partially submerged in water with or without de-icing salts after which the cycles are performed. However, since this part of the research focuses on mortar scale, making such huge cubes would both consume a lot of material and take up a lot of space. Besides, the goal of the freeze-thaw tests in mortar scale is to determine effects of mix design parameters on freeze-thaw resistance of alkali activated mortar in order to evaluate trends in freeze-thaw resistance. As no norm for mortar is available, the following process is used:

By casting 40x40x40mm cubes and submerging them completely in the de-icing fluid, the trend of freeze-thaw performance should quickly be visible. In total there will be four sets of cubes, which vary in their amount of cycles exposed to freezing and thawing: 2 cycles, 6 cycles, 14 cycles and 28 cycles. To further elaborate these trends, a reference mixture which is un-exposed to freezing and thawing is used.

A temperature controlled chest as described in NVN-CEN/TS 12390-9:2016 is used combined with the following procedural steps:

1. Cast the prisms and store them for 24h with foil on top to prevent evaporation
2. After 24h, remove foil from prisms, demould prisms and place them in the curing room.
3. After 3 days in the curing room, saw the prisms into 40x40x40mm cubes and completely immerse them in water to ensure full saturation. Put the cubes, immersed, in the curing room.

4. After another 3 days, remove the cubes from the curing room and put them in labelled plastic bags which can be sealed to prevent evaporation.
5. Add 3% NaCl solution in the plastic bags until cubes are completely submerged.
6. Put the bags in the temperature controlled chest, fill the chest with de-icing solution and put the chest in the freeze-thawing machine.

The procedure is different for different durations of freeze-thaw. For all x cycle specimens, the goal is to identify trends in freeze-thaw performance via compressive strength and to determine the scaled material. Therefore, the following procedure is used after x cycles of freezing and thawing:

Procedure (following steps 1-6)

7. After 2, 6, 14 or 28 cycles in the freeze-thaw machine, remove the specimens from the testing setup.
8. Put specimens in ultrasonic bath and subject for ultrasonic cleaning for three minutes.
9. Open the bag and remove the cubes. The cubes are to be brought to saturated surface dry state by using drying towels.
10. Collect scaled material in filter paper using a cut in the plastic bag. Rinse several times with water until all scaled material is collected in the filter. Dry the filters with scaled materials at 110 degrees Celsius for 24h.
11. Weigh the specimens in saturated surface dry state and note result.
12. Test the freeze-thaw specimens for compressive strength.
13. Test the reference specimens for compressive strength.
14. After 24h, remove the dried filters and weigh them. Scaled material can now be determined and can also be validated using the two prior weight measurements (before FT in surface saturated dry state and after FT in surface saturated dry state).
15. Put results in table and graph.
16. Interpretation of results and discussion.

Note:

Water absorption and Freeze-Thaw results are combined in chapter 5, as the combined results correlate and strive to couple theory towards research results.

Sulfate attack resistance

The used method

Sulfate attack is a very important durability parameter as it greatly affects concrete properties. To investigate degradation due to sulfate attack, a combination of mass loss, compressive strength and internal cracking using SEM is used. Sulfate attack is tested by using MgSO_4 in a high 10% solution concentration. This is specifically chosen as it affects alkali activated concrete more. A high concentration is chosen to enable accelerated testing, as sulfate attack tests can continue for months. A modified testing method as described in NPR-CEN/TR 15697:2008 is used.

Scope

Determination of effects of mix design parameters on sulfate attack resistance of alkali activated mortar in order to evaluate trends in sulfate-attack resistance so that final concrete-scale mix designs can be created keeping these trends in mind.

Test setup figure

Figure H.11 below shows the testing setup used for this experiment.

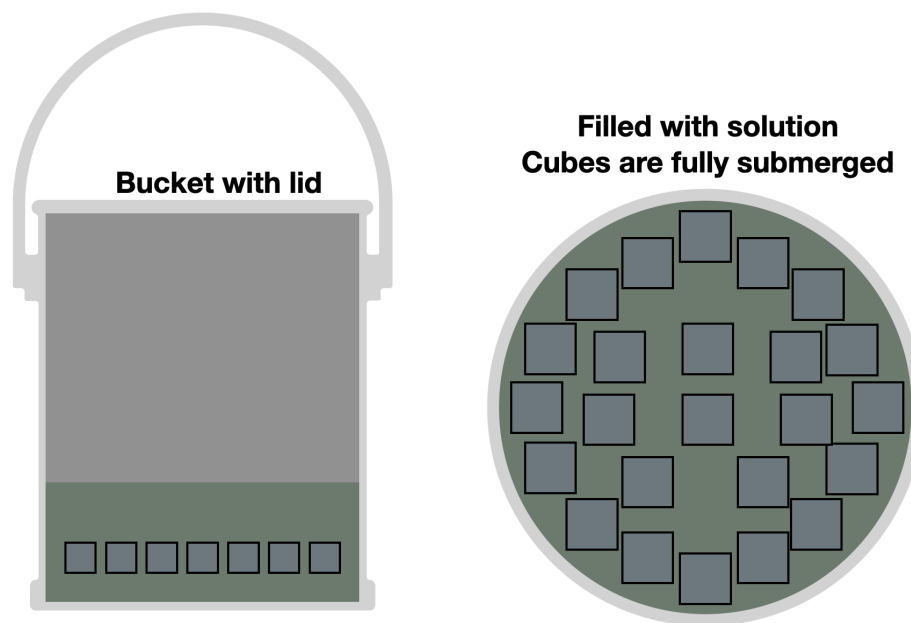


Figure H.11 - Testing setup for sulfate attack resistance of alkali activated mortar

Process

By casting 4x4x4 cm³ cubes and submerging them completely in the MgSO₄ solution, the trend of sulfate-attack performance should quickly be visible. In total there will be four sets of cubes, which vary in their amount of days exposed to the solution: 1, 3, 7 and 14 days. To further elaborate these trends, a reference mixture which is un-exposed to sulfate attack is used.

The following procedural steps are performed:

1. Cast the prisms and store them for 24h with foil on top to prevent evaporation
2. After 24h, remove foil from prisms, unmold prisms and place them in the curing room.
3. After 3 days in the curing room, saw the prisms into 40x40x40mm cubes and completely immerse them in water to ensure full saturation. Put the cubes, immersed, in the curing room.
4. After 7 days, remove cubes from water bath and dry (surface dry) and weigh them.
5. Put the cubes in the bucket with spacers underneath. Fill the bucket with MgSO₄ solution until the cubes are completely submerged.
6. Run SA for 1, 3, 7 and 14 days.
7. Once removed, dry cubes until the same surface dry state and weigh them, with any loose material removed.
8. Determine mass loss due to Sulfate attack.
9. Determine compressive strength of specimens
10. Put results in table and graph
11. Interpretation of results and discussion.

Concrete phase experiments

Workability

The used method

Workability is very important for dry-cast concrete. If the concrete is flowing a lot, it means that after remolding the concrete will sag causing deformation in the final products or complete failure of product production.

To examine workability for earth moist concrete, according to NEN-EN206+NEN8005:2017 the measure of compaction as described in EN12350-4 should be used.

Scope

Determining the level of compaction of concrete in order to validate its consistency and workability is sufficient for dry-cast production.

Requirement

For earth moist concrete, the degree of compatibility should be between 1.26-1.45.

Testing setup figure

Figure H.12 below shows the test setup for determination of workability.



Figure H.12 - Workability test setup

A compaction container, also known as “het vat van Waltz” in Dutch, a vibration table and measurement tape is needed to execute the workability test.

