

USE OF SEWAGE SLUDGE ASH IN REINFORCED CONCRETE RISK OF CORROSION



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Preface

This thesis is made as a completion of the master education in civil engineering at the Technical University of Denmark. The project counts for 35 ECTS points and it has been made in the period from September 1st 2013 to March 24th 2014.

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(Photo on front page: A small pile of LSA-depot)

DTU, Kongens Lyngby, March 24th 2014

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Abstract

The yearly emission of CO_2 has never been higher and cement production now accounts for more than 5% of the overall emission. The usage of (almost unlimited) secondary resources such as sewage sludge ash (SSA) as partly cement replacement would therefore benefit the environment in more than one way since it would also reduce the amount of SSA that has to be deposited.

The investigated ash (LSA-depot) hails from *Renseanlæg Lynetten* and it has been stored in a dammed up part of the sea Øresund with no roof for at least two years. After sampling it has been milled as previous experiments have shown this very beneficial for the strength of mortar made with SSAs.

The research is primarily focused on the risk of reinforcement corrosion caused by chloride ingress in concrete with LSA-depot.

A number of laboratory tests were however first made with the SSA in order to assess it by comparison with both other ashes and the current standards for concrete and admixtures in concrete. The pH value that was measured to be 12.6 is very beneficial for use in concrete as it fortifies the alkaline environment. The loss on ignition was really low and complies therefore well with the limits stated by standards for fly ash. When comparing the results with a previous sampling of LSA-depot both the pH value, conductivity and water solubility gave an indication towards that the previous sampling had been far more washed out which is not favourable for usage in concrete.

The content of micro and macro elements in the SSA restricts the possible usage according to Danish EPA. The chloride content of 0.18% exceeds the allowed amount for use in concrete.

Mortar specimens were cast both with and without LSA-depot. Samples with LSA-depot were made as 10% cement replacement and 5% sand replacement. Compression strength tests of the samples gave results that all meet the requirement for activity index when using fly ash in concrete. Results for porosity and density obtained by [Rem, 2013] were discussed but no variation in the porosity for reference and LSA-depot samples was found. This might be caused by the used test method as it does not reveal if there are differences in the pore size distribution. The chloride content in all the mortar specimens is beneath the limit for concrete and no significant variation between samples with and without LSA-depot was detected. Leaching tests conducted on pure LSA-depot and mortar with LSA-depot showed that no elements were washed out in significant amount.

The diffusion of chloride ions into mortar was compared for 10% cement replacement, 5% sand replacement and reference mortar samples and no difference was found. Tests were run for 7, 14 and 21 weeks and neither showed a variation between the three mortar recipes.

Experiments were carried out to investigate if chloride ions could bind to either pure SSA or mortar with SSA. Early results were overall very good. However after test periods of 16 weeks the pure SSA released more chloride than it had been measured to contain and mortar with unwashed LSA-depot was not able to bind as much chloride as the reference mortar. Mortar with washed LSA-depot had a better chloride binding ability than the reference mortar but results were not as good after 16 weeks as after 2 weeks.

Reinforced mortar specimens both with and without LSA-depot were exposed to cycles of being

soaked in salt water and drying out to provide the best circumstances for initiation of corrosion. The electrical potential was measured regularly for each specimen but although the measurements already after seven days indicated with 90% certainty that corrosion had been initiated for all specimens, no signs hereof were spotted during visual inspections after 6 and 17 weeks. The lack of corrosion was assumed to be caused by too short cycles that did not allow for the specimens to dry completely.

Neither the conducted experiments nor the previous research have provided critical evidence that LSA-depot is not useable in concrete. The SSA is not found to increase the risk of corrosion at all. A removal of the harmful elements in the SSA and a mandatory homogenisation process would however be very beneficial.

Sammenfatning

Den samlede årlige CO_2 -udledning har aldrig været højere og produktionen af cement står for hele 5% af den samlede udledning. Hvis en alternativ ressource som slamaske (SSA) - der findes i næsten ubegrænsede mængder - kan bruges som erstatning for en del af cementen i beton vil det ikke kun gavne miljøet i form af nedsat CO_2 -udledning men også ved at nedsætte mængden af SSA der skal deponeres betragteligt.

Slamasken som undersøges i nærværende projekt, stammer fra *Renseanlæg Lynetten* og har været opbevaret udendørs i en inddæmmet del af Øresund i minimum to år. Den benævnes LSA-depot. Efter udtagelsen af asken er den blevet knust i en ringknuser, da dette ved tidligere forsøg har vist sig at være gavnligt for trykstyrken af mørtel med SSA.

Undersøgelserne i denne rapport er hovedsageligt fokuseret på risikoen for armeringskorrosion forårsaget af klorider i beton med LSA-depot.

Først er der dog udført en karakteristik af slamasken ved hjælp af en række laboratorieforsøg. Resultaterne heraf er sammenlignet dels med andre asker og dels med gældende standarder for beton, flyveaske og andre tilsætninger. Askens pH værdi blev målt til 12,6, hvilket er yderst fordelagtigt til brug i beton, hvor det vil være med til at fastholde betonens basiske miljø. Glødetabet var meget lavt og overholder derfor fint kravene til flyveaske. Når resultater fra en tilsvarende undersøgelse af en tidligere udtagning af asken sammenlignes med de nuværende resultater peger både pH, ledningsevne og vandopløselighed på at den tidligere udtagning af asken var langt mere udvasket, hvilket ikke er fordelagtigt, hvis asken skal bruges i beton.

Indholdet af tungmetaller og andre farlige grundstoffer i asken begrænser anvendelsesmulighederne for asken betragteligt ifølge en bekendtgørelse fra Miljøstyrelsen. Kloridindholdet i asken på 0,18% overskrider desuden det tilladte for tilsætninger til beton.

Der er blevet støbt mørtelprøver både med og uden LSA-depot, førstnævnte er støbt både som 10% cementerstatning og 5% sanderstatning. Trykstyrketests viste at alle tre prøvetyper levede op til kravene der gælder for brug af flyveaske i beton. Resultater for porøsitet og densitet fra [Rem, 2013] er diskuteret, men en forskel mellem mørtel med og uden slamaske er ikke konstateret. Dette skyldes muligvis at den benyttede testmetode ikke kan påvise en variation i porestørrelsesfordelingen. Kloridindholdet i alle typer mørtelprøver er under grænsen for det tilladte og der blev heller ikke fundet nogen væsentlig forskel mellem prøver med og uden LSA-depot. Tests med både slamaske og mørtel med slamaske viste at ingen af delene udvasker nogle tungmetaller eller lignende miljøskadelige grundstoffer i foruroligende grad.

Der blev ikke konstateret nogen forskel i klorid diffusionen i henholdsvis reference mørtel, mørtel med 10% cementerstatning og mørtel med 5% sanderstatning. Diffusionen blev undersøgt med testperioder på 7, 14 og 21 uger og ingen af forsøgene viste nogen variation mellem de tre mørteltyper.

Kloridbinding til både aske og mørtel med aske er blevet undersøgt og de tidlige resultater var generelt positive. Efter 16 ugers test viste både vasket og uvasket LSA-depot sig dog at afgive en del klorid, endda også mere end det tidligere var målt at asken indeholder. Mørtel med uvasket aske var ikke i stand til at binde ligeså meget klorid som referencemørtlen efter 16 uger. Mørtel med vasket LSA-depot var dog bedre end referencen, men bindingen af kloridioner efter 16 uger var ikke ligeså god som efter kun to uger. Armerede mørtelprøver både med og uden LSA-depot har været udsat for skiftevis saltvand og udtørring for at skabe de bedste betingelser for igangsætning af korrosion. Undervejs blev det elektriske potentiale målt regelmæssigt og det blev med 90% sikkerhed konstateret at der var begyndende korrosion i samtlige prøveelementer indenfor de første syv dage. Dette blev dog ikke bekræftet ved visuel inspektion efter hverken 6 eller 17 uger. Det antages at den manglende korrosion skyldes for korte test cyklusser, som ikke tillod fuldstændig udtørring af mørtlen.

Der blev ikke konstateret tegn på at brugen af LSA-depot øger korrosionsrisikoen i beton. Således har hverken tidligere undersøgelser eller forsøg i dette projekt givet bevis for at slamasken ikke skulle være egnet til brug i beton. Det vil dog være yderst fordelagtigt, hvis tungmetallerne i asken fjernes og der indføres en obligatorisk homogenisering af LSA-depot så variationerne imellem de forskellige udtagninger minimeres.

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D Poster

1 Introduction

The latest analysis from Global Carbon Project on the yearly carbon dioxide emission [Global Carbon Project, 2013] showed the highest emissions from fossil fuel burning and cement production ever recorded in human history with a total of 9.7 ± 0.5 billion tonnes of CO_2 emitted in 2012. And on top of that the numbers for 2013 are expected to be even more horrifying with an increase of 2.1%.

Cement production was responsible for 5.3% of the total carbon dioxide emission in 2012 which is more than 0.5 billion tonnes of CO_2 .

Sewage sludge ash (SSA) originates from the incineration of sewage sludge at a sewage treatment plant. Most of the SSA is afterwards disposed at landfills. Due to the increasing world population the amount of deposited SSA have been skyrocketing for the last couple of decades and that will create problems eventually as it is not a sustainable solution. However it should be mentioned that a small part of the SSA is reused for different purposes e.g. some of the SSA from *Renseanlæg Lynetten* is used for building insulation material [Lynettefællesskabet I/S, 2013].

A Danish sponsored project running from 1998 to 2002 [Danish Centre for Green Concrete, 2014] worked with using SSA in concrete on a trial basis. While good results were obtained the project was not prolonged despite the fact that almost 10 million tonnes of concrete was produced in Denmark alone in 2006 [Betonindustriens Fællesråd, 2006] which makes the idea of reusing SSA in concrete a great sustainable idea. It would solve several problems at once i.e. reduce the amount of deposited SSA and also reduce CO_2 emission caused by cement production if the ash can replace a small part of the cement.

During the last couple of years several smaller projects on the use of SSA in concrete have been performed at DTU, among these are two projects that focused on the strength development of mortar with the ash LSA-depot as a replacement for a small amount of the cement [Carlsen & Petersen, 2013 and Rem, 2013]. LSA-depot hails from the sewage treatment plant *Renseanlæg Lynetten* and has after incineration been stored outdoors in a dammed up part of the sea Øresund.

The results were promising but more research is necessary before LSA-depot can be used in concrete which leads to the justification for this report. It seeks to investigate the influence from LSA-depot on the corrosion of reinforcement in concrete. As point of departure it is aimed to answer whether LSA-depot creates a beneficial or detrimental environment in concrete with regard to reinforcement corrosion. Where appropriate and possible the results of the research are compared to other SSAs.

The investigation is focused on chloride induced corrosion, hence corrosion caused by carbonation lies outside the scope of this project.

While the main scope of this report is investigation of the corrosive environment in concrete with LSA-depot, a general assessment of the SSA is also included. The results of the chara-terisation of LSA-depot are compared to both other SSAs, Danish declarations and relevant Eurocodes. Where Danish supplementary standards to the Eurocodes exist, these are used.

The title of this report is "Use of Sewage Sludge Ash in Reinforced Concrete - Risk of Corrosion" but the investigations are built on tests conducted with mortar. This is due to the fact that when working with small test specimens the bigger aggregates used in concrete could

interfere with the results and some tests would not even be possible to conduct with concrete. Since mortar is the weakest link in concrete and the aggregates have no influence on corrosion of the reinforcement the results obtained with mortar will be applicable for concrete as well.

This report is aimed partly at civil engineering students from fourth semester onwards and partly at the ZeroWaste Group at DTU. A certain amount of theory is therefore included, it is however expected that the reader knows the most essential basic theory regarding for instance concrete, mortar, admixtures, reinforcement and tests conducted with those materials.

2 Theory

2.1 Mortar and Concrete

The main components of mortar are cement, water and sand and by adding gravel concrete is obtained. Cement and water combined provides a cement paste that will bind all the materials together creating a composite material.

Besides the main components concrete usually also contains different admixtures that enhance the properties of either the fresh or the hardened concrete (or both).

The compositions of cement paste, mortar and concrete can be seen in figure 1 that shows the difference between the three materials.



Figure 1: Compositions of cement paste, mortar and concrete (After table D5.2 in [Geiker & Nielsen, 2008])

In the following sections the cement, admixtures and the properties of concrete are described more thoroughly.

2.1.1 Portland Cement

Cement consists mostly of calcium oxides coming from limestone (CaO), silicates coming from sand (SiO_2) and aluminates coming from clay (Al_2O_3) [Johannesson, 2012].

When cement is produced the first stage is preheating the raw materials to evaporate both the free and the weakly bound water in the clay and thereby obtaining 'meta'-clay. The 'meta'-clay will then slowly react with the free lime which will give the following solid-state type of reaction:

 $'Meta' - clay + CaO(s) \rightarrow CaO \cdot SiO_2(s) + CaO \cdot Al_2O_3(s) + CaO \cdot Fe_2O_3(s) (solid st.)$

With continued heating the first phase will bind free lime in another solid-state type of reaction and thereby form the cement clinker phase, belite:

$$CaO \cdot SiO_2(s) + CaO(s) \rightarrow 2CaO \cdot SiO_2(s) \text{ (solid st.)}$$

The second but most important cement phase, alite, is formed by a liquid-state type of reaction at sufficiently high temperatures which melts some phase boundaries that allows belite to react with free lime:

 $2CaO \cdot SiO_2(s) + CaO(s) + melt \rightarrow 3CaO \cdot SiO_2(s)$ (liquid st.)

The last two main cement clinker phases, aluminate $(3CaO \cdot Al_2O_3)$ and ferrite $(4CaO \cdot Al_2O_3 \cdot Fe_2O_3)$, are formed during the cooling process of the cement production. Table 1 provides an overview of the four main cement clinker phases.

Clinkon phage	Chemical formula	Chemical formula
Chinker phase	oxide notation	short notation
Alite	$3CaO \cdot SiO_2$	C_3S
Belite	$2CaO\cdot SiO_2$	C_2S
Aluminate	$3CaO \cdot Al_2O_3$	C_3A
Ferrite	$4CaO \cdot Al_2O_3 \cdot Fe_2O_3$	C_4AF

таріет. тпе шаш сешені сникег риазе	Table	1:	The	main	cement	clinker	phases
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When cement comes into contact with water the hydration process begins. The water reacts with all the cement clinkers simultaneously forming different solid hydration products which makes the hydration process a bit complex.

The two main solid hydration products that are formed are calcium-silicate-hydrate (C-S-H) and calcium-hydroxide (CH). They are formed together which is shown in the reactions below.

Reaction between alite and water:

 $2C_3S$ + 7H \rightarrow $C_3S_2H_4$ + 3CH(tricalcium silicate) (water) (C-S-H) (calcium-hydroxide)

Reaction between belite and water:

$2C_2S$	+	5H	\rightarrow	$C_3S_2H_4$	+	CH
(dicalcium silicate)		(water)		(C-S-H)		(calcium-hydroxide)

Gypsum is usually added to cement in order to control the speed of the hydration process and that allows for two other hydration products to be formed: ettringite and monosulfate. As an example these are formed in the two stages of C_3A hydration which is shown below.

First stage:

 C_3A + $3CS^*H_2$ + 26H \rightarrow $C_6AS_3^*H_{32}$ (tricalcium aluminate) (gypsum) (water) (ettringite)

Second stage:

2.1.2 Admixtures

Two types of admixtures are added to concrete to improve the properties of the fresh and hardened concrete: chemical admixtures and mineral admixtures [Johannesson, 2012]. The chemical admixtures include - but are not limited to - air-entraining agents, water-reducers, accelerators, retarders and superplasticizers. It is important to know that these are widely used in the production of concrete but besides that they are not relevant in this report and will therefore not be discussed further.



Figure 2: An overview of the different admixtures that can be used in concrete

There are three categories of mineral admixtures: pozzolans, cementitious materials and nonreactive materials. The last-mentioned includes hydrated lime and ground limestone and works as inert fillers. The cementitious materials cover ground-granulated blast-furnace slag (GG-BFS) which is a by-product from iron production. It does need some cement to be activated but due to its own hydraulic properties the amount of GGBFS can be up to 80% of the total content of cementitious materials in the concrete. It reacts slowly with water and has several positive effects on concrete including lowering of the permeability which reduces the rate of chloride ion diffusion, reduction of the heat of hydration which reduces the risk of thermal cracking and the ability to eliminate the need of low-alkali or sulfate-resistant portland cement [Johannesson, 2012].

The last and most interesting mineral admixtures are the pozzolans which include the natural volcanic ash and the industrial waste products fly ash and silica fume. A pozzolan is defined as "a siliceous and aluminous material which, in itself, processes little or no cemenstitious value but which will, in finely divided form in the presence of moisture, react chemically with calcium hydroxide at ordinary temperature to form compounds possessing cemenstitious properties" [ASTM C125]. The pozzolans share the ability to reduce the permeability of the concrete thus increasing the resistance to chloride penetration and making the concrete more durable. The fly ash also minimizes the risk of thermal cracking by reducing the heat of hydration. The strength in the concrete at later ages is improved with all the three types of pozzolans. Silica fume also improves the strength in the concrete at early ages and in some cases even allow for a smaller total amount of cementitious materials due to the enhancement in strength.

Pozzolans do not react with pure water but need the calcium-hydroxide solution to gain the cementitious properties. The reason for that is not fully known but one hypothesis is that the pozzolan particles react very fast with the water creating a gel-like cover on themselves and thereby preventing any further reactions. The alkaline solution in the water-cement mixture

then slowly breaks the cover down allowing for the pozzolan particles to react with the water forming C-S-H.

2.1.3 Influences on the Strength of Concrete

Several factors have an influence on the strength of a concrete or mortar including the cement and aggregate type. The biggest influence comes from the w/c-ratio which describes the weight ratio between water and cement in the concrete. The strength is lowered when the w/c-ratio is increased which is described in the empirical *Bolomey Formula* [Geiker & Nielsen, 2008]:

$$f_c = K\left(\frac{1}{w/c} - 0.5\right)$$

The constant K varies depending on the cement type.

The strength of a material is also affected by the porosity of said material which is described in the empirical *Ryschkewitch Formula* [Geiker & Nielsen, 2008]:

$$\sigma(p) = \sigma_0 \cdot e^{-B \cdot p}$$

The constant B is a material parameter that is usually set to 7. p is the porosity and σ_0 is the theoretical strength for the material with the porosity p = 0.

The porosity's influence on the strength of concrete is interesting due to the fact that some admixtures make the concrete more dense which then according to Ryschkewitch improves the strength of the concrete.

2.2 Sewage Sludge Ash

2.2.1 LSA-depot

The sewage sludge ash that is studied and tested in this report hails from Renseanlæg Lynetten that is located on Amager, Denmark. The sewage treatment plant is owned by eight municipalities in and around Copenhagen and the catchment area that is split with Renseanlæg Damhusåen covers $123km^2$ [Lynettefællesskabet I/S, 2013].

At the sewage treatment plant the wastewater and household sewage undergoes three processes: physical, biological and chemical treatment in order to remove organic particles, nitrogen, phosphorus and most of the pathogenic viruses and bacterias. The first stage is the physical treatment where the wastewater is lead through mechanical filters also known as bar screens and then on to sand and grease removal. The grease and fat are removed by blowing air in the base of the tank forcing the grease to the surface where it will be removed. In the last step of the physical process the wastewater is left stagnant in order for the heavy organic particles to precipitate. The particles are removed as the primary sludge.

In the biological process the nitrogen in the wastewater is transformed to free nitrogen equalling 79% of our air, the remaining organic particles are decomposed to water and carbondioxide and phosphorus accumulative bacterias are used to absorb the phosphorus before being removed from the wastewater.

The final stage is the chemical treatment where ferric chloride is added to the wastewater to remove the remaining phosphorus that the bacterias in the biological process did not absorb. The phosphorus and the biological sludge are precipitated and the water is then lead out in the ocean. The sludge is then divided in to active sludge that will be used future biological processes and excess sludge. The excess sludge will along with the primary sludge and grease from the physical treatment be transferred to heated tanks where it is left to rot for approximately three weeks. After that period the rotten sludge is first centrifuged, then dried and finally it is incinerated in a "fluid bed" oven. The incineration produces flue gas which is cleaned before being lead out and the ash that is stored in a $300m^3$ outdoor depot which is a dammed up part of the sea Øresund with no roof. The sludge ash, LSA-depot, is left in the depot for a minimum of two years before a sample has been taken out.

Previous Results with LSA-depot

This report builds on the previous experiments [Carlsen & Petersen, 2013 and Rem, 2013] with LSA-depot and it is therefore relevant to accentuate the most important results.

The research treated the strength development in mortar with sludge ash as cement and sand replacement. The first report ([Carlsen & Petersen, 2013]) focused on testing the effect of several simple pretreating techniques on the ash including acid washing and milling. Results showed that the milling of the SSA had a positive effect on the strength development in the mortar specimens. Experiments were made with both the SSA used as cement replacement and as sand replacement. The second report ([Rem, 2013]) aimed to investigate whether LSA-depot has pozzolanic properties or the ability to enhance the strength of mortar by a filler effect or neither. The focus was on using the SSA as only cement replacement since that is most profitable with regards to both economy and environment. Tests were made with both 10%, 20% and 1:2 cement replacement and the best results were obtained for the 10% specimens. The development of strength of the mortar was investigated up to 77 days of curing.

The SSA showed some tendency to develop strength at later ages but the reference strength was never reached by the specimens with ash. Whether LSA-depot has pozzolanic properties is therefore not fully determined yet.

Pretreatment

As mentioned above previous research has shown the best results with regards to the strength development in mortar with SSA admixture when the sewage sludge ash was milled. Therefore the LSA-depot that is used in mortar specimens is milled.

2.3 Literature Study on SSAs in Concrete, Chloride Ions and Corrosion

During the planning and conduction of the experiments for this report a literature study was made on previous research regarding SSA used in concrete, chloride binding into hydrated cement and techniques for estimation of the rate of corrosion. Several of the most interesting articles are mentioned during the following sections however for a complete overview of the studied literature table 2 provides a short recap of the results obtained in the previous research.

Topic	Results	Articles
The usage of SSA as cement replace- ment with a focus on the strength development	Results showed that neither of the investi- gated SSAs seemed to have any pozzolanic properties. Milling proved to have a pos- itive effect on the strength of the mortar with all kinds of SSAs. For SSA with high water solubility a simple washing of the SSA before using it as cement replacement also resulted in an increased strength.	Sewage sludge ash as cement replacement after simple pretreatment by [Ottosen et al., 2013a]. The article combines the research made by [Carlsen & Petersen, 2013], [Reiff & Meldgaard, 2013] and [Rem, 2013] among others.
Chloride binding into hardened ce- ment	Results showed that chloride binding strongly depends on the amount of alu- minum in the binder. Furthermore it was proved that most of the bound chloride in cement pastes, that have been exposed to high chloride concentrations, remains irre- versibly bound when the cement paste is later exposed to chloride-free solutions.	The effect of supple- mentary cementitious materials on chloride bind- ing in hardened cement paste by [Thomas et al., 2012]. Chloride binding into hydrated blended ce- ments: The influence of limestone and alkalinity by [Ipavec et al., 2013].
Corrosion behav- ior of reinforce- ment bars and chloride ingress into cement with mineral additions	Corrosion of rebars were investigated by electrochemical techniques. Results re- vealed that mortar with a partial substi- tution of cement with LFS (ladle furnace slag) performed just like the reference mor- tar with regard to corrosion behavior. Ex- periments made with fly ash or slag as cement replacement showed that neither of the samples with mineral additions had left the passive state after four years of curing whereas the reference samples had started to corrode prior to that.	Effects of some mineral additions to Portland ce- ment on reinforcement cor- rosion by [Andrade & Bu- ják, 2013]. Corrosion behavior of reinforcement bars embedded in mortar specimens containing ladle furnace slag in partial sub- stitution of aggregate and cement by [Prieto et al., 2012].

Table 2: Previous research relevant for this project

2.4 Relevant Standards for Assessment of LSA-depot

2.4.1 Requirements From DS/EN 450-1:2012

At the moment there are no European standards specifically regarding the use of SSA in concrete or mortar. But since the aim is that the use of SSA in concrete serves the same purpose as the use of fly ash it is considered relevant to compare the ash LSA-depot with the standard DS/EN 450-1:2012 *Fly ash for concrete - Part 1: Definition, specification and conformity criteria.* It is however noted that according to the definition of fly ash in DS/EN 450-1:2012 sewage sludge ash can not be defined as a fly ash.

The most important chemical and physical requirements according to the standard are listed

in table 3 and 4.

Subject	Test Method	Chemical F	Requirement
		Category A \leq	5.00~% by mass
Loss on ignition	EN 196-2	Category B \leq	7.00~% by mass
		Category C \leq	9.00~% by mass
Chloride content $[Cl^-]$	EN 196-2	\leq	0.10 % by mass
Sulphate content $[SO_3]$	EN 196-2	\leq	3.00~% by mass
Free calcium oxide	EN 451-1	\leq	1.50~% by mass
Reactive calcium oxide	EN 197-1:2011	\leq	10.00~% by mass
Reactive silicon dioxide	EN 197-1	2	25.00~% by mass
Silicon dioxide $[SiO_2]$			
Aluminium oxide $[Al_2O_3]$	EN 196-2	Total \leq	70.00~% by mass
Iron oxide $[Fe_2O_3]$			
Total content of alkalis	EN 196-2	<u> </u>	5.00 % by mass
Magnesium oxide $[MgO]$	EN 196-2	\leq	4.00~% by mass
Phosphate $[P_2O_5]$	ISO 29581-2	≤	5.00~% by mass

Table 3: The chemical requirements for fly as h used in concrete according to DS/EN 450-1:2012

$\operatorname{Subject}$	Method	Physical Requirement			
Finonoss	EN 451-2 or	Mass proportion of ash retained	Cat. N $<$ 40 % by mass		
гшенев	EN 933-10	sieve	Cat. S $<$ 12 % by mass		
Activity index	FN 106-1	After 28 days	> 75~%		
Activity index	EN 190-1	After 90 days	> 85~%		
Initial	FN 106-3	Initial setting time for a test with 25% ash and 75% cement			
setting time	EIN 190-5	shall not be more than twice as long as a 100% cement test			

Table 4: The most important physical requirements for fly as h used in concrete according to DS/EN 450-1:2012

The standard shall be considered along with the supplementary standard for Denmark DS 2426:2011. This standard specifies some criteria for the use of SSA in concrete in Denmark

which includes that the SSA should be labelled with a declaration showing the particle density and all the chemical parameters stated in DS/EN 450-1:2012. The values must be determined on tests performed at least four times a year according to the methods in DS/EN 450-1:2012. Furthermore the loss on ignition must fall into be category A.

2.4.2 Requirements From DS/EN 206-1:2002 and DS 2426:2011

The European standard DS/EN 206-1:2002 Concrete - Part 1: Specification, performance, production and conformity along with the Danish supplement DS 2426:2011 Concrete - Materials - Rules for application of EN 206-1 in Denmark set the requirements for the amount of chloride allowed in concrete in Denmark. The requirements are listed in table 5.

	Environmental Class				
Maximum Cl^- content by mass of cement	Passive	Moderate	Aggressive	Extra	
	1 465170	Moderate	nggressive	aggressive	
Concrete not containing steel reinforcement					
or other embedded metal with the exception	1.0%	1.0%	1.0%	1.0%	
of corrosion-resisting lifting devices					
Concrete containing reinforcement or other	0.4%	0.2%	$0.2\%^{1}$	$0.2\%^{1}$	
embedded metal	0.470	0.270	0.270 7	0.270	
Concrete containing prestressing	0.9%	0.2%	$0.20\%^{1}$	0.1%	
steel reinforcement	0.270	0.270	0.270 7	0.170	

¹⁾ In some cases a tightening of the requirements meaning a maximum chloride content of

0.1% can be necessary

Table 5: The Danish requirements to maximum chloride content in concrete according to DS 2426:2011

2.4.3 Content of Micro and Macro Elements

According to DS 2426:2011 SSA can be used in concrete as long as the amount of specific elements in the concrete is not increased significantly with the addition.

The Danish Environmental Protection Agency (EPA) has a declaration [Danish EPA, 2010] on the use of waste products in construction work which states the allowed amounts of specific elements in waste products. The requirements are listed in table 6.

		Category 1	Category 2 and 3
Lead (Pb)	[mg/kg]	0 - 40	> 40
Arsenic (As)	[mg/kg]	0 - 20	> 20
Cadmium (Cd)	[mg/kg]	0 - 0.5	> 0.5
Chromium (Cr)	[mg/kg]	0 - 500	> 500
Copper (Cu)	[mg/kg]	0 - 500	>500
Nickel (Ni)	[mg/kg]	0 - 30	> 30
Zinc (Zn)	[mg/kg]	0 - 500	> 500

Table 6: Allowed content of specific elements in waste products used for construction work according to the Danish Environmental Protection Agency

Waste products in category 1 can be used without permission to construction work regarding roads, pathways, ramps, barrages, baffle walls and similar constructions. Category 2 and 3 have more restrictions for usage and are only allowed for some of the constructions mentioned before. They deviate from each other in the allowed content of other elements.

2.5 Corrosion of Reinforcement

Corrosion is defined as an electrochemical attack on any kind of metal. The formation of iron oxides on steel reinforcement also known as rusting is one of the most well-known examples of electrochemical corrosion [Geiker & Nielsen, 2008].

Both water and oxygen is necessary for the corrosion process to take place. Depending on the amount of available oxygen and water the steel will either be covered in reddish brown flakes or a green/white paste. Figure 3 shows the principle of reinforcement corrosion.



Figure 3: Principle for corrosion of reinforcement in concrete. (After figure D5.31 in [Geiker & Nielsen, 2008])

As seen in figure 3 corrosion of a reinforcement bar requires two processes. The process at the anode $(Fe \rightarrow Fe^{++} + 2e^{-})$ can only take place if the process at the cathode

The initial production of $Fe_2O_3(s)$ and $Fe_3O_4(s)$ at the anode will create a passivating layer on the entire reinforcement bar that can almost prevent further corrosion. The passivation is however characterised by slowing the corrosion process extremely down rather than actually stopping it. Alkalinity enhances the passivation tremendously, which is shown in the Pourbaix diagram for Fe in figure 4.



Figure 4: Pourbaix diagram for Fe

The Pourbaix diagram is built based on the Nernst Equation and the equilibrium potentials for the electrode reactions listed below [Hansen, 1995].