Process

A compaction container with dimensions 200x200x400mm (LxWxH) is needed along with a Trowel and vibrating table. The following steps should be performed:

1. Clean the compaction container and moisten inner surfaces using a moist cloth.
2. Fill the container with the trowel, emptying the trowel once at all four corners of the compaction container.
3. Remove concrete from the upper edges with a straight edged scraper with a sawing action.
4. Compact using a vibration table until no further compaction is observable.
5. Determine internal height of the container (h) and the mean value of all four edges of the top of the concrete until the top of the compaction container (s).
6. Determine degree of compatibility c with: $c = \frac{h}{h - s}$ and report the result to the nearest 0,01.
7. Put results in table

8. Interpretation of results and discussion.

Water absorption

The used method

Water absorption can be connected to FT resistance as internal expanding water during a period of frost can cause failure to concrete. Water absorption is also relevant for concrete mix design and is therefore a very important test to be performed. Water absorption is tested in accordance with NEN-EN1338:2003, appendix E.

Scope

Determining water absorption of alkali activated concrete and blast furnace cement concrete.

Requirement

Water absorption of the specimens should be kept below 6%.

Testing setup figure

Figure H.13 below shows the test setup for water absorption.

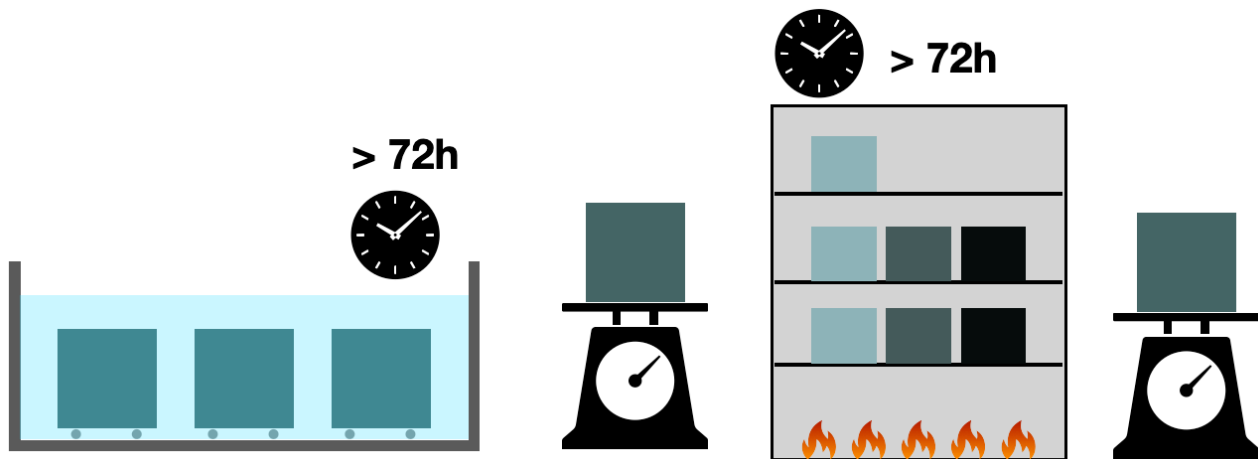


Figure H.13 - Water absorption test setup

The test setup is very straight forward as it only consists of a suitable large tray filled with water, a scale to measure the weight of the specimens and an oven to dry the specimens.

Process

For this experiment NEN-EN1338:2003, appendix E is used. The method requires a block with weight of maximum 5.0kg. Due to requirements from NEN-EN 7024-2, the specimen needs to be drilled from the 150x150x150mm cube. The desired dimensions is a 100mm diameter cylinder with a 100mm height, where the top of the cylinder is also the casting face of the cube.

The following steps should then be carried out:

1. Remove all dust with a brush and ensure a specimen temperature of 15-25 degrees Celsius.
2. Immerse specimens in water at temperature of 15-25 degrees Celsius where the minimum immersion period should be 3 days, spacing should be minimum 15mm and 20mm of water should at least be present on top of the specimens.
3. Measure the weight each 24h, constant weight is reached when the difference in mass is less than 0.1%.
4. Before weighing, wipe the specimen with a slight moist towel to remove any excess water. The concrete surface should be dull. Then weigh and note down (M_1).
5. Place each specimen in an oven with at least 15mm spacing and dry at 100-110 degrees Celsius for at least three days until constant mass.
6. Weigh specimen and note down (M_2).

7. Determine water absorption using: $W_a = \frac{M_1 - M_2}{M_2} \cdot 100 \%$
8. Put results in table.
9. Interpretation of results and discussion.

Freeze-thaw resistance

The used method

For the concrete freeze-thaw resistance annex D of EN 1338:2003(E) will be followed as closely as the available test equipment allows, in accordance with the list of requirements. This method features a preconditioned specimen which is subjected to 28 freeze-thaw cycles with a surface covered in 3% NaCl solution. Mass loss is determined, which should be $\leq 1.0 \text{ kg/m}^2$ with no individual values $> 1.5 \text{ kg/m}^2$ after 28 FT cycles.

Scope

Determine Freeze-thaw resistance of alkali activated concrete and reference concrete by means of measuring mass loss (scaling) according to the CDF method.

Testing setup figure

Figure H.14 below shows the test setup for determination of freeze-thaw resistance.

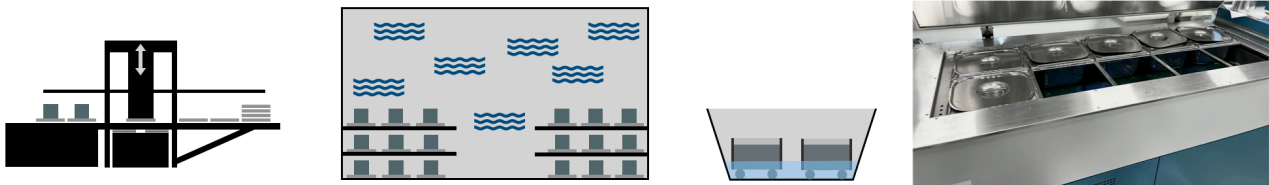


Figure H.14 - Testing setup for freeze-thaw resistance (from left to right: casting, curing, pre-saturation and freeze-thaw testing machine)

Requirement

Mass loss must be $\leq 1.0 \text{ kg/m}^2$ with no individual values $> 1.5 \text{ kg/m}^2$ after 28 FT cycles.

Process

For this experiment, a specimen with upper face area of 7500 mm^2 - 25000 mm^2 shall be used with a maximum thickness of 103mm. Therefore, the $150 \times 150 \times 150 \text{ mm}$ cubes are sawn into half such that specimens with size $150 \times 150 \times 75 \text{ mm}$ are used. The following steps should be taken:

1. Cast concrete and cure submerged in water of 20 degrees Celsius for 7 days.
2. Remove specimens and cure specimens for 21 days at ambient temperature and relative humidity of 55-75 %. 21-26 days after casting, the specimens should be wrapped with butyl tape to insulate the sides.
3. 28 days after casting, the specimens are weighed and put inside a platform where they are put on 5mm spacers and submerged in 10mm NaCl solution and covered. They need to be submerged for 7 days and monitored daily (add solution if needed, weigh specimens).
4. Put the specimen upside down with spacers in the Freeze-thaw machine with 10mm 3 % NaCl solution.
5. Perform 28 cycles, where after 7 and 14 cycles more solution should be added if needed during the thaw period.
6. After 4, 6, 14 and 28 cycles collect scaled material by brushing it off the concrete and exposing concrete to ultrasonic vibrations and dry the material for at least 24 h and determine its mass.
7. Determine mass loss per unit area using $L = \frac{M}{A}$.
8. Put results in table/graph.

9. Interpretation of results and discussion.

Compressive strength

The used method

Compressive strength is a great parameter to describe the strength of the concrete and it is used through this whole research. As for concrete compressive strength, the method as described in NEN-EN 12390-3:2019(E) is used.

Scope

Determination of strength of concrete after 1, 7 and 28 days curing

Testing setup figure

Figure H.15 below shows the test setup for compressive strength.