$Fe^{++}(aq)$	+	$2e^{-}$			\rightarrow	Fe(s)		
$Fe^{+++}(aq)$	+	e^-			\rightarrow	$Fe^{++}(aq)$		
$Fe_3O_4(s)$	+	$8H^+(aq)$	+	$8e^-$	\rightarrow	3Fe(s)	+	$4H_2O(l)$
$Fe_2O_3(s)$	+	$6H^+(aq)$	+	$6e^-$	\rightarrow	2Fe(s)	+	$3H_2O(l)$
$Fe_3O_4(s)$	+	$8H^+(aq)$	+	$2e^{-}$	\rightarrow	$3Fe^{++}(aq)$	+	$4H_2O(l)$
$Fe_2O_3(s)$	+	$6H^+(aq)$	+	$2e^{-}$	\rightarrow	$2Fe^{++}(aq)$	+	$3H_2O(l)$

There are two primary threats regarding corrosion of the reinforcement in concrete: carbonation and chloride ingress. Carbonation is briefly explained below whereas the theory on chloride ions is covered more thoroughly in the following section.

Carbonation decreases the alkalinity of the concrete lowering the pH to around 8 at worst. According to the Pourbaix diagram in figure 4 this might result in a degradation of the surface passivation on the steel which will then lead to corrosion of the reinforcement. Carbonation is caused by a reaction between the carbon dioxide from the air and the calcium hydroxide in the concrete:

$$CO_2 + Ca(OH)_2 \rightarrow CaCO_3 + H_2O$$

2.5.1 Chloride and Concrete

The presence of chloride ions in concrete can abolish the passivating layer despite a high alkalinity of the concrete environment. The chloride ions will pit the passivating layer thereby allowing water and oxygen to reach the reinforcement bar. The remaining passivating layer will then act as the cathode since it will appear the most precious metal and the depassivated area will be the anode.

It is not fully known what causes the chloride ions to prompt the pitting but it has been established that a low concentration of chloride ions in concrete will only cause a low rate of corrosion but the risk of corrosion is increased with the chloride concentration.

Mechanisms of Chloride Ion Transport

There are three mechanisms of chloride ion transport in concrete: diffusion, capillary absorption and hydrostatic pressure.

Diffusion is driven by concentration gradients. This means that there must be a continuous liquid phase in the concrete. The chloride ion concentration gradient then occurs when there is only a little amount of free chloride ions in the concrete and plenty outside. This method can draw the chloride ions all the way to the reinforcement bar as there will continue to be a concentration gradient from one point in the concrete to a point closer to the reinforcement.

Capillary absorption is driven by moisture gradients and it is therefore a common form of chloride ion transport for constructions that are exposed to wetting and drying cycles with water containing chloride ions. Capillary suction forces water that is encountered with a dry surface in to the pore structure. If the water contains chloride ions as e.g. seawater or melted snow from areas that have been salted the chloride will be drawn in to the concrete and even though the surface eventually will dry again the chloride ions will stay in the concrete.

The capillary forces are usually not able to transport the chloride ions very far into the concrete but the core of the structure will often still be wet and the chloride ions can therefore continue their journey towards the reinforcement bar by means of diffusion.

The transport mechanism of hydrostatic pressure is driven by pressure gradients. If a concrete structure is exposed to a hydrostatic head on one face and the water contains chloride ions then these may permeate into the concrete.

Chloride Binding

Chloride penetration into concrete is slowed significantly down when a part of the chloride ions binds to the concrete [Thomas et al., 2012]. When the chloride ions are bound to the concrete

they are no longer free in the pore solution and will therefore not help towards the degradation of the passivating layer on the reinforcement. The period before initiation of corrosion is thereby prolonged.

There are two mechanisms for chloride binding [Ipavec et al., 2013 and Thomas et al., 2012]. The physical binding occurs when the chloride ions are adsorped onto the surface of C-S-H. The chemical binding happens when chloride ions react with the cement clinker phase C_3A to form calcium chloro-aluminate, $C_3A \cdot CaCl_2 \cdot 10H_2O$ which is also known as Friedel's salt.

Previous studies have shown that various supplementary cementitious materials (SCM) such as fly ash, silica fume, slag and metakaolin all have an effect on the ability for the concrete to bind the chloride ions. The increase of C-S-H by addition of a SCM generally increases the chloride binding although with the exception when the SCM is silica fume. When using silica fume as SCM previous results have shown a reduction of chloride binding which is thought to be due to a lowered calcium/silica ratio.

The amount of alumina in the SCM has a significant effect on the chloride binding which results from [Thomas et al., 2012] clearly shows. When comparing concrete with a 0.5 ratio of water to cementitious materials there was a distinct correlation between the variation in the chloride binding and the alumina content of the SCM. Metakaolin (approximately 45% Al_2O_3) showed by far the largest chloride binding capacity. The chloride binding was also increased with fly ash and slag (10-25% Al_2O_3) although not as much as for the metakaolin. The concrete with silica fume (<0.5% Al_2O_3) showed less capacity of chloride binding than the reference concrete.

Furthermore results from desorption tests [Thomas et al., 2012] have shown that a portion of the bound chloride ions is irreversibly bound to the concrete as they are not released when the concrete is exposed to a chloride-free solution. The amount of irreversibly bound chloride ions does not vary much when comparing the percentage of chloride ions that remains bound for a reference concrete and concrete with respectively metakaolin and silica fume.

2.5.2 Rate of Corrosion

The Pourbaix diagram shows the equilibrium conditions. It is therefore not possible to estimate the rate of corrosion based on the diagram.

Previous research have dealt with estimation of the corrosion initiation and the rate of corrosion. [Andrade & Buják, 2013] measures the electrical potential, E_{corr} , and considers either the steel to be passive or the concrete to be dry when E_{corr} is more positive than $-350mV_{SCE}$. Hence active corrosion is expected to take place when E_{corr} is below $-350mV_{SCE}$.

There is however an incongruence between that limit and [Klinghoffer, 1993] that gives the same limit value but measured with a CSE electrode.

Measurements of corrosion potential is also used by [Prieto et. al., 2012] as a qualitative technique to estimate whether a rebar embedded in mortar is either passive or active with regard to corrosion. It is found to be a method that is highly sensitive to the dampness of the mortar which can result in fluctuating results. On the limits for active and passive stage [Prieto et al., 2013] used E_{corr} more negative than 231mV measured with a SSCE electrode to indicate active corrosion with a probability of 90%.

3 Materials and Methods

An overview of the tests that have been conducted is given below. All the tests are described more thoroughly in the following sections.

- Characterisation of LSA-depot
- Material testing
 - Compressive strength
 - Density and porosity
 - Chloride content in mortar
 - Leaching
- Chloride diffusion
- Chloride binding
 - Screening tests
 - Chloride binding into LSA-depot
 - Chloride binding into mortar samples with LSA-depot
 - Chloride binding into reference mortar
- Rate of corrosion
 - Electrical potential tests
 - Visual tests

3.1 LSA-depot

The SSA was sampled on 11th March 2013 and it has been stored in a plastic container in the same indoor environment since. This has given approximately the same conditions for the SSA in all the conducted tests which is confirmed by measuring the water content twice with six weeks in between the two tests.

For most tests the SSA has been milled at 1100 revolutions for 20 seconds. Whenever unmilled SSA has been used it is clearly stated.

3.2 Characterisation of LSA-depot

Table 7 shows which tests were performed in order to characterise the properties of the SSA and the purpose of each test. After the table each test is mentioned with a reference to the test method and any additional information or deviation from the test method is stated.

Test	Scope
рН	Determined to gain an insight into the chemical properties of the SSA. A high pH value is desirable in order to minimise the risk of corrosion.
Conductivity	Determined to gain an insight into the chemical properties of the SSA.
Water content	The water content plays an important role in the w/c ratio of the mortar or concrete.
Loss on ignition	The loss on ignition expresses the amount of organic material in the SSA. There are requirements with regard to the loss on ignition for SSA used in concrete as described in section 2.4.1.
Water solubility	The amount of solid material in a mortar with SSA is affected by the amount of material that is dissolved in water during the casting.
Micro/macro elements	The amount of micro and macro elements in the SSA is measured by use of ICP analysis.
Water soluble anions	The amount of water soluble anions in the SSA is measured by use of Ion Chromatography.
Grain size	The fineness of the SSA is determined.
SEM analysis	The SEM analysis provides magnified pictures of the SSA which makes it possible to see the shape of the particles.

Table 7: Tests used to characterise the properties of LSA-depot

3.2.1 pH

The pH value was measured with triplicate determination as described in Appendix A.1.

3.2.2 Conductivity

The conductivity was measured with triplicate determination as described in Appendix A.2.

3.2.3 Water Content

The water content was measured with triplicate determination as described in Appendix A.3. The test was performed twice with six weeks in between. The first test was made with unmilled ash whereas the second test was made with milled ash. The ashes had been stored identically.

3.2.4 Loss on Ignition

The loss on ignition was measured with triplicate determination as described in Appendix A.4.

3.2.5 Water Solubility

The water solubility was measured with duplicate determination as described in Appendix A.5.

3.2.6 Content of Micro and Macro Elements

The content of micro and macro elements in the SSA is determined by an ICP analysis. ICP analysis was performed on three samples that were prepared as described in Appendix A.6. The ICP analysis was performed by a laboratory technician.

Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) is a technique where inductively coupled plasma is used to produce excited atoms and ions in the tested sample. Excited atoms are atoms that are in an unstable state due to an extraordinary energy induction e.g. from a plasma. These atoms and ions will then emit electromagnetic radiation and since each element has a characteristic wavelength it is possible to determine the amount of each element in the sample.

LSA-depot is investigated for the content of Al, As, Ba, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, P, Pb, V and Zn.

3.2.7 Water Soluble Anions

The amount of water soluble anions in the SSA is determined by an ion chromatography. Ion chromatography was performed on three samples that were prepared as described in Appendix A.7. The ion chromatography was performed by a laboratory technician.

The concentration of each anion is determined based on their retention time. The sample solution is passed through a pressurised chromatographic column where the ions are absorbed by the column. An eluent i.e. an ion extraction liquid is then run through the column and the absorbed anions start to separate from the column. Chloride ions will separate first, then nitrate ions and at last sulphate ions. The retention time then determines the ionic concentration in the sample.

LSA-depot is investigated for the concentrations of the three anions Cl^- , NO_3^- and SO_4^{2-} .

3.2.8 Grain size

The grain size was measured with duplicate determination as described in Appendix A.8. The tests were performed with both milled and unmilled LSA-depot.

3.2.9 SEM Analysis

Scanning Electron Microscope (SEM) analysis is done by scanning a small sample in vacuum with a focused beam of electrons. The atoms in the sample interact with the electrons and produce signals that the SEM apparatus detects and converts into images with great depth.

3.3 Material Testing

The material testing was primarily conducted on mortar specimens. Therefore the procedure of casting the mortar specimens is covered first and then the procedure of the actual tests are described.

3.3.1 Casting of Mortar Specimens

The mortar specimens were cast and tested according to the standard DS/EN 196-1:2005 *Methods of testing cement - Part 1: Determination of Strength.* Deviations made from the requirements prescribed in the standard are mentioned below.

The specimens were denominated A, B and C with the B-specimen being the middle one in the mould.

The used mortar recipes are shown in table 8. LSA-depot had been milled for all specimens containing SSA.

	Cement	LSA-depot	Sand	Water
	[g]	$[\mathbf{g}]$	[g]	[g]
Reference	450.00 ± 0.10	-	1350.00 ± 0.10	225.00 ± 0.10
10% cement replacement	405.00 ± 0.10	$45.00~\pm~0.10$	1350.00 ± 0.10	225.00 ± 0.10
5% sand replacement	450.00 ± 0.10	$67.50~\pm~0.10$	1282.50 ± 0.10	225.00 ± 0.10

Table 8: The used recipes for the mortar specimens

Some of the mortar samples with LSA-depot seemed more dry than the reference samples and extra water was therefore added during the 90 seconds pause in the mixing. The amount of extra water added was an estimation of what would be adequate to obtain the same workability of the mortar compared to the reference samples. The exact amounts are noted in Appendix B.3.

Recipe	No.	Reinforced	Purpose
5% sand replacement, 78 days	3	No	Chloride diffusion test
10% cement replacement, 78 days	5	No	Chloride diffusion test
Reference, 78 days	6	No	Chloride diffusion test
	8	No	Compression strength test
Deference 78 days			Chloride content in mortar
Reference, 78 days	0		Leaching
			Chloride binding
		No	Compression strength test
10% cement replacement, 78 days	9		Chloride content in mortar
ro days			Leaching
5% sand replacement,			Compression strength test
78 days	10	No	Leaching
Reference, 29 days	11	Yes	Rate of corrosion, visual test
10% cement replacement, 29 days	12	Yes	Rate of corrosion, visual test
Reference, 29 days	13	Yes	Rate of corrosion, electrical potential test
10% cement replacement, 29 days	14	Yes	Rate of corrosion, electrical potential test
Reference with 3% salt water, 29 days	15	Yes	Rate of corrosion, electrical potential test
10% cement replacement with 3% salt water, 29 days	16	Yes	Rate of corrosion, electrical potential test
Reference with 3% salt water, 29 days	17	Yes	Rate of corrosion, visual test
10% cement replacement with 3% salt water, 29 days	18	Yes	Rate of corrosion, visual test
10% cement replacement, 29 days	19	No	Chloride binding
10% cement replacement with washed LSA-depot, 29 days	20	No	Chloride binding

Table 9: An overview of the mortar specimens that have been cast and the purpose of each sample $% \mathcal{A}$

The tolerances on the dimensions of the mortar specimens were a little larger than specified in DS/EN 196-1:2005. The prescribed and actual dimensions and tolerances of the specimens used for compressive strength tests are listed in table 10.

	Height	Width	Length
	[mm]	[mm]	[mm]
Prescribed in DS/EN 196-1	$40.1~\pm~0.1$	$40.0~\pm~0.2$	$160.0~\pm~1.0$
Specimens for compressive strength tests	$40.0~\pm~0.1$	$40.0~\pm~0.8$	$160.0~\pm~1.0$

Table 10: The dimensions and tolerances for the mortar specimens

Temperature and Humidity

The actual conditions for the specimens deviated a little from the prescription in the standard. Table 11 shows the prescribed and actual temperatures and humidities during casting, storage and curing. The temperature and humidity was measured every 30 minutes during one month with a TGU-4500 datalogger.

		Casting	Storage before demoulding	Curing
Prescribed surrounding temperature	$[^{\circ}C]$	20 ± 2	$20~\pm~1$	-
Actual surrounding temperature		22 ± 3	$22~\pm~3$	-
Prescribed relative humidity		≥ 50	≥ 90	-
Actual relative humidity	[%]	$50.9~\pm~7.1$	$50.9~\pm~7.1$	-
Prescribed water temperature	$[^{\circ}C]$	-	-	$20~\pm~1$
Actual water temperature	$[^{\circ}C]$	-	-	22 ± 3

Table 11: The prescribed and actual temperatures and humidities

Curing

The specimens were demoulded as close to 24 hours after moulding as possible ensuring the conditions to be equal. The exact times for casting and demoulding each specimen are noted in Appendix B.

The specimens were cured horizontally in water as specified in the standard although with the exception that they were placed on top of each other meaning that the water did not have free access to all six sides.

The curing period for each sample can be seen in table 9.

Reinforcement

A number of samples were cast with reinforcement. A single reinforcement bar with a diameter of approximately 8 mm was placed in the center of each specimen. The bar was held in place by two pieces of wood as shown in figure 5 and the reinforcement bar therefore peeped out in both ends of the specimen.