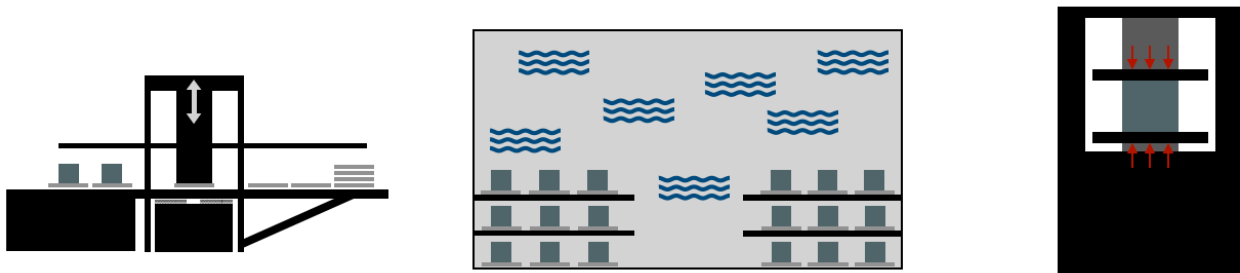


Figure H.15 - Testing setup for compressive strength (from left to right: casting, curing and compressive strength testing machine)

Requirement

Compressive strength should be ≥ 12 MPa after 1 day curing and ≥ 37 MPa after 28 days curing.

Process

The test specimens that are used are 150 x 150 x 150 mm in size, in accordance with EN12390-1. The following steps should be performed for measuring compressive strength:

1. Specimens should be tested within 10h from removal from curing room and testing facility should be between 15-25 degrees Celsius.
2. Put specimen in testing machine with label pointed towards person.
3. Turn on machine and test with constant loading rate of 13.5 kN/s.
4. Write down failure load (F, in kN).
5. Determine compressive strength using: $f_c = \frac{F}{A_c}$ where A_c is the area that is exposed to compressing (150 x 150 mm).
6. Put results in table and graph.
7. Interpretation of results and discussion.

Additional LCA data

The process tree data is enlarged in the following figures:

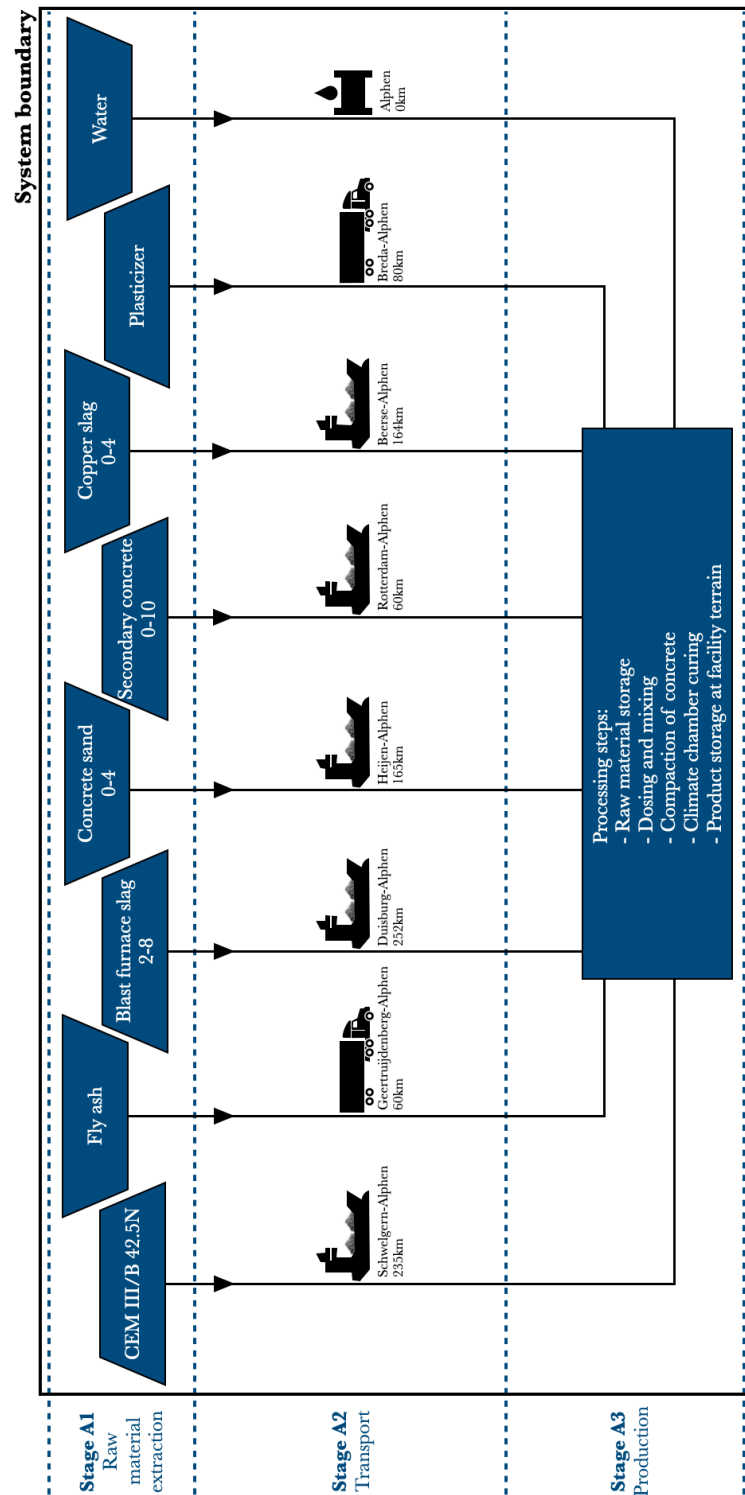


Figure I.1 - Process tree reference mixture

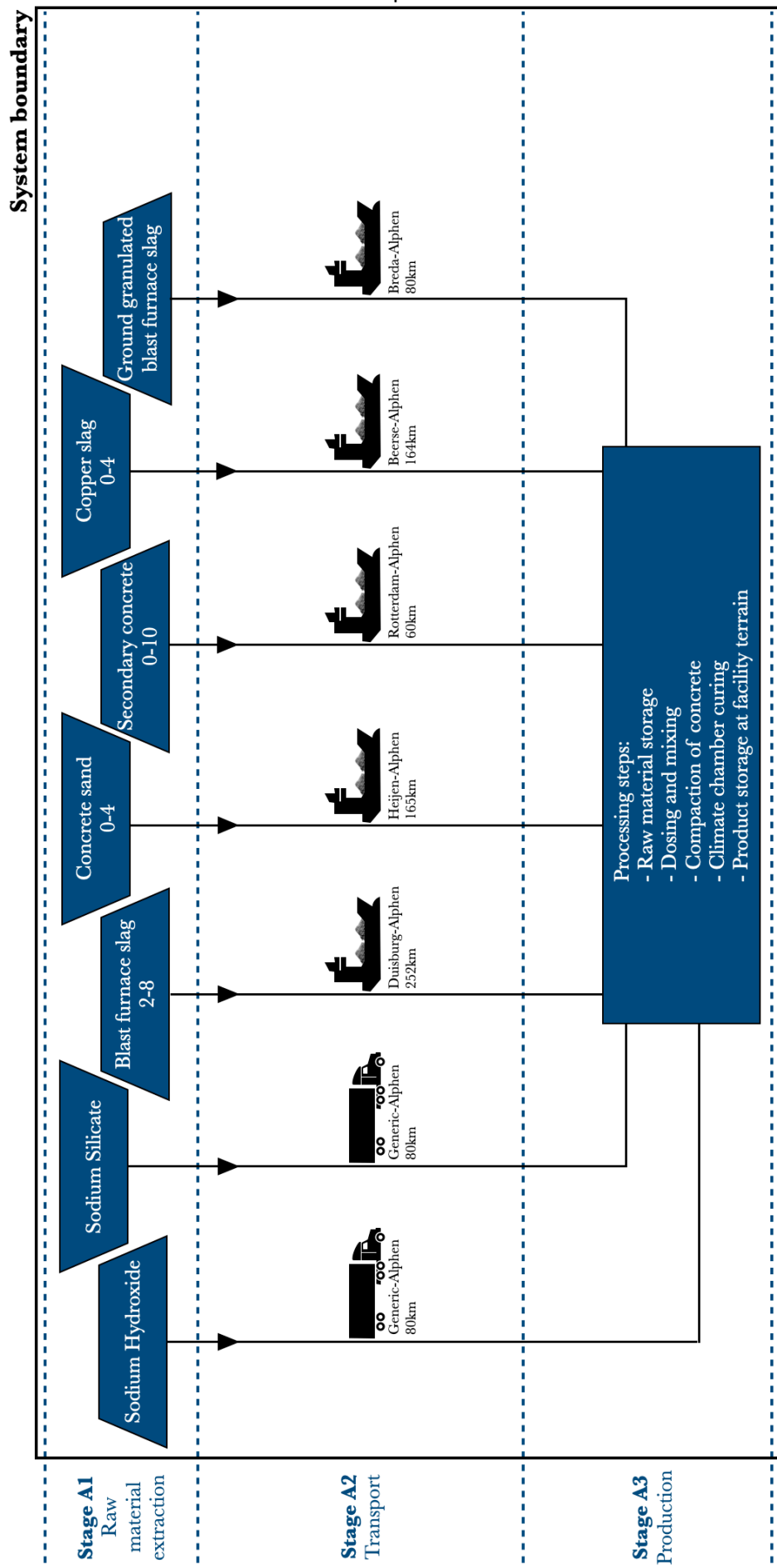


Figure I.2 - Process tree low MKI mixture

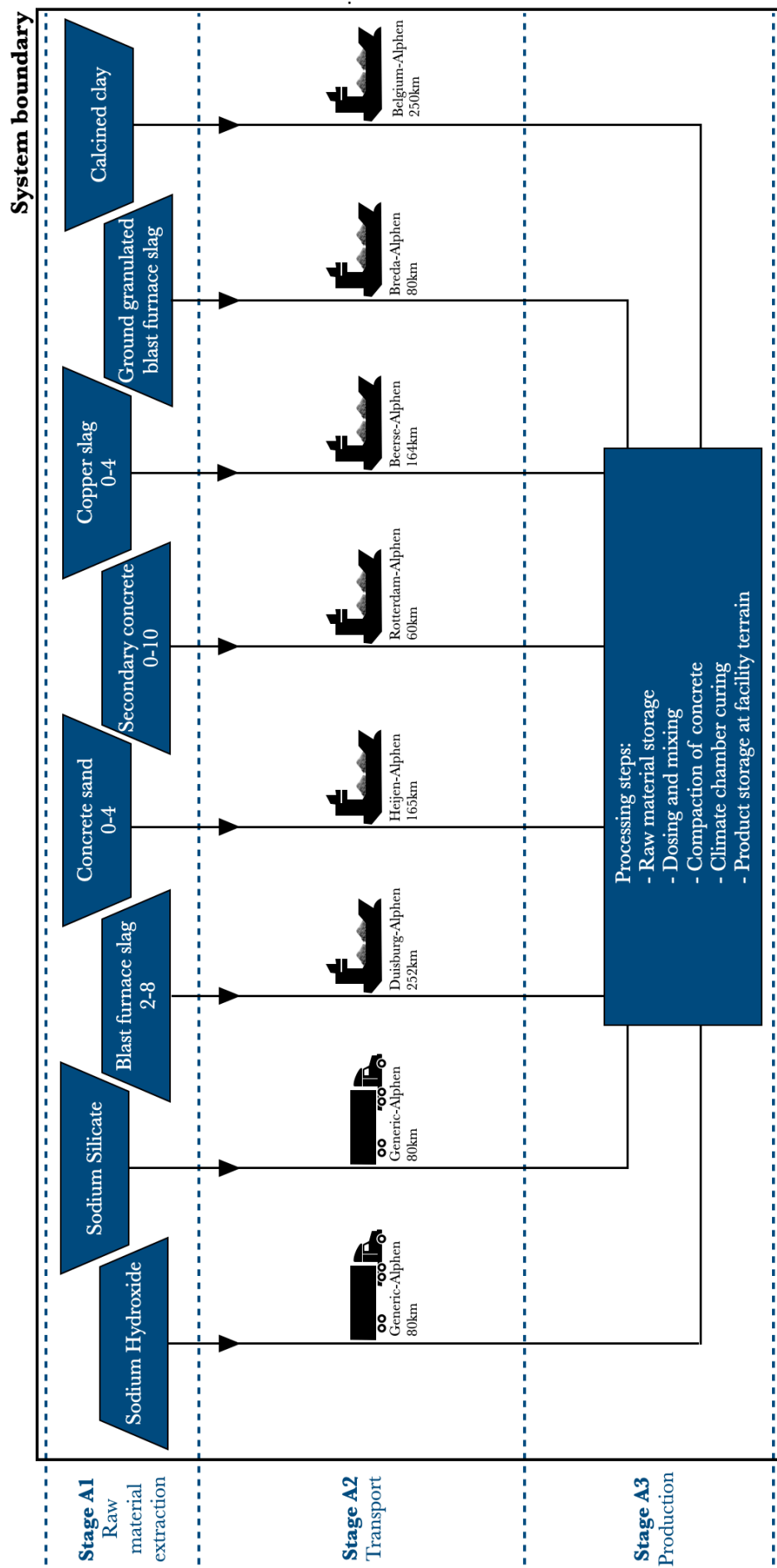


Figure I.3 - Process tree performance mixture

The quality of used data is presented in the Table below:

Horizontally aggregated processes					
To be evaluated	All inputs and outputs (economical streams, with the exception of the product, and environmental interventions) of a group process; or the typing of a group process in relation to the LCA where it is used				
Applicable at	A process that is represented as 'average' of a comparable process of different production locations; or assessment of process data of a group when used in a LCA.				
Indicator	Pedigree score	1	2	3	4
Completeness					
Completeness environmental interventions	All environmental interventions from the LCA-2 list have a value	All environmental interventions that reasonably could be expected, have a value	Some interventions are not included which could have been expected, but which are expected to be less relevant for the environmental profile of the process	Interventions are missing which could have been expected, which are expected to be relevant for the environmental profile of the process or from which beforehand cannot be assessed if they are relevant or not	Missing interventions unknown
Example	The value can be zero. The value may be put to zero if elaborated.				
Completeness economical streams	All streams are qualified and quantified	All streams are qualified. Streams that are expected to be relevant for the environmental profile of the process are quantified.	All streams are qualified. The largest of the material- and energy streams are quantified.	The economical streams for which data was available are quantified.	The completeness of economical streams is unclear/unknown
Example	Streams = raw materials, energy, emissions, waste. (e.g. each additive is named and the amount used as well)	e.g. Additives that look like the main materials are not quantified (water emission not quantified)			
Mass balance on process level	Closure >95%	Closure 90-95%	Closure 80-90%	Closure 70-80%	Closure <70% or unknown
Example	Mass balance = total mass ingoing raw materials with respect to the summation of products + emissions + waste				
Mass balance on company level	Of the companies that determine >80% of production volume, the mass balance per company is >95% enclosed	Of the companies that determine >80% of production volume, the mass balance per company is >90% enclosed	Of the companies that determine >80% of production volume, the mass balance per company is >80% enclosed	Of the companies that determine >80% of production volume, the mass balance per company is >70% enclosed	Of the companies that determine >80% of production volume, the mass balance per company is <70% enclosed or unknown

Geographical representivity	The area covered by the group is directly related to the desired area	The area covered by the group is larger than the desired area	The area covered by the group knows equal production conditions as the desired area	The area covered by the group knows partial equal production conditions	The area covered by the group consists of completely different production conditons/geographical representativity unknown
Example		West-European data, used in the Netherlands	Data from products produces in the Netherlands, for which German data is used.		
Completeness number of locations/technological representativity	All companies in the group delivered data	Representative cut of the group with regards to technological differences. Differences are well represented in averages.	Average of group that represents technological differences	Random average from the group	Technological differences not taken into account
Technological representativity	Data from companies, processes and product of the study	Data from proces/product of the study, but from another company than the group represents	Data from process/product from study, but another technology	Data from comparable processes/products, but same technology	Data from comparable processes and materials, but different technology
Example		German rocks for which data from the Netherlands are used	For a PVC product data is used from a different PVC-process.		
Consistency and reproductivity					
Uniformity and consistency	The data that determines >80% of the environmental effects are collected with the same methods and accuracy.	The data that determines >80% of the environmental effects are collected with the same methods.	The data that determines >80% of the environmental effects are collected with the same methods and based on the best available and validated data	The data that determines >80% of the environmental effects are based on available data, collected with an equal method	The data that determines >80% of the environmental effects are based on different sources with different accuracies without validation of underlying deviations.

Example	Mass balance = total amount of used raw materials with respect to the total production + waste + emissions (buy/sell, with correction for supplies)				
Energy balance on company level	Of the companies that determine >80% of the production volume, the energy balance is enclosed by >95% per company	Of the companies that determine >80% of the production volume, the energy balance is enclosed by >90% per company	Of the companies that determine >80% of the production volume, the energy balance is enclosed by >80% per company	Of the companies that determine >80% of the production volume, the energy balance is enclosed by >70% per company	Of the companies that determine >80% of the production volume, the energy balance is enclosed by <70% per company or unknown
Example	Sum of energy usage of individual processes with respect to energy bill				
Representativity					
Time bound representativity of process with respect to year of evaluation	<2 year difference; or (choose best option): All underlying processes are suitable for the period that is studied in the LCA	2-5 year difference; or (choose best option): One of the underlying processes is changed on details. This leads to an approximate change of less than 5% in the average raw material streams.	5-10 year difference; or (choose best option): Part of the underlying processes is changed on details. This leads to an approximate change of less than 5-20% in the average raw material streams.	10-15 year difference; or (choose best option): Most of the underlying processes is changed on details. This leads to an approximate change of >20% in the average raw material streams.	>15 year difference or unknown; or (choose best option): The process is not applied anymore in the studied period, or: the underlying processes are mostly changed. This may lead to differences in raw material streams of >20%
Example	Data are from 1999 and are provided in 2000 to be valid for the period 1999-2001	Data is from 1999 and is provided in 2003			
Completeness amount of locations/geographical representativity	All companies in the group delivered data	Representative cut of the group with regards to geographical differences in streams (e.g. transportation distance, temperature dependency, regulations). Differences are well represented in averages.	Average of group that represents geographical differences	Random average from the group	Geographical differences not taken into account

Uniformity and consistency	The data that determines >80% of the environmental effects are collected with the same methods and accuracy.	The data that determines >80% of the environmental effects are collected with the same methods.	The data that determines >80% of the environmental effects are collected with the same methods and based on the best available and validated data	The data that determines >80% of the environmental effects are based on available data, collected with an equal method	The data that determines >80% of the environmental effects are based on different sources with different accuracies without validation of underlying deviations.
Example	Energy and emission data from the same registration systems.	Energy and emission data based on measurements	Combination of measured and estimated values with elaborated deviations, collected following the same procedure	Companies filled a single questionnaire, but have not investigated mutual differences.	Combination of literature data from different companies from different years with different data
Reproductability for third parties	Fully reproductable	Process description fully quantitative with the used environmental interventions for the processes that determine >80% of the environmental effects.	Process description complete and quantitatively reproductable	Process description qualitative and for the main part not reproductable	Not reproductable

Figure I.4 - Quality of data for all LCA calculations

Note that all data in Figure I.4 is only true in combination with the confidential appendices.

References

- Abed, M.A. & Balázs, G.L. (2021, October 15). Concrete performance in cold regions: understanding concrete's resistance to freezing/thawing cycles. *Sustainability of concrete with synthetic and recycled aggregates*. IntechOpen. <https://doi.org/10.5772/intechopen.99968>
- Alanazi, H., Yang, M., Zhang, D. & Gao, Z.J. (2017, January). Early strength and durability of geopolymer concrete. *Magazine of Concrete Research*, 69, 46-54. <https://doi.org/10.1680/jmacr.16.00118>
- Akbarnezhad, A., Huan, M., Mesgari, S. Castel, A. (2015, October 13). Recycling of geopolymer concrete. *Construction and building materials*, 101, 152-158. <http://dx.doi.org/10.1016/j.conbuildmat.2015.10.037>
- American Society for Testing and Materials. (2022, January 13). *ASTM C642-21 - Standard test method for density, absorption and voids in hardened concrete*.
- Anton Paar. (s.d.). *Mercury Intrusion Porosimetry basics: measuring pores in solids*. <https://wiki.anton-paar.com/en/mercury-intrusion-porosimetry-basics-measuring-pores-in-solids/>
- Assi, L., Carter, K., Deaver, E., Anay, R., & Ziehl, P. (2018, Oktober 10). Sustainable concrete: Building a greener future. *Journal of Cleaner Production*, 198, 1641-1651. <https://doi.org/10.1016/j.jclepro.2018.07.123>
- Aziz, I., Al Bakri Abdullah, M., Mohd Salleh, M., Yoriya, S., Chaiprapa, J., Rojviriya, C., & Li, L. (2020, November 24). Microstructure and porosity evolution of alkali activated slag at various heating temperatures. *Journal of Materials Research and Technology*, 9, 15894-15907. <https://doi.org/10.1016/j.jmrt.2020.11.041>
- Bakharev, T., Sanjayan, J.G. & Cheng, Y. (1998, September 14). Alkali activation of Australian slag cements. *Cement and concrete research*, 29, 113-120. [https://doi.org/10.1016/S0008-8846\(98\)00170-7](https://doi.org/10.1016/S0008-8846(98)00170-7)
- Barabanshchikov, Y., Usanova, K., Akimov, S., & Bily, P. (2020). Low heat concrete with ground granulated blast furnace slag. *IOP Conference series Materials Science and Engineering*, 896(1). <http://dx.doi.org/10.1088/1757-899X/896/1/012098>
- Barral, M. (2019, February 19). *Svante Arrhenius, the Man Who Foresaw Climate Change*. Open mind BBVA. <https://www.bbvaopenmind.com/en/science/leading-figures/svante-arrhenius-the-man-who-foresaw-climate-change/>
- Bature, A.S., Khorami, M., Ganjian, E., Tyrer, M. (2020, November 18). Influence of alkali activator type and proportion on strength performance of calcined clay geopolymer mortar. *Construction and Building Materials*, 267, 120446. <https://doi.org/10.1016/j.conbuildmat.2020.120446>
- Betonakkoord. (s.d.). *Betonakkoord*. Betonakkoord. <https://www.betonakkoord.nl>
- Betonvereniging (s.d.). *Hoofdstuk 2: maken van betonsamenstellingen*. Betonvereniging. <https://maken.wikiwijs.nl/userfiles/a/a532e470135c8d6eb1720f9cbd6a3a0427ff773f.pdf>