Figure 5: Mould with reinforcement bar held in place by pieces of wood

3.3.2 Compressive Strength Tests

The compressive strength of mortar with LSA-depot has previously been investigated thoroughly which is documented in reports by [Rem, 2013] and [Carlsen & Petersen, 2013]. The strength is however one of the most important and critical issues when considering replacement of sand and especially cement with SSA in concrete or mortar and therefore the compressive strength was determined for test specimens equal to those tested for chloride diffusion.

The tests to determine the compressive strength of the mortar were done according to DS/EN 196-1:2005 with the exception that the auxiliary plates were only 5 mm thick instead of the 10 mm specified in the standard.

The compressive strength was determined for three different samples: a reference, a 5% sand replacement and a 10% cement replacement sample. They were cured for the same amount of time as the samples for the chloride diffusion test which was 78 days.

3.3.3 Density and Porosity

There is a clear correlation between the density and porosity of concrete. The diffusion of substances into concrete is strongly affected by the porosity and therefore also of the density of the concrete. This makes it interesting to compare the density and porosity for mortar with cement replacement by LSA-depot with reference mortar.

The density and porosity of both reference samples and 10% cement replacement samples were determined by [Rem, 2013] as described in Appendix A.12. The experiments were conducted with the newest sampling of LSA-depot.

The results from the previous research were used and compared to results for other ashes in this report.

3.3.4 Chloride Content in Mortar

The content of chloride in LSA-depot was determined during the characterisation of the SSA but it is also interesting to establish whether concrete with LSA-depot complies with the requirements regarding chloride content in concrete stated by DS 2426:2011.

The chloride content was determined by crushing mortar to powder with a hammer before leaving it to dry in an oven heated to 105° C over night. The chloride was then extracted by use of the test method described in Appendix A.11 and then measured by use of titration with

silver nitrate as described in Appendix A.9.

The test was conducted with duplicate determination for both a 10% cement replacement sample and a reference sample. Both had been cured for 78 days.

Since the requirements on chloride content are about concrete instead of mortar the reference sample was tested to give a frame of reference for the 10% cement replacement sample.

3.3.5 Leaching

One important step in the process of determining whether LSA-depot is suitable to use in concrete is to investigate if the SSA itself or mortar with the SSA leaches elements that are harmful to the environment. It is essential that neither pure LSA-depot nor LSA-depot used in concrete can constitute a risk to nature. There is always a possibility that a construction made of concrete at some point will be torn down. In that case at least a part of the concrete will be pulverised and left in the nature where rain will do its best at washing any harmful elements out of the concrete powder. The powder represents the worst case scenario when the SSA is used in concrete which is the reason that the leaching test is conducted with finely crushed mortar. Furthermore the leaching of the pure LSA-depot is also tested as that pose as a threat before being added to the mortar.

Leaching of LSA-depot

The leaching of LSA-depot was measured with triplicate determination as described in Appendix A.10.

Leaching of Mortar with LSA-depot

The leaching of mortar was determined for three different samples: 5% sand replacement, 10% cement replacement an a reference sample. They had been cured for 78 days and were then pulverised by use of a hammer. The pulverised mortar was dried at $105^{\circ}C$ and the leaching was then measured with triplicate determination as described in Appendix A.10.

3.4 Chloride Diffusion

The content of chloride in LSA-depot was determined in the characterisation of the SSA. The next step was naturally to investigate if the replacement of cement or sand by LSA-depot has an influence on the diffusion of chloride into mortar. Diffusion is a significant cause of chloride ingress in concrete making it a very important subject to test.

The chloride ingress was checked with the hope that the diffusion was not increased when replacing cement with LSA-depot. Furthermore the chloride diffusion was investigated in specimens where sand was replaced with LSA-depot to see if the chloride ingress could be slowed down by replacing sand with LSA-depot. If so it would justify that replacement even though it does not meet the overall original scope of reducing the cement production for concrete. The tests were made with 3% salt water which is chosen based on the fact that the average

salinity of the oceans on the earth is estimated to be 3.5% [European Space Agency, 2013].

The chloride diffusion was tested with three different samples: a reference, a 5% sand replacement and a 10% cement replacement sample. They were cured for 78 days before being painted with three layers of epoxy on the four long sides. This ensured that the chloride would only penetrate through the ends of the specimens. After the painting was completed the samples were left completely soaked in distilled water for approximately three hours. It was afterwards confirmed that the specimens were waterlogged by weighing them three times during 30 minutes. The specimens were then placed horizontally in a container holding distilled water with 3%NaCl. The container was placed in the same environment as described for curing in section 3.3.1.



Figure 6: Samples painted with epoxy on the long sides

One specimen from each sample was taken out of the salt water after seven weeks. The epoxy paint was scraped off and the specimens were then sliced as shown in figure 7. The slicing was performed with a hammer and chisel making the slices a little rough. The alternative to hammer and chisel was using a wet saw and thereby potentially washing the chloride ions out of the concrete which was highly unwanted.



Figure 7: Subdivision of the specimens for investigation of chloride diffusion

Each slice was crushed very finely and placed in a separate petri dish. The petri dishes were then left in an oven heated to 105° C over night.

The chloride was then extracted in all the slices by use of the test method described in Appendix A.11. The amount of chloride in each slice was then determined by use of titration with silver nitrate as described in Appendix A.9.

3.5 Chloride Binding

For reinforced concrete constructions exposed to chloride, an admixture that could help prevent the corrosion of the reinforcement by binding the chloride that inevitably will penetrate the concrete sooner or later, would be a huge asset. With that in mind it was investigated if either raw LSA-depot or hydrated LSA-depot can bind chloride ions. Mortar samples with LSA-depot were used to test the hydrated SSA but since that method could not unveil whether the chloride was bound to the cement or the SSA tests were also made with reference mortar.

At the moment there are no standard test methods to determine the effect of SSA on the chloride binding of cement pastes. Screening tests were therefore conducted first in order to establish the exact test method used for the following experiments. The used salt water concentrations were decided partly based on similar experiments conducted by [Thomas et al., 2012] and partly based on assumed realistic NaCl exposures for concrete constructions.

3.5.1 Screening Tests

Screening Test I

Initially the chloride binding to the milled unwashed LSA-depot was tested at four different NaCl concentrations for one week. This was done by mixing 5.00 g of LSA-depot with 15 mL of respectively 1%, 3%, 10% and 20% saltwater. The samples were placed on a laboratory agitating table. As a reference one sample with 5.00 g of LSA-depot and 15 mL pure distilled water was made and given the same conditions.

After one week the samples were filtered with suction and the amount of chloride in the fluid was measured by use of titration with silver nitrate. The test method for titration with silver nitrate is described in Appendix A.9.

Screening Test II

The second screening test was made with LSA-depot that was both milled and washed. The chloride binding was tested with the exact same method as screening test I but only with the following three NaCl concentrations: 1%, 3% and 10%. Once again a sample with pure distilled water was made as a reference.

3.5.2 Chloride Binding into LSA-depot

The test method was determined based on the results from the screening tests and was very similar to those. The 10% and 20% concentrations of NaCl were eliminated due to problems with non-available or inaccurate results when determining the chloride content of the sample solutions.

The chloride binding into the pure SSA was tested with both milled unwashed LSA-depot and milled washed LSA-depot. Four solutions with different concentrations of NaCl were used: 0%, 1%, 3% and 5%. Tests were made with four different test periods being two, four, six and 16 weeks prompting a total of 32 samples.

An overview of all the samples made can be seen in table 12.

The solutions were made with distilled water and respectively 0%, 1%, 3% and 5% NaCl. The samples were made with each 5.00 g of LSA-depot mixed with 15 mL of solution in plast vials that were placed on an agitating table for the entire test period. Afterwards the samples were filtered with suction and the amount of chloride in the fluid was measured by titration with silver nitrate as described in Appendix A.9.

	0% NaCl	1% NaCl	3% NaCl	5% NaCl	
2 weeks	A1: Unwashed SSA	A2: Unwashed SSA	A3: Unwashed SSA	A4: Unwashed SSA	
	B1: Mortar with unwashed SSA	B2: Mortar with unwashed SSA	B3: Mortar with unwashed SSA	B4: Mortar with unwashed SSA	
	C1: Washed SSA	C2: Washed SSA	C3: Washed SSA	C4: Washed SSA	
	D1: Mortar with washed SSA	D2: Mortar with washed SSA	D3: Mortar with washed SSA	D4: Mortar with washed SSA	
	E1: Reference mor- tar	E2: Reference mor- tar	E3: Reference mor- tar	E4: Reference mor- tar	
	A5: Unwashed SSA	A6: Unwashed SSA	A7: Unwashed SSA	A8: Unwashed SSA	
	B5: Mortar with unwashed SSA	B6: Mortar with unwashed SSA	B7: Mortar with unwashed SSA	B8: Mortar with unwashed SSA	
veeks	C5: Washed SSA	C6: Washed SSA	C7: Washed SSA	C8: Washed SSA	
4 w	D5: Mortar with washed SSA	D6: Mortar with washed SSA	D7: Mortar with washed SSA	D8: Mortar with washed SSA	
	E5: Reference mor- tar	E6: Reference mor- tar	E7: Reference mor- tar	E8: Reference mor- tar	
	A9: Unwashed SSA	A10:Unwashed SSA	A11:Unwashed SSA	A12:Unwashed SSA	
	B9: Mortar with unwashed SSA	B10: Mortar with unwashed SSA	B11: Mortar with unwashed SSA	B12:Mortar with unwashed SSA	
veeks	C9: Washed SSA	C10: Washed SSA	C11:Washed SSA	C12:Washed SSA	
6 v	D9: Mortar with washed SSA	D10:Mortar with washed SSA	D11:Mortar with washed SSA	D12:Mortar with washed SSA	
	E9: Reference mor- tar	E10: Reference mor- tar	E11:Reference mor- tar	E12: Reference mor- tar	
	A13:Unwashed SSA	A14:Unwashed SSA	A15:Unwashed SSA	A16:Unwashed SSA	
16 weeks	B13:Mortar with unwashed SSA	B14: Mortar with unwashed SSA	B15:Mortar with unwashed SSA	B16:Mortar with unwashed SSA	
	C13: Washed SSA	C14: Washed SSA	C15:Washed SSA	C16:Washed SSA	
	D13:Mortar with washed SSA	D14:Mortar with washed SSA	D15:Mortar with washed SSA	D16:Mortar with washed SSA	
	E13: Reference mor- tar	E14: Reference mor- tar	E15:Reference mor- tar	E16: Reference mor- tar	

Table 12: An overview of the tests of chloride binding to both pure LSA-depot and mortar with LSA-depot.
3.5.3 Chloride Binding into Mortar with LSA-depot

Mortar samples with 10% cement replacement of respectively unwashed milled LSA-depot and washed milled LSA-depot were cast as described in section 3.3.1. After 29 days of curing one specimen from each sample was pulverised as seen in figure 8.

The test method used to determine the chloride binding into mortar with LSA-depot was similiar to that for the pure LSA-depot. Tests were conducted with four different solutions of salt water being (0%, 1%, 3% and 5%) and four different test periods (two, four, six and 16 weeks) prompting a total of 32 samples. An overview of those can be seen in table 12.

5.00 g crushed mortar was mixed with 15 mL of solution in plast vials and placed on an agitating table for the entire test period. The samples were filtered with suction afterwards and titrated with silver nitrate to determine the amount of chloride in the fluid. The method for titration with silver nitrate is described in Appendix A.9.



Figure 8: Pulverisation of mortar specimen with 10% cement replacement



Figure 9: Samples prepared with respectively LSA-depot and crushed mortar in salt water solutions

3.5.4 Chloride Binding into Reference Mortar

Reference mortar samples without any SSA were cast as described in section 3.3.1 and cured for 78 days.

The test method was the exact same as used for the mortar with LSA-depot and an overview of the samples can be seen in table 12.

3.6 Rate of Corrosion

When the passivating layer on the reinforcement in concrete has been destroyed a repair or replacement of the structure will at some point be inevitable. But the rate at which the corrosion of the reinforcement happens is important since it will provide a shorter or longer period of time from the initiation of the corrosion to the point where a renovation is crucial.

Mortar samples were made to investigate the rate of corrosion in mortar respectively with and without SSA. Due to limited time the samples were tested under the worst imaginable conditions which speeded up the process of corrosion.

The worst-case scenario in the real world is a construction such as a bridge pier that is placed in seawater where either waves or tide causes the construction to alternately wet and dry. These conditions were imitated by exposing the samples to salt water for seven days and then leaving them to dry out for seven days repeating the cycle several times.

As described in section 3.3.1 a number of reinforced mortar specimens were cast. Two different test methods were used to illustrate the rate of corrosion and both of them are described below.

Altogether eight samples with reinforcement were made, four of these were cast using 3% saltwater instead of distilled water. Table 13 shows the samples and the test method used for each of them.

Sample no.	Type	Water	Test method
11	Reference	Distilled	Visual Test
12	10~% cement replacement	Distilled	Visual Test
13	Reference	Distilled	Electrical Potential Test
14	10~% cement replacement	Distilled	Electrical Potential Test
15	Reference	3~% NaCl	Electrical Potential Test
16	10~% cement replacement	3~% NaCl	Electrical Potential Test
17	Reference	3~% NaCl	Visual Test
18	10~% cement replacement	3~% NaCl	Visual Test

Table 13: The composition and purpose of the reinforced mortar samples

3.6.1 Electrical Potential Test

The mortar samples were cured for 29 days under conditions described in section 3.3.1. The specimens were then painted with two layers of epoxy in one end so that both the reinforcement bar and the mortar were completely covered. This was done to ensure that the distance from the surrounding environment to the reinforcement bar was kept the same and to prevent the initiation of the corrosion where the reinforcement bar peeps out. After the painting was completed the specimens were left completely covered by distilled water for at least three days. The specimens were placed upright in a container with the epoxy painted end downwards and supported by small blocks underneath. Distilled water with 3% NaCl was added until approximately 0.5 cm from the top of the mortar samples and the level of the water was marked on the container for later refilling. Two different containers were used for the samples cast with and without saltwater.

The electrical potential was measured by use of a potentiometer. It was connected to a reference electrode immersed in the saltwater and to the reinforcement bar of a specimen by use of an alligator clip.

The first measurement was taken after approximately two hours and confirmed to be stable by two extra measurements after 10 and 20 minutes respectively.

The specimens were left in the saltwater for seven days and the electrical potential was measured again before taking the specimens out of the water. They were left at normal indoor conditions for seven days and then put back in the saltwater as previously. The test cycle of seven days in water and seven days of drying out was repeated for 10 weeks and then all the samples were left in salt water for six weeks before ending the experiment with one week of drying. For the specimens made with NaCl the first test cycle was limited to eight weeks but the rest was identical.

The salt water was changed when the specimens were put back after the seven days of drying.



Figure 10: The test setup for measuring the electrical potential

Prior to measuring the electrical potential sandpaper was used to remove the rust on the reinforcement bars in order to obtain stable and reliable results. A complete removal of the rust was however not possible.



Figure 11: Specimens for both electrical potential tests and visual tests placed in salt water



Figure 12: Specimens that have just been taken out of the salt water to dry out for seven days

3.6.2 Visual Test

The test method for the visual test was very much alike the one for the electrical potential test. The preparation of the specimens was the same except both ends of the visual test specimens were painted with epoxy as there was no need to keep the reinforcement bar accessible. The specimens were placed upright in the same container with the electrical potential test specimens to ensure that they were exposed to the same conditions.

The visual test followed the same test cycle as the electrical potential test including the six weeks they were left in salt water. The test period varied for the specimens as the first specimen was taken out after 6 weeks and cut across with a wet saw to expose the reinforcement bar for corrosion investigation. At the end of the test period the remaining specimens were both cut across like the first one and the reinforcement bar was also exposed by removal of mortar with a hammer and a chisel making it possible to see a larger part of the rebar.