- Borges, P.H.R., Banthia, N., Alcamand, H.A., Vasconcelos, W.L., Nunes, E.H.M. (2016, April 22). Performance of blended metakaolin/blastfurnace slag alkali-activated mortars. *Cement and Concrete Composites*, 71, 42-52. <http://dx.doi.org/10.1016/j.cemconcomp.2016.04.008>
- British Standards Institution [BSI]. (1975). *BS 812-105.1:1975 - Methods for determination of particle shape and flakiness index*.
- Cement concrete & Aggregates Australia. (2018, January). *Ground slag properties, characterisation and uses*. Cement concrete & Aggregates Australia. [https://www.ccaa.com.au/documents/TECH_NOTE_78 - Ground_Slag.pdf](https://www.ccaa.com.au/documents/TECH_NOTE_78_-_Ground_Slag.pdf)
- Chaliasou, N.A., Heath, A., Holley, J.C., Paine, K. (2018, May 29). *Effect of recycled geopolymer concrete aggregate on strength development and consistence of Portland cement concretes* [Power Point slides]. Engineering Conferences International. https://dc.engconfintl.org/geopolymers/?utm_source=dc.engconfintl.org%2Fgeopolymers%2F80&utm_medium=PDF&utm_campaign=PDFCoverPages
- Chen, B. & Wang, J. (2021, July 12). Experimental study on the durability of alkali-activated slag concrete after freeze-thaw cycle. *Advances in materials science and engineering*, 2021, 19. <https://doi.org/10.1155/2021/9915639>
- Chen, S., Zhang, Y., Yan, D., Jin, Jiangchuxiong, Tian, Y., . . . Fujitsu, S. (2020, September 10). The influence of Si/Al ratio on sulfate durability of metakaolin-based alkali activated. *Construction and Building Materials*, 265. <https://doi.org/10.1016/j.conbuildmat.2020.120735>
- Chen, W., Li, B., Wang, J., Thom, N. (2020, December 24). Effects of alkali dosage and silicate modulus on autogenous shrinkage of alkali-activated slag cement paste. *Cement and concrete research*, 141, 106322. <https://doi.org/10.1016/j.cemconres.2020.106322>
- Choi, S. & Lee, K.M. (2019, June 27). Influence of Na₂O Content and Ms (SiO₂/Na₂O) of Alkaline Activator on Workability and Setting of Alkali-Activated Slag Paste. *Materials*, 12, 2072. <http://dx.doi.org/10.3390/ma12132072>
- Collins, F. & Sanjayan, J.G. (1998, October 26). Strength and shrinkage properties of alkali-activated slag concrete containing porous coarse aggregate. *Cement and concrete research*, 29, 607-610. [https://doi.org/10.1016/S0008-8846\(98\)00203-8](https://doi.org/10.1016/S0008-8846(98)00203-8)
- Collins, F. & Sanjayan, J.G. (2001, January 3). Microcracking and strength development of alkali activated slag concrete. *Cement & Concrete Composites*, 23, 345-352. [https://doi.org/10.1016/S0958-9465\(01\)00003-8](https://doi.org/10.1016/S0958-9465(01)00003-8)
- Coppola, L., Coffetti, D., Crotti, E., Gazzinga, G., Pastore, T. (2020, April 27). The durability of one-part alkali-activated slag-based mortars in different environments. *Sustainability* 2020, 12, 3561. <https://doi.org/10.3390/su12093561>
- Cyr, M. & Pouhet, R. (2015, December). The frost resistance of alkali-activated cement-based binders. *Handbook of alkali-activated cements, mortars and concretes*, 2015, 293-318. <https://doi.org/10.1533/9781782422884.3.293>

- Dar, A.A., Anuradha, N. (2018). An application of Taguchi L9 method in black scholes model for European Call option. *International Journal of Entrepreneurship*. <https://www.abacademies.org/articles/an-application-of-taguchi-l9-method-in-black-scholes-model-for-european-call-option-7040.html>
- Detwiler, R. (2021, June 7). *What is sulfate attack?* Beton consulting engineers. <https://www.betonconsultingeng.com/what-is-sulfate-attack/>
- Dong, M., Elchalakani, M., Karrech, A. (2020, June). Curing conditions of alkali activated Fly Ash and Slag Mortar. *Journal of Materials in Civil Engineering*, 32, 04020122. [http://dx.doi.org/10.1061/\(ASCE\)MT.1943-5533.0003233](http://dx.doi.org/10.1061/(ASCE)MT.1943-5533.0003233)
- Duurzame ontwikkeling. (2021). *Verenigde naties*. Duurzame ontwikkeling. <https://www.duurzameontwikkeling.be/nl/internationaal-beleid/verenigde-naties>
- Duxson, P., Provis, J.L. (2008, December 15). Designing precursors for geopolymer concrete. *Journal of the American Ceramic Society*, 91, 3864-3869. <https://doi.org/10.1111/j.1551-2916.2008.02787.x>
- Ecoinvent. (s.d.). *Life Cycle Assessment (LCA) - Complete beginner's guide*. <https://ecochain.com/knowledge/life-cycle-assessment-lca-guide/>
- EPA. (s.d.). *Global greenhouse gas emissions data*. EPA. <https://www.epa.gov/ghgemissions/global-greenhouse-gas-emissions-data>
- Fagerlund, G. (1995). Freeze-thaw resistance of concrete: destruction mechanisms, concrete technology, test methods, quality control: a contribution to the BRITE/EURAM project BREU-CT92-0591 "The residual service life of concrete structures". *Report TVBM, 3060*. <https://lucris.lub.lu.se/ws/portalfiles/portal/4591559/1762611.pdf>
- Fernández-Jiménez, A., Palomo, A. (2003, June 24). Characterization of fly ashes. Potential reactivity as alkaline cements. *Fuel*, 18, 2559-2265. [https://doi.org/10.1016/S0016-2361\(03\)00194-7](https://doi.org/10.1016/S0016-2361(03)00194-7)
- Fraley, S., Oom, M., Terrien, B., & Zalewski, J. (2021, December 4). *Design of experiments via Taguchi methods - orthogonal arrays*. Engineering LibreTexts. [https://eng.libretexts.org/Bookshelves/Industrial_and_Systems_Engineering/Book%3A_Chemical_Process_Dynamics_and_Controls_\(Woolf\)/14%3A_Design_of_Experiments/14.01%3A_Design_of_Experiments_via_Taguchi_Methods_-_Orthogonal_Arrays](https://eng.libretexts.org/Bookshelves/Industrial_and_Systems_Engineering/Book%3A_Chemical_Process_Dynamics_and_Controls_(Woolf)/14%3A_Design_of_Experiments/14.01%3A_Design_of_Experiments_via_Taguchi_Methods_-_Orthogonal_Arrays)
- Fu, Y., Cai, L., & Wu, Y. (2011, Februari 18). Freeze-thaw cycle test and damage mechanics models of alkali-activated slag concrete. *Construction and Building Materials*, 7, 3144-3148. <https://doi.org/10.1016/j.conbuildmat.2020.120735>
- Gagg, C. (2014, February 28). Cement and concrete as an engineering material: an historic appraisal and case study analysis. *Engineering Failure Analysis*, 40, 114-140. <https://doi.org/10.1016/j.engfailanal.2014.02.004>
- Hadi, M., Farhan, N., & Sheikh, M. (2017, March 06). Design of alkali activated concrete with GGBFS at ambient temperature curing condition using Taguchi method. *Construction and Building Materials*, 140, 424-431. <https://doi.org/10.1016/j.conbuildmat.2017.02.131>

- Haha, B.M., Lothenbach, B., Le Saout, G., Wannefeld, F. (2011). Influence of slag chemistry on the hydration of alkali-activated blast-furnace slag - Part I: Effect of MgO. *Cement and Concrete research*, 41, 955-963. <http://dx.doi.org/10.1016/j.cemconres.2011.05.002>
- Hsu, T.T.C., Slate, F.O., Sturman, G.M., Winter, G. (1963, Februari 1). Microcracking of plain concrete and the shape of the stress-strain curve. *Journal proceedings*, 60, 209-224. <https://www.concrete.org/publications/internationalconcreteabstractsportal.aspx?m=details&ID=7852>
- Hu, X., Shi, C., Liu, X., Zhang, Z. (2020, July 2). Studying the effect of alkali dosage on microstructure development of alkali-activated slag pastes by electrical impedance spectroscopy (EIS). *Construction and building materials*, 261, 119982. <https://doi.org/10.1016/j.conbuildmat.2020.119982>
- Humad, A.M., Provis, J.L., Habermehl-Cwirzen, K., Rajczakowska, M., Cwirzen, A. (2021, January 2). Creep and long-term properties of alkali-activated Swedish-slag concrete. *Journal of material in civil engineering*, 33 (2). [https://doi.org/10.1061/\(asce\)mt.1943-5533.0003381](https://doi.org/10.1061/(asce)mt.1943-5533.0003381)
- Huseien, G.F., Mirza, J., Ismail, M., Ghoshal, S.K., Ariffin, M.A.M. (2018). Effect of metakaolin replaced granulated blast furnace slag on fresh and early strength properties of alkali activated mortar. *Ain Shams Engineering Journal*, 9, 1557-1566. <http://dx.doi.org/10.1016/j.asej.2016.11.011>
- Hüsken, G., & Brouwers, H. J. H. (2008). A new mix design concept for earth moist concrete: A theoretical and experimental study. *Cement and concrete research*, 38(10), 1246-1259. <https://doi.org/10.1016/j.cemconres.2008.04.002>
- Ismail, I., Bernal, S., Provis, J., Hamdan, S., & van Deventer, J. (2012, July 11). Microstructural changes in alkali activated fly ash/slag alkali activators with sulfate exposure. *Materials and Structures*, 46, 361-373. <https://doi.org/10.1617/s11527-012-9906-2>
- Jiang, H., Qi, Zhaojun., Yilmaz, E., Han, J., Qiu, J., Dong, C. (2019, May 26). Effectiveness of alkali-activated slag as alternative binder on workability and early age compressive strength of cemented paste backfills. *Construction and building materials*, 218, 689-700. <https://doi.org/10.1016/j.conbuildmat.2019.05.162>
- Kajaste, R., & Hurme, M. (s.d.). *Cement industry greenhouse emissions - management options and abatement cost*. Matheo conferences. <https://conferences.matheo.si/event/0/contribution/96/material/paper/0.pdf>
- Khalifa, A., Cizer, Ö., Pontikes, Y., Heath, A., Patureau, P., Bernal, S., & Marsh, A. (2020, April 12). Advances in alkali-activation of clay minerals. *Cement and Concrete Research*, 132. <https://doi.org/10.1016/j.cemconres.2020.106050>
- Klimaat akkoord. (2019, June 28). *Klimaatakkoord hoofdstuk industrie*. Klimaat akkoord. <https://www.klimaatakkoord.nl/industrie/documenten/publicaties/2019/06/28/klimaatakkoord-hoofdstuk-industrie>
- Kramer, W. (2020, April 20). *Cementmarkt in Nederland*. Betonhuis. <https://betonhuis.nl/cement/cementmarkt-nederland>

- Kumari, N., & Mohan, C. (2021, June 24). *Basics of clay minerals and their characteristic properties*. Intechopen. <https://www.intechopen.com/chapters/76780>
- Li, W., Pour-Ghaz, M., Castro, J., Weiss, J. (2012, March 1). Water absorption and critical degree of saturation relating to freeze-thaw damage in concrete pavement joints. *Journal of materials in civil engineering*, 24. [https://doi.org/10.1061/\(ASCE\)MT.1943-5533.0000383](https://doi.org/10.1061/(ASCE)MT.1943-5533.0000383)
- Mahmood, A.H. (2020). *Is cooling of alkaline activator for 24 hours (for alkali-activated binders) scientifically important?* Researchgate. <https://www.researchgate.net/post/Is-cooling-of-alkaline-activator-for-24-hours-for-alkali-activated-binders-scientifically-important>
- Mangor, K. (2020). *Revetments*. Coastal Wiki. <http://www.coastalwiki.org/wiki/Revetments>
- Nanayakkara, O., Gunasekara, C., Sandanayake, M., Law, D.W., Nguyen, K., Xia, J., Setunge, S. (2020, November 12). Alkali activated slag concrete incorporating recycled aggregate concrete: Long term performance and sustainability aspect. *Construction and building materials*, 271, 121512. <https://doi.org/10.1016/j.conbuildmat.2020.121512>
- Nationale MilieuDatabase (s.d.). *Bepalingsmethode*. Nationale MilieuDatabase. <https://milieudatabase.nl/milieuprestatie/bepalingsmethode/>
- Nederlands Normalisatie Instituut. (2008, June 1). *NPR-CEN/TR 15697:2008 en - Cement - Presetatiebeproeving voor de weerstand van sulfaat - Rapport over de stand van de techniek*.
- Nederlands Normalisatie Instituut. (2016, December 1). *NVN-CEN/TS 12390-9:2016 en - Beproeving van verhard beton - Deel 9: Vorst/dooi bestandheid - Afschilfering*.
- Nederlands Normalisatie Instituut. (2017, December 1). *NEN-EN 206+NEN 8005:2017 nl - Beton - Specificatie, eigenschappen, vervaardiging en conformiteit + Nederlandse invulling van NEN-EN 206*.
- Nederlands Normalisatie Instituut. (2012, January 1). *NEN-EN 933-1:2012 en - Beproevingsmethoden voor geometrische eigenschappen van toeslagmaterialen - Deel 1: Bepaling van de korrelgrootteverdeling - Zeefmethode*.
- Nederlands Normalisatie Instituut. (2019, March 16). *NEN-EN 1015-3:1999 en - Beproevingsmethoden voor mortel voor metselwerk - Deel 3: Bepaling van de consistentie van mortelspecie (met schoktafel)*.
- Nederlands Normalisatie Instituut. (2019, November 20). *NEN-EN 1015-11:2019 en - Beproevingsmethoden voor mortel voor metselwerk - Deel 11: Bepaling van de buigtrek- en druksterkte van verharde mortel*.
- Nederlands Normalisatie Instituut. (2022, March 1). *NEN-EN 1097-6:2022 en - Beproevingsmethoden voor de bepaling van mechanische en fysische eigenschappen van toeslagmaterialen - Deel 6: Bepaling van de deeltjesdichtheid en de wateropname*.
- Nederlands Normalisatie Instituut. (2005, January 31). *NEN-EN 1338:2003 nl - Betonstraatstenen - Eisen en beproevingsmethoden*.