4 Results and Discussion

4.1 Assessment of LSA-depot

Table 14 shows the results from five of the tests made in order to characterise LSA-depot from *Renseanlæg Lynetten*. The results are shown as the mean \pm the standard deviation. All of the results from the tests performed in this project can be seen in Appendix B.1.

As mentioned previously Carlsen & Petersen have also worked with LSA-depot primarily focusing on characterisation of the SSA and the strength of mortar with either sand or cement replacement [Carlsen & Petersen, 2013]. The portion of LSA-depot worked with previously was sampled 15th June 2012 which was approximately nine months prior to the sampling made for this project. It is however relevant to investigate and compare variations between the present results and the results achieved by Carlsen & Petersen and these are therefore included in the table.

The results for the characterisation are also compared to results from similar tests conducted with LSA-new. LSA-new is sewage sludge ash from Lynetten Renseanlæg as well but sampled right after the treatment process was finished and has therefore not been stored in the depot. The previous results can be seen in Appendix C.

The results below are discussed separately in the following sections.

		LSA-depot present results	LSA-depot previous results	LSA-new previous results
Sampling date		11th March 2013	15th June 2012	14th June 2012
pH	[-]	12.6 ± 0.01	8.3 ± 0.03	12.4 ± 0.05
Conductivity	$[\mathrm{mS/cm}]$	9.97 ± 0.82	5.59 ± 0.11	7.81 ± 0.02
Water content	[%]	3.93 ± 0.07	14.4 ± 0.09	0.09 ± 0.03
Loss on ignition	[%]	0.47 ± 0.04	0.16 ± 0.00	0.25 ± 0.05
Water solubility	[%]	5.6 ± 0.79	0.84	5.12

Table 14: Results from the present characterisation of LSA-depot compared to previous results for LSA-depot from [Carlsen & Petersen, 2013] and results for LSA-new from [Reiff & Meldgaard, 2013]

It is noted that there are generally quite large differences between the two samplings of the LSA-depot. In previous studies with LSA-depot (Rem, 2013) there was also noted a great variation in the color of the two samplings of SSA which can be seen in figure 13. A full characterisation of the newest sampling was however not conducted in the previous research which makes it even more important to compare the two samplings.



Figure 13: Left: A great difference in the color of the two samplings of LSA-depot [Rem, 2013]. Right: The difference in color of LSA-depot is also visible in mortar samples. From top: reference sample, 10% cement replacement with the newest sampling and 10% cement replacement with the previous sampling [Rem, 2013]

It is unknown whether this is due to a difference in where in the depot the samplings have been made from or it can be explained by a difference in the amount of time the SSA was in the depot. Both portions have been stored for at least two years but a more precise indication of the storage period is not known. It does however emphasise that a characterisation of LSA-depot is highly necessary before conducting experiments with it. Also a method for homogenisation of the content in the entire depot is needed before an industrial usage can be considered. The present results for LSA-depot are very similar to those achieved for LSA-new. There is only one major difference being the water content which can probably be explained by the fact that LSA-new was sampled right after the incineration.

A comparison of the results for the previous sampling of LSA-depot shows some irregularities. The conductivity and water solubility are usually associated but this does not seem to be the case for these results as the difference among them is much larger than for the present sampling of LSA-depot or LSA-new. The reason for the irregular results is however unknown. The results for the individual tests are now discussed separately.

4.1.1 pH

There is a great difference in the measured pH values for the two samplings of LSA-depot. The result from the present tests is significantly better than the previous one. This is based on the effect an alkaline environment in the concrete has on reinforcement. The high pH is normally caused partly by the formation of $Ca(OH)_2$ during hydration and partly by the release of alkali metal ions from the cement.

By replacing some of the cement in concrete with SSA less calciumhydroxide will be formed which can cause a lower pH. A high pH value of the SSA is therefore a great advantage as it will contribute to the pacifying environment around the reinforcement.

The difference between the results for the two samplings of LSA-depot is however not so good as a pH of around only 8 for LSA-depot could possibly create problems if used in reinforced concrete.

4.1.2 Conductivity

The conductivity gives an indication of the amount of soluble ions in the SSA. The smaller amount the better as it equals more solid material when the SSA is used in concrete. There is a bit of a difference between the results from the previous and present tests with the first sampling of LSA-depot providing the best results. The result for LSA-new is placed in between indicating that the storage in the depot might not change much about the conductivity of the SSA.

The conductivity for LSA in general is however much better than that for co-combustion ash from straw and wood which is found to be 146.1 mS/cm for unwashed ash and 10.2 mS/cm for washed ash [Madsen & Fjelstrup, 2013]. The present result for LSA-depot is quite close to that of the washed ash but this is when the SSA is not washed.

4.1.3 Water Content

The water content for the latest sampling of LSA-depot is found to be very stable. Tests were performed with six weeks in between two triplicate determinations to confirm that the storage of LSA-depot had given approximately the same conditions for the SSA in all the conducted tests throughout the project. The mean value of the water content was the same for the first three tests and the last three tests and the standard deviation calculated for all test results was as small as 0.07%. This is a solid confirmation of the assumed stable storage conditions.

The water content measured in the present tests is much lower than what was found in the previous tests for LSA-depot. It could be necessary to account for a high water content in the SSA when determining the w/c-ratio of the mortar. But since it is not known if the water in the SSA is bound in some way or free to react it is difficult to determine how to account for it. A low water content is therefore much preferred as it can almost be neglected which makes it reasonable to not account for it when determining the w/c-ratio. The water content of LSA-new is almost non-existent which makes good sense considered that the SSA was sampled right after the incineration.

4.1.4 Loss on Ignition

The loss on ignition measured for LSA-new and both samplings of LSA-depot are very low which means that the content of organic material in the SSA is miniscule. According to section 2.4.1 the allowed loss on ignition for SSA used in concrete is 5.00% and it is noted that all three samplings of SSA with a maximum measured loss on ignition of 0.47% by far complies with that criteria.

4.1.5 Water Solubility

The water solubility for the two samplings of LSA-depot varies quite a lot with the result from the previous test being significantly better than the present result. The result for LSA-new is however really close to the results from the present test of LSA-depot. The lesser the water solubility the more solid material will be kept in a mortar made with the SSA which has an influence on both strength and porosity.

When comparing the difference in the results for the water solubility with the variations in the other results there is a trend towards that the SSA from first sampling of LSA-depot might have been washed out more during the storage period. This theory is based on the fact that both the pH value, the conductivity and the water solubility for the first sampling are substantially

smaller than the results obtained with both the latest sampling of LSA-depot and the sampling of LSA-new. It fits well with the water content of the first sampling being much higher than both LSA-new and that of the most recent sampling of LSA-depot.

4.1.6 Content of Micro and Macro Elements

Table 15 shows the relevant results from the ICP analysis. The results are shown as the mean \pm the standard deviation and can also be found in Appendix B.1.

The results from the ICP analysis are first discussed and compared to results from previous tests made with LSA-depot and LSA-new. Afterwards the results are compared to the criteria from the standards mentioned in section 2.4.3.

The results taken from [Carlsen & Petersen, 2013] and [Reiff & Meldgaard, 2013] can be seen in Appendix C.

		LSA-depot present results	LSA-depot previous results	LSA-new previous results
Sampling date		11th March 2013	15th June 2012	14th June 2012
Phosphorus, P	[mg/kg]	$68,300 \pm 1450$	-	$161,\!000\pm12,\!000$
Magnesium, Mg	[mg/kg]	$14,200 \pm 416$	-	-
Zinc, Zn	[mg/kg]	2270 ± 66.7	2810 ± 117	3060 ± 222
Copper, Cu	[mg/kg]	507 ± 7.58	694 ± 6.49	711 ± 5.65
Lead, Pb	[mg/kg]	98.8 ± 5.44	99.5 ± 1.08	102 ± 2.15
Chromium, Cr	[mg/kg]	44.9 ± 0.40	28.6 ± 0.61	29.7 ± 0.66
Nickel, Ni	[mg/kg]	37.8 ± 0.14	35.4 ± 0.58	35.2 ± 0.72
Arsenic, As	[mg/kg]	3.54 ± 0.67	-	-
Cadmium, Cd	[mg/kg]	2.20 ± 0.12	2.16 ± 0.09	2.21 ± 0.05

Table 15: Results from the present characterisation of LSA-depot compared to previous results for LSA-depot from [Carlsen & Petersen, 2013] and results for LSA-new from [Reiff & Meldgaard, 2013]

The results are generally very reliable with only minor standard deviations. For some of the elements there are huge variations among the three SSAs which is discussed further below. The first thing that is noted when comparing the results from the three samplings of SSA is the major difference in phosphorus (P) content between LSA-new and the newest sampling of LSA-depot. Unfortunately no information on the phosphorus content in the previous sampling of LSA-depot exists.

There are no limits on the amount of phosphorus in concrete but as it is an essential yet limited resource for the agriculture the content is interesting to know with an eye to extracting the phosphorus [Ottosen et al., 2013b]. There is however a limit on the content of phosphate (P_2O_5) in fly ash used in concrete according to DS/EN 450. P_2O_5 can be measured by XRF-analysis following ISO 29581-2. A good consistency between P content measured by ICP and phosphate content measured by XRF has previously been seen for similar SSAs (Ottosen et al., 2013) and the conversion can be found in Appendix B.1.

The phosphate content in LSA-depot is calculated to be 15.6% by mass which is more than three times the allowed amount (5.00% by mass). It is however possible to extract the phosphorus with the method invented by [Ottosen et al., 2013b] and the phosphate content is therefore not a major problem.

The difference in phosphate content between LSA-depot and LSA-new is naturally very distinct with the content of phosphate in LSA-new reaching approximately 37% by mass. Since it will be necessary to extract phosphorus from both SSAs before they can be used in concrete the variation is not frightening.

Results for the magnesium content are only available for the newest sampling of LSA-depot and a comparison between the SSAs is therefore not possible. DS/EN 450-1:2012 has a requirement on the allowed amount of magnesium oxide in fly ashes for concrete and the magnesium content is therefore converted into 2.35% MgO which is shown in Appendix B.1. Compared to the allowed content of 4.00% MgO it is noted that LSA-depot is well within the limits for fly ashes.

	LSA-depot present results	LSA-depot previous results	LSA-new previous results
Sampling date	11th March 2013	15th June 2012	14th June 2012
Lead, Pb	Cat. 2 and 3	Cat. 2 and 3	Cat. 2 and 3
Arsenic, As	Cat. 1	-	-
Cadmium, Cd	Cat. 2 and 3	Cat. 2 and 3	Cat. 2 and 3
Chromium, Cr	Cat. 1	Cat. 1	Cat. 1
Copper, Cu	Cat. 2 and 3	Cat. 2 and 3	Cat. 2 and 3
Nickel, Ni	Cat. 2 and 3	Cat. 2 and 3	Cat. 2 and 3
Zinc, Zn	Cat. 2 and 3	Cat. 2 and 3	Cat. 2 and 3

The results from the three experiments are compared to the limits for waste products used in concrete stated by the Danish Environmental Protection Agency which is shown in table 16.

Table 16: Results for the three samplings of LSA-new and LSA-depot categorised according to the criteria stated by the Danish Environmental Protection Agency for waste products used in concrete

It is noted that all the three SSAs only fall into category 1 with regard to As and Cr whereas the content of the other elements is significantly higher than the limits. There is a bit of a variation in the amount of Cr between the newest sampling of LSA-depot and the two other SSAs but they are all far beneath the criterion making the difference unimportant. The allowed amount of Pb is 40 mg/kg but the SSAs are quite constant around 100 mg/kg putting them into the category with very restricted usage.

Both the content of Ni and Cd are very equal for all three SSAs. There is measured approx-

imately 35-38 mg/kg Ni in the SSAs which is only slightly more than what is accepted for category 1 (30 mg/kg). The Cd content is on the other hand very far from the limit. 0.5 mg/kg Cd is accepted for category 1 meaning that the three SSAs contain up to 450% more than allowed.

The amount of Cu in the newest sampling of LSA-depot is nearly within the limits but the variation between the three SSAs is a bit disturbing. The limit is at 500 mg/kg Cu and the previous experiments gave results around 700 mg/kg. The good result from the present sampling is thereby not backed up by previous tests but instead it is standing alone against the two other quite similar results. The variation between the new and the previous samplings of SSA continues with the Zi content. With the limit for category 1 being at 500 mg/kg neither of the three SSAs are anywhere close to falling into that category. But what is a bit unsettling is the results for LSA-new and the previous sampling of LSA-depot are around 2800-3100 mg/kg whereas the present sampling of LSA-depot contained 2270 mg/kg.

Generally the variations in the results show that more analyses of different samplings are necessary in order to determine the average content of the interesting elements in the SSAs. Although if future results prove to be as fluctuating as the present ones then a homogenisation of the SSA must be engineered.

4.1.7 Water Soluble Anions

Table 17 shows the result of the ion chromatography where the amount of water soluable anions were determined. The full results can be found in Appendix B.1. Furthermore the results for the same analysis are shown for the previous sampling of LSA-depot and LSA-new allowing for a comparison. The results of these tests can be found in Appendix C.1 and C.2.

		LSA-depot present results	LSA-depot previous results	LSA-new previous results
Sampling date		11th March 2013	15th June 2012	14th June 2012
Chloride Ions, Cl^-	[mg/kg]	1800 ± 5.57	1330 ± 32.6	96.5 ± 10.0
Sulphate Ions, SO_4^{2-}	[mg/kg]	8250 ± 105	$13{,}000\pm30.6$	$10,\!200\pm222$

Table 17: Results from the present characterisation of LSA-depot compared to previous results for LSA-depot from [Carlsen & Petersen, 2013] and results for LSA-new from [Reiff & Meldgaard, 2013]

The amount of water soluble anions in the SSAs is interesting as there are requirements to both the content of sulphate and chloride from DS/EN 50-1:2012 as described in section 2.4.1. When comparing the sulphate content among the three SSAs it is noted that there is a difference but no real consistency as LSA-new lies in between the two samplings of LSA-depot. With a limit on 3.00% for fly ash all of the three SSAs with sulphate contents ranging from 0.83% to 1.30% are within the requirements.

It is however the opposite case when looking at the chloride content in the SSAs. The limit for usage in concrete is 0.10% by mass which means that only LSA-new with a content of less than 0.01% complies with the criterion. LSA-depot contain respectively 0.18% and 0.13% for

the present and the previous sampling putting both of them above the limit. The reason for the great variation between LSA-depot and LSA-new should probably be found in the outdoor storage of LSA-depot. The chloride from Øresund might bind to the SSA thereby increasing the chloride content of LSA-depot.

4.1.8 Grain Size

The grain size distribution was investigated for both milled and unmilled LSA-depot in order to see if the SSA meet to requirements for fly ash according to DS/EN 450-1:2012 as explained in section 2.4.1. Figure 14 shows the result of the experiment. The results can also be found in Appendix B.1 where the exact numbers appear.



Figure 14: Cumulated grain size distributions for both milled and unmilled LSA-depot with indication of $45 \mu m$ limit

There is a distinct variation in the grain size distributions for the milled and the unmilled LSAdepot which was expected. It is noted that the two determinations for the milled SSA are very close whereas the graphs for unmilled SSA are a bit further apart. The reason for this could be that the experiments were conducted with very small amounts of SSA so the variations in the raw LSA-depot can have a big influence on the result.