- Nederlands Normalisatie Instituut. (2015, November 24). *NEN 7024-1+C1:2005 nl - Zetsteen - Deel 1: Algemene eisen*.
- Nederlands Normalisatie Instituut. (2015, April 23). *NEN 7024-2 - Zetsteen - Deel 2: Zetsteen van cementbeton, zonder interlocking en zonder wapening*.
- Nederlands Normalisatie Instituut. (2019, June 1). *NEN-EN 12350-4:2019 en - Beproeven van betonspecie - Deel 4: Verdichtingsmaat*.
- Nederlands Normalisatie Instituut. (2019, July 05). *NEN-EN 12390-3:2019 en - Beproeving van verhard beton - Deel 3: Druksterkte van proefstukken*.
- Neville, A. (s.d.). Properties of concrete. In A. Neville, *Properties of concrete*. Pearson: 2011.
- Nippon Slag Association. (s.d.). *Generation of iron and steel slag*. Nippon Slag Association. <https://www.slg.jp/e/slag/process.html>
- Ontwerptool Groen Beton. (s.d.). *Ontwerptool Groen Beton v5.2*. <https://www.ontwerptoolgroenbeton.nl>
- PCA. (s.d.). *Freeze-thaw resistance*. PCA, America's cement manufacturers. <https://www.cement.org/Learn/concrete-technology/durability/freeze-thaw-resistance>
- Pouhet, R. (2016, April 5). Formulation and durability of metakaolin-based alkali activateds. Civil Engineering. Université Paul Sabatier - Toulouse III. <https://tel.archives-ouvertes.fr/tel-01297848>
- Provis, J.L. (2018, March 2). Alkali-activated materials. *Cement and concrete research* 114 (2018), 40-48. <http://dx.doi.org/10.1016/j.cemconres.2017.02.009>
- Provis, J. L., & Deventer, J. S. (2009). Geopolymers: structure, processing, properties and industrial applications. In J. L. Provis, & J. S. Deventer, *Geopolymers: structure, processing, properties and industrial applications*. Cambridge, UK: Woodhead publishing limited.
- Provis, J., & van Deventer, J. (S.d.). Alkali-activated materials, state-of-the-art report, RILEM TC 224-AAM. In J. L. Provis, & J. S. van Deventer, *Alkali-activated materials*.
- Puertas, F., Gutiérrez, R., Fernández-Jiménez, A., Delvasto, S., & Maldonado, J. (2002, September 30). Alkaline cement mortars. Chemical resistance to sulfate and seawater attack. *Materiales de Construcción*, 52. <https://doi.org/10.3989/mc.2002.v52.i267.326>
- Puertas, F., Amat, T., Fernández-Jiménez, A., Vázquez, T. (2003, June 27). Mechanical and durable behavior of alkaline cement mortars reinforced with polypropylene fibers. *Cement and concrete research*, 33, 2031-2036. [https://doi.org/10.1016/S0008-8846\(03\)00222-9](https://doi.org/10.1016/S0008-8846(03)00222-9)
- Rakhimova, N.R. & Rakhimov, R.Z. (2018, August 16). Reaction products, structure and properties of alkali-activated metakaolin cements incorporated with supplementary materials - a review. *Journal of Materials Research and Technology*, 8, 1522-1531. <https://doi.org/10.1016/j.jmrt.2018.07.006>

- Rijksoverheid. (2019, June 28). *Het klimaatakkoord in (meer dan) 70 vragen*. Rijksoverheid. <https://www.rijksoverheid.nl/ministeries/ministerie-van-economische-zaken-en-klimaat/documenten/publicaties/2019/06/28/het-klimaatakkoord-in-meer-dan-70-vragen>
- San Nicolas, R., Cyr, M., Escadrilles, G. (2013, September 17). Characteristics and applications of flash metakaolin. *Applied clay science*, 83-84, 253-262. <https://doi.org/10.1016/j.clay.2013.08.036>
- SGS Intron. (2020, April 14). *Ontwikkelingen betreffende hoofdbestanddelen voor klinkergebaseerde cementen en alkali activerenden*. Betonakkoord. (link of report is on this page) <https://www.betonakkoord.nl/actueel/nieuws/nieuwsberichten/beschikbaarheid-bindmiddelen-beton/>
- Shahrajabian, F. & Behfarnia, K. (2018, May 9). The effects of nano particles of freeze and thaw resistance of alkali-activated slag concrete. *Construction and building materials*, 176, 172-178. <https://doi.org/10.1016/j.conbuildmat.2018.05.033>
- Shi, C.J., Day, R.L. (1995, April 4). A calorimetric study of early hydration of alkali-slag cements. *Cement and Concrete Research*, 25, 1333-1346. [https://doi.org/10.1016/0008-8846\(95\)00126-W](https://doi.org/10.1016/0008-8846(95)00126-W)
- Shi, Z., Shi, C., Wan, S., Ou, Z. (2017, March 22). Effect of alkali dosage on alkali-silica reaction in sodium hydroxide activated slag mortars. *Construction and building materials*, 143, 16-23. <http://dx.doi.org/10.1016/j.conbuildmat.2017.03.125>
- Song, S. & Jennings, H.M. (1998, November 16). Pore solution chemistry of alkali-activated ground granulated blast-furnace slag. *Cement and Concrete research* 29 (1999), 159-170. [https://doi.org/10.1016/S0008-8846\(98\)00212-9](https://doi.org/10.1016/S0008-8846(98)00212-9)
- Stichting nationale milieudatabase. (2022, March). Bepalingsmethode Milieuprestatie Bouwwerken. <https://milieudatabase.nl/milieuprestatie/bepalingsmethode/>
- U.S. Department of Transportation, federal highway administration. (2016, March 08). *User guidelines for waste and byproduct materials in pavement construction*. U.S. Department of Transportation, federal highway administration. <https://www.fhwa.dot.gov/publications/research/infrastructure/structures/97148/bfs1.cfm>
- Ugurlu, A., Karakoç, M., & Özcan, A. (2021, July 29). Effect of binder content and recycled concrete aggregate on freeze-thaw and sulfate resistance of GGBFS based alkali activated concretes. *Construction and Building Materials*, 301. <https://doi.org/10.1016/j.conbuildmat.2021.124246>
- Valenza, J.J., Scherer, G.W. (2006, March 23). Mechanism for salt scaling. *Journal of the American ceramic society*, 89, 1161-1179. <https://doi.org/10.1111/j.1551-2916.2006.00913.x>
- Vogt, O., Ukrainczyk, N., Ballschmiede, C., Koenders, E. (2019, October 24). Reactivity and microstructure of metakaolin based alkali activateds: effect of fly ash and liquid/solid contents. *Materials (Basel)*, 12, 3485. <https://dx.doi.org/10.3390%2Fma12213485>

- Winnefeld, F., Gluth, G. J., Bernal, S. A., Bignozzi, M. C., Carabba, L., Chithiraputhiran, S., Stephan, D. (2020, November 11). RILEM TC 247-DTA round robin test: sulfate resistance, alkali-silica reaction and freeze–thaw resistance of alkali- activated concretes. *Materials and structures*, 53. <https://doi.org/10.1617/s11527-020-01562-0>
- WWF. (s.d.). *The effects of climate change*. WWF. <https://www.wwf.org.uk/learn/effects-of/climate-change#climate-change-and-oceans>
- Ye, G. (2020, December 17). New types of concrete [Power Point slides].
- Ye, H., Chen, Z., Huang, L. (2019, August 30). Mechanism of sulfate attack on alkali-activated slag: The role of activator composition. *Cement and Concrete research*, 125, 105868. <https://doi.org/10.1016/j.cemconres.2019.105868>
- Zakka, W. P., Abdul Shukor Lim, N. H., & Chau Khun, M. (2021). A scientometric review of alkali activated concrete. *Journal of Cleaner Production*, 280. <https://doi.org/10.1016/j.jclepro.2020.124353>
- Zhang, Z, Hu, Y., Zhang, H., Luo, L., Yao, S. (2010, March 15). Electrochemical layer-by-layer modified imprinted sensor based on multi-walled carbon nanotubes and sol-gel materials for sensitive determination of thymidine. *Journal of Electrochemical Chemistry*, 644, 7-12. <http://dx.doi.org/10.1016/j.jelechem.2010.03.015>
- Zhu, H., Liang, G., Li, H., Wu, Q., Zhang, C., Yin, Z., Hua, S. (2021, January 11). Insights to the sulfate resistance and microstructures of alkali-activated metakaolin/slag pastes. *Applied clay science*, 202, 105968. <https://doi.org/10.1016/j.clay.2020.105968>
- Zuo, Y. (2019). *Experimental study and Numerical Simulation of the Reaction Process and Microstructure Formation of Alkali-Activated Materials*. (1 ed.). Delft University of Technology. <https://doi.org/10.4233/uuid:193a4016-5fc7-401b-babe-722ff6a95a6c>