Fly ash used in concrete must fall into one of two categories stated by DS/EN 450-1:2012 defines by the amount of ash that is retained when sieved on a $45\mu m$ sieve. Category S retains maximum 12% whereas category N can retain up to 40%. The two samples of milled LSA-depot both contain 16% particles larger than $45\mu m$ putting it in category N for fly ash. The unmilled LSA-depot does however not have the demanded fineness for fly ash as the mean value for the two samples is 54% retained ash. The individual results for the two samples were respectively 46% and 63% meaning that neither of the samples lived up to the requirement. Despite the big variation in the results for the two unmilled samples further tests are not interesting since both results were above the limit.

The results for the milled LSA-depot are very promising. The 20 seconds of milling has placed the SSA in the bottom half of category N and it is therefore possible that a slightly prolonged milling could place it in category S instead. On the other hand if there is no need for the SSA to fall into category S the milling time could probably be shortened a bit.

4.1.9 SEM Analysis

The present sampling of LSA-depot and regular coal fly ash were investigated with a SEM apparatur by [Rem, 2013] and the results are displayed in figure 15 and 16. Figure 18 and 17 show similar photos for respectively a sampling of LSA-new [Andreasen & Jørgensen, 2014] and a SSA from Avedøre Rensningsanlæg [Kurt, 2014]. All the SSAs have been milled.



Figure 15: Coal fly ash magnified 400 times [Rem, 2013]



Figure 16: LSA-depot magnified 400 times [Rem, 2013]



Figure 17: LSA-new magnified 500 times [Andreasen & Jørgensen, 2014]



Figure 18: SSA from Avedøre magnified 500 times [Kurt, 2014]

When comparing all the samples it is important to note that the magnification is not the same. A distinct difference in the structure of the ashes is generally seen between the coal fly ash and the three SSAs. Some of the coal fly ash particles can however look a bit like the SSA particles although this is rejected when magnifying the coal fly ash further as seen in figure 19.

The coal fly ash particles are all spherical whilst the SSAs have very flaky structures. The LSA-ashes almost seem a bit porous and airy which gives rise to thoughts on the effect on the packing of the concrete with LSA-ash.

A comparison of the Avedøre-SSA and the LSA-ash shows that the first-mentioned seem denser. This is easiest seen when comparing LSA-new and Avedøre-SSA that are shown with the same magnification. A part of the reason could be that perhaps Avedøre-SSA have not been milled for as long as the LSA-ashes since the particles generally seem larger or the particles are just harder to crush meaning that a longer milling time is necessary to obtain the same particle size.



Figure 19: Coal fly ash magnified 1500 times [Carlsen & Petersen, 2013]

4.2 Material Testing

A number of specimens were cast for different tests e.g. compression strength tests, chloride diffusion tests etc. The data regarding the casting of these specimens can be seen in Appendix B.3.

4.2.1 Compressive Strength

As mentioned previously the influence on the compressive strength of mortar from the replacement of either sand or cement by LSA-depot has been investigated thoroughly by both [Rem, 2013] and [Carlsen & Petersen, 2013]. Additional tests were carried out in this project as a reference to the chloride diffusion experiments and the results hereof can be seen in figure 20. In Appendix B.3 the entire set of data can be seen.

The previously obtained results are not discussed in this report as they have already been evaluated profoundly.

Figure 20 shows the difference between the compressive strengths for the three mortar recipes. The standard deviations for the strengths vary a little but they are all in the range of what is acceptable.

It is observed that the reference sample and the 5% sand replacement sample have very similar compression strength. When comparing the exact numbers in Appendix B.3 it is noted that the sand replacement sample actually has the highest strength. The cement replacement sample lies a bit beneath the others which was expected based on the previous results.



Figure 20: Comparison of the compressive strengths for mortar with and without LSA-depot. Mean values with the standard deviations are shown

Extra water was added to both replacement samples which increased the w/c-ratios. As known from the Bolomey formula this has an effect on the strength of the mortar and therefore the water-to-cement ratio (w/c-ratio) and the water-to-powder-materials ratio which include LSA-depot (w/p-ratio) are shown for the three samples in table 18.

	w/c-ratio	w/p-ratio
Reference	0.50	0.50
10% cement replacement	0.57	0.51
5% sand replacement	0.51	0.45

Table 18: Water-to-cement ratios (w/c-ratios) and water-to-powder-materials ratios (w/p-ratios) for the three samples

When comparing both the w/c-ratios and the w/p-ratios in table 18 it is noted that there is a bit of a variation. The water-to-cement ratios for the reference and the sand replacement samples are almost identical which fits very well with the compression strengths. If including LSA-depot as a cementitious material the w/p-ratio is a lot lower for the sand replacement sample compared to the reference sample which is not reflected in the compression strengths. The same is the case for the cement replacement sample where the w/p-ratio is almost equal with the one for the reference sample but the strengths are quite different according to figure 20. The w/c-ratios therefore seem to be more accurate emphasising the conclusion from previous reports that LSA-depot can not replace cement without a loss of strength in the mortar.

As described in section 2.4.1 there is a requirement from DS/EN 450-1:2012 regarding the compressive strength of mortar with fly ash and although LSA-depot can not be characterised as a fly ash it still makes sense to ensure that it complies with the criteria from the standard. The requirement from DS/EN 450-1:2012 regarding the activity index is however based on mortar

with 25% cement replacement by fly ash but based on results from [Rem, 2013] where 20% of the cement was replaced with LSA-depot it was chosen to only test 10% cement replacement in this project.

Table 19 shows the activity indexes for both the sample with sand replacement and the sample with cement replacement.

	Activity Index	Compared with requirement for 28 days	Compared with requirement for 90 days
10% cement replacement, $78~\mathrm{days}$	92~%	OK	OK
5% s and replacement, $78~\mathrm{days}$	100~%	OK	OK

Table 19: Activity indexes for mortar samples with LSA-depot compared with requirements from DS/EN 450-1:2012

The samples with LSA-depot were hardened for 78 days which falls in between the two requirements from the standard. As it is seen in table 19 the mortar recipes meet both the 28 days and the 90 days criterion. This complies well with the previously obtained results for strength development in mortar with LSA-depot.

4.2.2 Density and Porosity

No new experiments have been carried out since results with the newest sampling of LSA-depot exists from [Rem, 2013]. Table 20 shows the mean values \pm the standard deviation for both the density and porosity for the tested samples. The full results from [Rem, 2013] are shown in Appendix C.3.

	Density	Porosity
	$[kg/m^3]$	[%]
Reference, 28 days	2140 ± 5.77	14.7 ± 0.33
10% cement replacement, $28~{\rm days}$	2150 ± 5.77	14.7 ± 0.57

Table 20: Results for the density and porosity of respectively a reference sample and a 10% cement replacement sample with LSA-depot from [Rem, 2013]

When comparing the results for the reference sample with the 10% cement replacement sample it is quickly noted that there is almost no difference at all. The tiny variation in densities is not significant and especially not when taking the standard deviations into consideration.

A difference among the three specimens for each sample is observed when looking at the individual results for both the reference sample and the 10% cement replacement sample. In Appendix C.3 the results for the individual specimens for density and porosity are compared in figure 53 and it is seen that the B-specimens for both the reference sample and the 10% cement replacement sample have a lower density than the A and C-specimens. This indicates that the middle specimen in the mould was generally not vibrated as well as the outer specimens. This fits in with the observations that were made during the casting of the specimens where it was often noted that the surface of the middle specimen was less smooth after the vibration compared to the outer specimens. The porosity is therefore also higher for the B-specimens which is a natural consequence of the less dense mortar.

The influence of LSA-depot on the density and porosity was investigated with a hope that the SSA would make the mortar more compact as it could affect both the strength of the mortar (as described by the *Ryschkewitch Formula*) and the chloride penetration resistance. Although the density of the sample with LSA-depot was marginally larger than the reference sample, a variation of 10 kg/m^3 is not enough to conclude that the replacement of cement by SSA has an influence on the density and porosity of the mortar.

Similar experiments have been conducted with a sampling of LSA-new from 11.03.2013 by [Andreasen & Jørgensen, 2014] and the results hereof can be seen in Appendix C.4. Besides the reference sample and a 10% cement replacement sample the density and porosity have also been determined for a 5% sand replacement sample with LSA-new. Figure 21 shows the comparison of the porosities of mortar samples with respectively LSA-depot and LSA-new. A similar figure for the comparison of densities can be seen in Appendix C.4.



Figure 21: Comparison of the porosities of mortar samples with LSA-new and LSA-depot. Mean values with the standard deviations are shown

It is observed that there is quite a big difference between the porosities for the samples with LSA-depot and the samples with LSA-new with the former having approximately twice the porosity as the latter. But more importantly it is noted that the variation among the three samples with LSA-new is almost non-existent. This substantiates the conclusion made for LSA-depot, that the results gained from the density and porosity experiments can not be used to deduce that the addition of SSA has an effect on the density of the mortar.

Altogether it can be concluded that no influence on the density and porosity from the addition of SSA to mortar samples was measurable with the used test methods. The replacement of

either the cement or sand with SSA can however still have an effect on the porosity of the mortar since the test method used for these porosity determinations does not express anything about the pore size distribution in the mortar. The addition of SSA to the mortar could possibly change distribution of the pore sizes so that the pores are generally smaller which would increase the chloride penetration resistance of the mortar. However neither a confirmation nor a disconfirmation of that hypothesis was reached with the used test method.

4.2.3 Chloride Content in Mortar

The chloride content for mortar with 10% cement replaced with LSA-depot was measured to 0.16% which is only a little more than the result for the reference sample at 0.15%. The results can be found in Appendix B.3. Both results are beneath the limits stated by DS 2426:2011 that were shown in section 2.4.2 except for concrete with prestressed reinforcement in extra aggressive environment. With regard to chloride content the mortar with LSA-depot can therefore be used for almost all purposes.

It is noted that a little difference in the chloride content between the sample with LSA-depot and the reference sample was detected. The variation might be caused by the chloride content in LSA-depot but since it is quite insignificant no further investigations are made on the difference in chloride content of cement and LSA-depot.

4.2.4 Leaching

The leaching of both pure LSA-depot and mortar with and without LSA-depot was tested and the results hereof can be seen in Appendix B.3. Since there are no restrictions on the leaching of SSA it seems relevant to focus on the elements that were used for categorisation of LSA-depot in section 4.1.6. The results for these elements are shown in table 21. Additionally the leaching of phosphorus is shown since an overload of phosphorus led into the environment can result in eutrophication of rivers and lakes [USGS, 2013]. Eutrophication is a an increase of the mineral and organic nutrient that results in a reduction of the dissolved oxygen in the water body. Oxygen is essential for a lot of organisms including fish.

Element		Pure LSA-depot	5% sand replacement mortar	10% cement replacement mortar	Reference mortar
Phosphorus	[mg/kg]	0.99 ± 0.21	0.92 ± 0.28	0.72 ± 0.25	0.65 ± 0.21
Chromium	[mg/kg]	1.53 ± 0.05	0.07 ± 0.00	0.09 ± 0.00	0.09 ± 0.01
Copper	[mg/kg]	0.06 ± 0.02	0.02 ± 0.00	0.02 ± 0.00	0.02 ± 0.00
Lead	[mg/kg]	0.06 ± 0.03	0.05 ± 0.01	0.04 ± 0.01	0.03 ± 0.03
Magnesium	[mg/kg]	0.01 ± 0.00	0.01 ± 0.00	0.01 ± 0.01	0.00 ± 0.00
Nickel	[mg/kg]	0.00 ± 0.01	0.02 ± 0.01	0.00 ± 0.01	0.01 ± 0.01
Zinc	[mg/kg]	0.01 ± 0.01	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00
Arsen	[mg/kg]	0.00 ± 0.00	0.00 ± 0.00	0.01 ± 0.03	0.00 ± 0.00
Cadmium	[mg/kg]	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00

Table 21: Results from leaching experiment with both pure LSA-depot, mortar with LSA-depot and reference mortar

It is quickly observed that neither pure LSA-depot nor mortar with LSA-depot leaches critical amounts of any element and for most elements nothing is leached at all.

When comparing the leaching from the pure LSA-depot with the content of the same elements in table 15 in section 4.1.6 it is noted that the leaching is basically negligible. The element that is leached the most is chromium but at 1.53 mg per kg SSA it is still next to nothing. It is however noted that chromium(VI) is very movable at high pH-values but based on the available results it is not possible to determine which kind of chromium the SSA contains.

The leaching from the mortar specimens containing LSA-depot is compared with the reference mortar and it is seen that when taking the standard deviations into consideration the SSA does not have an influence on the leaching at all.

The amount of leached phosphorus is also completely insignificant.

4.3 Chloride Diffusion

The diffusion of chloride ions into three different mortar samples was tested after 7, 14 and 21 weeks in 3% salt water. The diffusion profiles after seven and 21 weeks are shown in figure 22 and 23 respectively. The profile for the 14 weeks test along with all the data from the tests can be found in Appendix B.4.



Figure 22: Comparison of the chloride diffusion after seven weeks in salt water

Figure 22 shows that there is no difference in the diffusion of chloride ions into mortar with and without LSA-depot. In the first two centimeters it is noted that the 10% cement replacement sample is not quite on top of the other two graphs but this is easily explained in the test method where only a small part of each slice is actually used for the chloride determination. After seven weeks the chloride ions have penetrated the outer two centimeters of the mortar and the graph is quite steep.



Figure 23: Comparison of the chloride diffusion after 21 weeks in salt water

After 21 weeks the graph looks a bit more flat compared to the seven weeks figure. This is due to the fact that the increase in the chloride content in the outermost centimeter relatively speaking is not as big as the increase further into the specimen. The chloride has penetrated approximately three and a half centimeters after 21 weeks. Compared to the results after seven and 14 weeks which were respectively two and three centimeters of penetration the rate of diffusion is found to be non-linear. This makes a lot of sense since the outermost centimeters keep absorbing some of the chloride ions that are on their way further in to the mortar. It could be interesting to conduct similar experiments over a longer period to determine if the diffusion keeps being slowed even more down.

Since all three test periods provide the same result with regard to the identical chloride diffusion for mortar with and without LSA-depot, it is safe to conclude that there is no proof that the addition of LSA-depot to mortar will enhance the risk of reinforcement corrosion due to diffusion of chloride ions.



Figure 24: Comparison of the test periods for chloride diffusion into 10% cement replacement samples placed in salt water

Figure 24 compares the diffusion profiles for the different test periods for the 10% cement replacement sample. Similar graphs are made for the reference and the 5% sand replacement samples and these can be seen in Appendix B.4.

It is noted how identical the chloride content is from four centimeters and inwards for the three test periods which underlines that the chloride ions have not penetrated so far in to the mortar.

The chloride diffusion into mortar with 10% LSA-depot as cement replacement is compared to similar tests conducted with LSA-new [Andreasen & Jørgensen, 2014] and Avedøre SSA [Kurt, 2014]. In order to verify the comparison the results for the appertaining reference samples are first compared which can be seen in figure 25. It is noted that there is difference in the curing time for Avedøre SSA (32 days) and the two LSA-ashes (both 78 days). There is also a variation in the test period for the chloride diffusion test but the three periods that were most alike have been chosen for comparison. The data from [Andreasen & Jørgensen, 2014] and [Kurt, 2014] is shown in Appendices C.4 and C.5.



Figure 25: Comparison of reference samples from this report, [Andreasen & Jørgensen, 2014] and [Kurt, 2014]. The curing time was 32 days for [Kurt, 2014] and 78 days for the other two

The diffusion profiles for the three reference samples are close to identical only with small variations being caused by differences in the size of the mortar slices. It is therefore considered relevant to compare the chloride diffusion in mortar with 10% cement replacement which can be seen in figure 26.



Figure 26: Comparison of the 10% cement replacement samples with LSA-depot, LSA-new [Andreasen & Jørgensen, 2014] and Avedøre SSA [Kurt, 2014]. The curing time was 32 days for Avedøre SSA and 78 days for the two LSA-ashes

When comparing the chloride diffusion for samples with the different SSAs it is seen that there is almost no variation. Avedøre SSA deviates a bit but this is due to the more rough division of the sample with larger slices. The chloride content in the specimen with LSA-new is a little higher compared to LSA-depot from three centimeters and inwards, this is however estimated

to be caused by uncertainty of measurements. Furthermore since the tests have been conducted separately there might be variations in the degree of pulverisation of the mortar.

Based on the results that have been covered in this section it is concluded that neither of the SSAs enhances the risk of reinforcement corrosion caused by chloride diffusion.

4.4 Chloride Binding

Since previous research [Thomas et al., 2012 and Ipavec et al., 2013] has shown that alumina has a positive effect on chloride binding, the amount of aluminium determined by ICP analysis is converted into aluminium oxide. Aluminium oxide can be measured by XRF analysis but research by [Ottosen et al., 2013b] has given a conversion method that has previously proven to be very consistent and it is therefore used. Both the content of aluminium and aluminium oxide in LSA-depot can be seen in Appendix B.1.

The content of Al_2O_5 in LSA-depot is found to be 2.52%. Compared to the supplementary cementitious materials investigated by [Thomas et al., 2012] it falls in between silica fume ($< 0.5\% \ Al_2O_5$) and ordinary portland cement ($\approx 4\% \ Al_2O_5$) but far below metakaolin (45% Al_2O_5) and fly ash (25% Al_2O_5) which proved to enhance the chloride binding in hardened cement. This indicates that LSA-depot should not be very good at chloride binding and mortar with LSA-depot should be expected to be less capable of binding chloride than reference mortar.

However also the amount of C-S-H has an influence on the chlorinde binding and there might be other factors with influence that have yet to be discovered which justify the extensive research of chloride binding into LSA-depot and mortar with LSA-depot that is being discussed in the following sections.

Screening Tests

The results of the two screening tests can be seen in figure 27. The solutions with 20% NaCl for unwashed LSA-depot and 10% NaCl for washed LSA-depot did not give any reliable results as the remaining chloride content was too large to be measured properly. These results are therefore not included in the figure. In Appendix B.5 the exact numbers can be found.



Figure 27: Results from the screening tests of chloride binding to LSA-depot expressed as mg Cl^- per g LSA-depot. A negative value indicates that the SSA overall released chloride instead of absorbing it

Figure 27 shows that both washed and unwashed LSA-depot can bind chloride. There is a similarity in the two graphs but they are slightly displaced with the washed LSA-depot already showing binding ability at a NaCl concentration of 1% whereas the unwashed LSA-depot needs a 10% NaCl concentration to start binding the chloride ions. The displacement of the graphs derives from the fact that the washed LSA-depot only releases a little bit of chloride since most of the chloride ions have been washed out prior to the experiment. The unwashed LSA-depot on the other hand releases a lot of chloride which can be seen in the result for the 0% NaCl dissolution.

Since the washed LSA-depot proves to bind chloride ions even at very low concentrations of the NaCl solution further experiments it is decided to limit further experiments to a maximum NaCl concentration of 5%. This also reduces the risk of inaccurate chloride measurements caused by too large amounts of chloride ions in the dissolutions.

4.4.1 Chloride Binding into LSA-depot

The results for the chloride binding to the pure LSA-depot are first discussed separately for the unwashed and washed SSA respectively and then compared later on. All the results can be seen in Appendix B.5.

Overall several chloride measurements diverged from what could be expected based on the other results in that group of samples (e.g. the result for 3% NaCl concentration for unwashed LSA-depot after 16 weeks as seen in figure 28). Such deviations could have possibly been minimised by conducting all tests with triple determination. Due to limited time it was however chosen to prioritise the amount of different investigations over the triple determination to provide the best foundation for further experiments.

The test method itself was tested concurrently with the second screening test by making a sample with 3% salt water and no LSA-depot. The chloride content deviated 0.2 g/L (as seen in Appendix B.5 from what it was supposed to be which is considered to be an acceptable deviation and a good verification of the test method.



Tests with Unwashed LSA-depot



Figure 28 illustrates the variations between the four different test periods for unwashed LSAdepot. It is noted how similar the three shortest periods are whereas the development of the chloride binding in relation to the NaCl concentration after 16 weeks deviates a lot. It takes approximately a 2.5% NaCl concentration of the dissolution for the SSA to start binding the chloride instead of releasing it. The 16 weeks sample takes a very unexpected path as the increased NaCl concentration causes the SSA to release more chloride with an exception from 3 to 5%. This variation is however expected to be due to an inaccuracy in the measurement.



Figure 29: Chloride binding as a function of the test period

The release of chloride ions after 16 weeks is even more evident in figure 29 where the chloride

binding is shown as a function of the test period.

When the four NaCl concentrations are compared like that it becomes quite clear exactly how consistent the chloride release is from the 0% sample at all test periods. The amount of released chloride ions is constant around 1.8 mg/g which corresponds extremely well with the measured chloride content of LSA-depot as seen previously in table 17.

Both the 1, 3 and 5% samples release more chloride ions after 16 weeks than what the SSA was actually measured to contain which is unexpected. The fact that all three samples release more chloride than they were supposed to contain indicates that it is not caused by inaccurate measurements. This however leads to a suspicion that the ion chromatography used for determination of the chloride content of LSA-depot is not precise as there must be a reserve of chloride ions in the SSA that is released by the presence of NaCl over time.

Another thesis is that a small part of the 16 weeks sample solutions has evaporated and thereby increased the NaCl concentration of the remaining part of the solution. During the experiment it was however not noted that the 16 week samples should have been more empty than the others and this thesis is therefore not considered reliable.

Tests with Washed LSA-depot



Figure 30: Chloride binding as a function of the NaCl concentration in the solution that LSA-depot was tested in

The development of the chloride binding as a function of the NaCl concentration of the sample solution is shown in figure 30. It is observed that the 6 weeks sample differs from the other three in both direction and values. The remaining three samples all seem more alike in their development but it is again noted how the binding ability is decreased over time ending with chloride ions being released from the SSA after 16 weeks.



Figure 31: Chloride binding as a function of the test period

Figure 31 is included to illustrate especially how consistent the results for the 0% sample are. There is no result after 16 weeks as the sample had dried out but based on the results for the unwashed LSA-depot it is definitely expected that the completely horisontal line would have been continued.

Most of the chloride ions have been washed out of the washed LSA-depot which is confirmed by the 0% samples and it is therefore a little unexpected that chloride ions are released from both the 1 and 5% samples.

Comparison of Unwashed and Washed LSA-depot

The results for the unwashed and washed LSA-depot are compared in figure 32 and 33. It is split into two figures to allow for a better overview as the graphs would be very close and some of them even on top of each other if all the results were shown together in one figure. It is important to notice that the scales on the two y-axes are different. Both figures show the chloride binding as a function of the NaCl concentration of the sample solution.



Figure 32: Comparison of the chloride binding to unwashed and washed LSA-depot after test periods of 2 and 4 weeks



Figure 33: Comparison of the chloride binding to unwashed and washed LSA-depot after test periods of 6 and 16 weeks

With minor exceptions the two graphs for each test period follow approximately the same path but they are displaced parallelly. The displacement is equal to the amount of chloride ions that have been washed out of the SSA. There are no indications towards that the washed LSA-depot is better at binding the chloride than the unwashed, it only releases less chloride on its own as most of it has been removed during the washing.

The SSA generally seem to be able to bind chloride during the first four to six weeks but as discussed previously when looking at the results for the 16 weeks tests LSA-depot actually releases more chloride ions than it was originally measured to contain. The reason for this is unknown but it is not uncommon for chemical reactions to be slow.

The results are far from being as good as hoped but they definitely provide valuable knowledge

on LSA-depot. It leads to two different relevant future investigations as similar tests should be conducted with longer test periods to see if LSA-depot will release even more chloride and other SSAs should be tested as well to determine if they also release chloride.

4.4.2 Chloride Binding into Mortar Samples with LSA-depot

All the results for chloride binding to mortar can be seen in Appendix B.5. Figure 34 provides a full overview by comparing both mortar with unwashed and washed LSA-depot and reference mortar. It can seem a bit confusing with all the information collected in one figure though and the results for unwashed and washed SSA are therefore discussed separately in the following sections.



Figure 34: Comparison of mortar with unwashed and washed LSA-depot and reference mortar. Black = Unwashed LSA-depot. Red = Washed LSA-depot. Blue = Reference.

When observing figure 34 it is first of all noticed that neither of the mortar samples overall release chloride ions. Another remarkable observation is how close the graphs are from 0 to 1% NaCl concentration, from 1% and onwards they are more spread out with the reference results approximately placed in the middle.

Due to large differences in the test methods for these chloride binding tests and the experiments conducted by [Thomas et al., 2012] and [Ipavec et al., 2013] it is not considered relevant to compare the results hereof.

Tests with Unwashed LSA-depot

The chloride binding as a function of the NaCl concentration of the sample solution is shown for mortar with unwashed LSA-depot in figure 35. The results are very similar for sample solutions with 0 and 1% NaCl but when comparing the higher NaCl concentrations it is noted that the reference samples are still reasonably close regardless the test period whereas the samples with LSA-depot are spread out. The 2 weeks sample with LSA-depot have a much better chloride binding ability than the reference sample at both 3 and 5% NaCl. That is however turned completely upside down when observing the 16 weeks samples.



Figure 35: Comparison of chloride binding to mortar with and without LSA-depot as a function of the NaCl concentration in the test solution. Black = With LSA-depot. Red = Reference.

The tendency for the 16 weeks samples are very similar to that of the pure LSA-depot where it ended up releasing a lot of chloride ions despite great chloride binding ability for the 2 weeks test period. It seems like the same principle accounts when the SSA is part of the mortar which means that the hydration of unwashed LSA-depot does not make any changes to the chloride binding ability. The reason that chloride ions are not overall released from the mortar with unwashed LSA-depot must be caused by the hydrated cement that can bind some chloride.

Tests with Washed LSA-depot



Figure 36: Comparison of chloride binding to mortar with and without LSA-depot as a function of the NaCl concentration in the test solution. Black = With LSA-depot. Red = Reference.

Figure 36 and 37 show the chloride binding as a function of respectively the NaCl concentration of the sample solution and the test period.

In figure 36 it is first of all noticed how the samples with LSA-depot have bound more chloride than the corresponding reference sample except for the 4 weeks test period. This is a very positive development.

For 5% NaCl sample solution it is however noted that the results for both 6 and 16 weeks with LSA-depot fall below the 2 weeks sample which leads to concerns on how the result would look with even longer test periods.



Figure 37: Comparison of chloride binding to mortar with and without LSA-depot as a function of the test period. Black = With LSA-depot. Red = Reference.

Figure 37 makes it even more clear how the samples with LSA-depot have a better ability to bind chloride than the corresponding reference sample. Despite the small concern regarding what longer test periods would result in it is a very positive development that mortar with washed LSA-depot does not seem to release chloride ions as it happened with the pure SSA.

4.5 Rate of Corrosion

During the wet/dry cycles of the experiment it was observed how NaCl was transported from the salt water to the top of the mortar samples that were held out of the water. The area on the top around the reinforcement bar also came to look very bad quite fast with big patches of red and black rust.

Figure 38 and 39 show the progress during the test periods for respectively the specimens cast with distilled water and the ones cast with 3% salt water.





(d) After 7 weeks



(e) After 9 weeks



(c) After 5 weeks



(f) After 16 weeks

Figure 38: Observations during the test period for specimens cast with distilled water



Figure 39: Observations during the test period for specimens cast with 3% salt water

It was found to be a bit surprising that the NaCl could even penetrate through the epoxy paint

on top of the visual test specimens.

It is assumed that the salt deposits are a result of capillary forces that have pulled the salt water to top of the specimens where the water continuously evaporated leaving the salt behind in big patches.

The amount of salt that had been transported to the top did not vary much between the specimens cast with and without NaCl which suggests that the salt from the casting was not moved.

4.5.1 Electrical Potential Test

The electrical potential tests have been made with a SCE electrode (saturated calomel electrode) and the measurements are converted to SHE values (standard hydrogen electrode) by adding 242mV to the numeric measurement.

The limit stated by [Klinghoffer, 1993] at $-350mV_{CSE}$ for active corrosion is converted to SHE by adding 300mV to the numeric limit.

The standard potentials for reference electrodes used for conversion can be seen in table 22 [Kurt, 2014].

Electrode	Electrolyte	SHE potential
[-]	[—]	$[mV_{SHE}]$
Standard hydrogen electrode (SHE)	$H_2 (1 \text{ atm})$	0
Standard hydrogen electrode (SHE)	$H^+~(\mathrm{a}=1)$	0
Saturated calomel electrode (SCE)	Saturated KCl	+ 242
1 N calomel electrode (NCE)	1.0 M KCl	+ 281
Calomel	$0.1 \mathrm{M} \mathrm{KCl}$	+ 333.5
Silver/silver chloride electrode (SSC)	1.0 M KCl	+ 222.4
${\bf Copper/copper \ sulphate \ electrode \ (CSE)}$	Salt water	ca. + 300
Zinc	Salt water	- 790

Table 22: Standard potentials for reference electrodes used for conversion to SHE values by adding to numeric measurements [Kurt, 2014]

Before discussing the actual results from the tests a validation of the results is attempted by comparing the electrical potential measurements for the reference specimens with results from similar experiments conducted by [Andreasen & Jørgensen, 2014] and [Kurt, 2014]. The general procedure for all three experiments was the same with only minor variations. The comparison of the reference specimens should therefore serve as an extended multiple determination and if the results are consistent it provides a verification of the obtained results.

Figure 40 shows the comparison of the reference specimens. Due to several variations within each experiment it is chosen to show the results for all nine specimens instead of making mean values that would be highly influenced by e.g. the drops in electrical potential that two specimens experienced after 35 days.



Figure 40: Comparison of the results for reference specimens obtained in this project, by [Andreasen & Jørgensen, 2014] and by [Kurt, 2014]

It is seen that overall the potential measurements for the reference samples follow each other really well. The most noticable difference is at the very beginning of the test period where the potential for the three specimens from [Andreasen & Jørgensen, 2014] is a lot lower than for the other six specimens. Due to a similar development in the growth of the potential for all of the specimens the variation in the potential at the start influences the amount of time it takes for the specimens to cross the critical line (the potential that states a 90% risk for initiation of corrosion as described previously).

Generally it is considered that the comparison of the reference results serve as a good indication that the experiments conducted in this project provided reliable and stable results.

4.5.2 Specimens Cast with Distilled Water



Figure 41: Comparison of reference and 10% cement replacement samples

Figure 41 shows the results of the electrical potential measurements on the specimens cast with distilled water. The data from the results can also be found in Appendix B.6.

When observing the graph one thing in particular leaps out and that is the 10% cement replacement specimen that is far below the other specimens. Nothing seemed different about that specimen during the test besides the odd measurements. During casting it was one of the outer specimens in the mould which means that insufficient vibration should not be the reason. The middle specimen in the moulds often seemed to be vibrated a little less than the outer specimens but this does not seem to be the explanation here. Given that the specimen does catch up with the others after 49 days it is more plausible that the reason for the low measurements is to be found in the amount of rust on the reinforcement bar where the alligator clip was attached. Despite serious effort to remove the rust before measuring the potential it was not possible to remove it completely and it might very well have made a difference.

The rest of the measurements are very much alike and if a difference should be found then it would be that the reference sample generally gave a little higher potentials than the sample with LSA-depot. Both the specimens with and without LSA-depot did however cross the critical line stated by [Klinghoffer, 1993] very fast. This should indicate that the corrosion was initiated within the first seven days. The visual tests did however not confirm that thesis. Figure 42 shows the visual test specimens that have been cut across with the upper row being the reference sample and the bottom row showing the 10% cement replacement sample.



(a) Dry reference specimen after six weeks



(d) Dry 10% cement specimen after six weeks



(b) Wet reference specimen after 17 weeks



(e) Wet 10% cement specimen after 17 weeks



(c) Dry reference specimen after 17 weeks



(f) Dry 10% cement specimen after $17~{\rm weeks}$

Figure 42: Corrosion investigation of specimens after six and 17 weeks. Photos of dry specimens were taken two days after the cutting

The specimens had been left to dry out for a week before being cut but the wet saw obviously

soaked them during the cutting process which made it hard to detect any corrosion. With the first specimens that were cut across after six weeks they were therefore left to dry for two days before being investigated and photographed. During the two days the specimens were held in a tight zip bag that limited the amount of oxygen available for corrosion. Based on the amount of rust that developed during those two days it was chosen for the specimens cut across after 17 weeks to do both an inspection right after the cutting and again after two days.

As the photos in figure 42 show there was a difference in the amount of rust developed on the reference and the 10% cement replacement specimens when looking at them after two days. It is however hard to draw any conclusion on that as long as the photos taken right after the cutting show no corrosion on either of the specimens.

In another attempt to detect any differences between the samples with and without LSA-depot the reinforcement bars in the last specimen from each sample were exposed by removal of the mortar which can be seen in figure 43. The figure also shows a reinforcement bar before it was embedded in the mortar.



(a) Reference specimen after 17 weeks

(b) 10% cement specimen after 17 weeks

(c) Reinforcement bar before embednment in mortar

Figure 43: Corrosion investigation of specimens after 17 weeks compared to a reinforcement bar before embedment in mortar

As figure 43 shows rust was actually detected on the reference specimen whereas no corrosion was spotted on the 10% cement replacement specimen. However when taking into account how the reinforcement bar looked before the embedment the rust that is seen on the reference sample might very likely stem from before the embedment and not be a result of the corrosion test.

The lack of corrosion despite the potential measurements that indicated an initiation of corrosion very strongly, is assumed to be caused by deficiency of oxygen close to the reinforcement. It is believed that either the length of the drying periods in the test cycles might have been too short or the mortar was too dense for oxygen to penetrate properly. If the test cycles were too short so that only the outermost centimeter of the specimen dried it will have affected the corrosion process. This thesis is illustrated in figure 44.



Figure 44: It might only be the outermost centimeter that has dried during the test cycles which will have resulted in a lack of oxygen that is necessary for corrosion to happen

The experiments could have been made with a less dense mortar which would have speeded up the corrosion but it was more relevant to conduct the tests with the same mortar as investigated during the rest of the project.

A comparison of the obtained results with [Prieto et al., 2013] emphasises that there is no real correlation between the electrical potential and the corrosion of the reinforcement. [Prieto et al., 2013] suggests that the potential is associated more with the humidity of the mortar which seems reliable based on the present experiments.

Their potential measurements also rose above the 90% limit within the first few weeks and were then quite stable for the next 500 days as seen in figure 45. The development of their results was part of the reason why the test period for the present experiments was not extended further since no surprises in the form of deviations in the graphs were expected.



Figure 45: Result from [Prieto et al., 2013] for specimens with different percentages of chloride ions
4.5.3 Specimens Cast with 3% Salt Water

The corrosion tests were also conducted on samples that were cast with 3% salt water instead of distilled water. Figure 46 shows the comparison of the electrical potential for the reference specimens and the 10% cement replacement specimens. The data from the experiment can be seen in Appendix B.6.



Figure 46: Comparison of reference and 10% cement replacement samples cast with 3% salt water

When looking at figure 46 it is noted how all the potential measurements are very stable in between 750mV and 850mV. There is a great variation between the potential development for these specimens cast with salt water and the ones cast with distilled water (shown previously in figure 41) with the most remarkable difference being the first 14 days of the experiments.

According to the potential measurements the corrosion of these specimens cast with salt water should have been initiated from the very beginning of the test. However when performing a corrosion inspection by removal of the mortar around the reinforcement bar after 15 weeks no corrosion was spotted as seen in figure 47.

The lack of visible corrosion leads to the same conclusion as for the specimens cast with distilled water: the electrical potential is associated more with the humidity of the mortar than with the initiation of corrosion.



Figure 47: Corrosion investigation of specimens after 15 weeks

Despite the variations in the electrical potential development for specimens cast with distilled water and salt water no difference was seen in the corrosion investigation after 15 weeks.

The salt water samples were however also investigated for corrosion by being cut across just like the samples cast with distilled water. This was done after six and 15 weeks and the results can be seen in figure 48. The same circumstances with the documentation of wet and dry conditions as described for the specimens with distilled water apply for this test.



(a) Dry reference specimen after six weeks



(d) Dry 10% cement specimen after six weeks



(b) Wet reference specimen after 15 weeks



(e) Wet 10% cement specimen after 15 weeks



(c) Dry reference specimen after 15 weeks



(f) Dry 10% cement specimen after 15 weeks

Figure 48: Corrosion investigation of specimens after six and 15 weeks. Photos of dry specimens were taken two days after the cutting

Similarly to the samples cast with distilled water figure 48 shows how no corrosion can be spotted on the samples cast with salt water right after they have been cut across. But when looking at the specimens that have dried out after being cut across quite a lot of rust is noticed. The amount of rust far exceeds what was seen on the specimens cast with distilled water. This could potentially be caused by small differences in the storage of the specimens between the cutting and the corrosion investigation but it also leads to a possible hypothesis on the influence from the NaCl in the casting water. The elevated content of chloride ions in the specimens might have degraded the passivating layer on the reinforcement bars worse than for the specimens cast with distilled water and when oxygen became available after the cutting then the salt water specimens were more vulnerable and therefore corroded more.

4.6 Overall Discussion

The assessment of LSA-depot for use in concrete based on the characterisation of the ash brought two main things in focus. A relatively high content of heavy metals and other elements that are harmful to the environment restricts the possibilities for the use of concrete with LSA-depot. The declaration from Danish Environmental Protection Agency [Danish EPA, 2013] places the SSA in category 2 which limits the usage considerably. Research on how to remove heavy metals from SSA by electrodialytic extraction has been and is still being conducted at DTU [Kirkelund et al., 2013]. It would therefore be very advisable to follow the results of the research closely and implement it as part of the preparation of the SSA on par with the milling as soon as possible.

The other thing that was brought into focus by the characterisation is the necessity of a procedure for homogenisation of SSAs. Large variations in different samplings of the same SSA have been observed both in this report and by [Andreasen & Jørgensen, 2014] and more consistency is needed before the SSAs can be used in concrete.

LSA-depot has been compared with the eurocode for fly ash and a Danish declaration on waste products used for construction work, but in order to ease the implementation of usage of SSA in concrete it is considered relevant and important to compile the requirements in a Danish or European standard.

During the characterisation of LSA-depot a relatively high content of chloride ions in the SSA was determined especially when compared with LSA-new. This did however not affect mortar with a 10% cement replacement of LSA-depot where the same chloride content was measured for the reference sample and the sample with LSA-depot. Neither did it seem to have any influence on the initiation of corrosion during the experiments with electrical potential measurement and visual inspection of reinforced mortar specimens.

The high chloride content led to problems when investigating the chloride binding ability of LSA-depot. Both washed and unwashed pure LSA-depot and mortar with unwashed LSA-depot released chloride ions when tested for longer periods of time but mortar with 10% cement replaced with washed LSA-depot showed better chloride binding capability than the reference mortar. The amount of bound chloride did however decrease with the length of the test period so more experiments are needed in order to determine if LSA-depot can actually reduce the risk of corrosion by binding the chloride ions that diffuse into concrete constructions. Based on the obtained results so far it is recommended that from now on LSA-depot is washed before being used in mortar as an overall release of chloride ions in mortar is very unfavourably.

A profound beneficial environment with regard to minimising the risk of corrosion was not found in mortar with LSA-depot as hoped. But neither was the opposite stated which must be regarded as a success. The obtained results indicate that LSA-depot can be used in concrete without increasing the risk of corrosion but the addition of the SSA does not entail that a less dense concrete with LSA-depot can provide the same protection of the reinforcement as a standard concrete without LSA-depot.

Several of the test methods used in this project were invented as part of the project, and are therefore to a great extent a compromise between what would provide noticeable results within the available time frame and what would be the most relevant and interesting investigations with LSA-depot.

For the experiments with reinforced mortar specimens more results could have possibly been obtained if a less dense mortar had been used allowing for both chloride ions and air to penetrate faster. This was however not chosen in order to maintain the common thread throughout the project by conducting tests with the same mortar recipes and thereby allowing comparison across all the investigations where relevant.

4.7 Suggestions for Future Research

The investigations performed in this project has provided a lot of interesting results that has led to important conclusions. However it has also triggered a number of new research fields that future experiments could seek to explore further.

The assessment of LSA-depot combined with the comparison with previous research established that there is a great difference both between the different SSAs and even more important between the different samplings of each SSA. This means that a characterisation of a SSA is crucial before doing further experiments with it. It also leads to a request for an establishment of a homogenisation process that can be used for all SSAs.

Based on the obtained results with chloride binding to both pure LSA-depot and mortar with LSA-depot it is considered very interesting to conduct prolonged versions of similar experiments. This should be done to see how the chloride ions will react with longer time for reaction. Also all SSAs should from now on be tested for chloride binding capability to establish if they actually release chloride ions which will make them unsuitable for use in reinforced concrete.

[Kurt, 2014] has conducted experiments on current induced chloride diffusion and similar experiments could be interesting to perform with LSA-depot. It can speed up the diffusion of chloride ions in to the mortar making shorter test periods possible.

The tests on the rate of corrosion did not provide any clear results on the initiation of corrosion which is probably partly due to the length of the test cycles that did not allow enough time for the mortar to dry out completely. Similar experiments should therefore be carried out with longer test cycles. Another idea for optimisation is to use a less dense mortar which will make it easier for oxygen to reach the reinforcement bar.

5 Conclusion

A series of laboratory experiments has been carried out in order to assess the eligibility of LSAdepot for use in reinforced concrete. LSA-depot is sewage sludge ash (SSA) from *Renseanlæg Lynetten* that has been stored for at least two years in an outdoor depot. The results were compared with standards where applicable and with results from similar tests for other SSAs.

Large variations were generally noted between the present and a previous sampling of LSAdepot when comparing pH, water content, conductivity and water solubility. This led to a demand for a homogenisation of the SSA before it can be used in concrete. A very high pH value was measured for the present sampling which will help creating an alkaline environment in concrete with LSA-depot.

Due to the content of various micro and macro elements in LSA-depot it falls into category 2 according to a declaration from the Danish Environmental Protection Agency. This restricts the options for usage of concrete with the SSA. Furthermore large differences were again observed between the two samplings. Despite the high content of several elements that are harmful to the environment neither were leached in critical amounts during tests with both pure LSA-depot and mortar containing the SSA.

The content of chloride ions in the pure LSA-depot exceeds the limit for fly ash but when looking at mortar with 10% cement replaced by LSA-depot, the chloride content was similar to a reference mortar with no SSA.

The replacement of neither cement nor sand with LSA-depot made a difference for the diffusion of chloride ions into mortar. Diffusion profiles for mortar with and without LSA-depot were identical after test periods of both 7, 14 and 21 weeks.

Both unwashed and washed pure LSA-depot released chloride ions instead of binding them when tested for 16 weeks. More promising results were obtained with mortar containing washed LSA-depot where the chloride binding ability proved to be better than for the reference mortar. However it was generally observed that the chemical reactions with releasing chloride ions were quite slow and mortar with washed SSA should therefore be tested for a longer period of time.

Electrical potential measurements of reinforced mortar specimens exposed to wet/dry cycles with salt water showed similar results for samples with and without LSA-depot. The measurements indicated with 90% certainty that corrosion had initiated within the first seven days of the experiment but this was not confirmed when the reinforcement bars were visually inspected after 6 and 17 weeks with no spotting of corrosion. The electrical potential is therefore considered to be more associated with the humidity of the mortar rather than the corrosion initiation.

Based on the conducted experiments it is established that LSA-depot does not increase the risk of corrosion when used in mortar as cement or sand replacement. Neither did any results indicate that the addition of LSA-depot is beneficial towards creating a favourable environment in concrete for reinforcement.

It is stressed that a method for homogenisation of LSA-depot should be developed but the overall assessment of the SSA for use in concrete is very positive. It seems possible to reduce the CO_2 emission in the future by replacing a part of the cement with LSA-depot in concrete for some purposes.

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A Test Methods

A.1 pH in 1M KCl Suspension

The pH value of the SSA is measured potentiomatrically in a 1:2.5 suspension of SSA and potassium chloride.

Apparatus and Materials

- $\bullet\,$ A pH meter with a combination electrode and an accuracy of 0.05 pH units
- 74.56 g potassium chloride dissolved into 1000.00 mL destilled water
- $\bullet\,$ Standard buffer solutions of pH 4.00 and 7.00
- Technical scale
- Finnpipette

Procedure

5.00 g dry SSA is poured into a 20mL plastic vial and 12.50 mL 1M KCl solution is added by use of a finnpipette. The suspencion is placed on an agitating table for one hour. Afterwards the sample is left for 5-10 minutes to settle.

Standardize the pH meter by means of the standard solutions.

When measuring the pH value the electrode is placed in the upper part of the solution. The result is read on the apparatus when the display shows "STAB". In between eash measurement the electrode is rinsed thoroughly with destilled water and the drop is carefully wiped off with a piece of paper.

Waste Management

Potassium chloride and the buffer solutions are poured into the sink.

The plastic vial with the suspension is collected in a container for soil waste.

A.2 Conductivity of SSA

Apparatus and Materials

- Electrical conductivity meter
- Volumetric pipette
- Agitating table
- Plastic bottle
- Technical scale

Procedure

10.00 g dry SSA is poured into a 50 mL plastic bottle. 25.00 mL destilled water is added by use of a volumetric pipette. The sample is left on an agitating table for 30 minutes followed by 20 minutes of settling or until a liquid fase has separated from the SSA.

The conductivity electrode is rinsed with destilled water, placed in the liquid and "SAMPLE" is pressed on the electrical conductivity meter. When the display shows "STAB" the result can be read.

Waste Management

The samples are poured into a container marked X 4.41 (heavy metals).

A.3 Water Content

Apparatus and Materials

- $\bullet\,$ Oven heated to $105^{\circ}\mathrm{C}$
- Beaker
- Technical scale

Procedure

The weight of a beaker is noted down. SSA is poured into the beaker and the combined weight is then noted down. The beaker is left overnight in an oven heated to 105°C. When the beaker is cooled the combined weight of beaker and SSA is then noted down.

Waste Management

The SSA is either kept for further tests or poured into a container for soil waste.

A.4 Loss on Ignition

Apparatus and Materials

- Muffle furnace
- Desiccator
- A crucible
- A pair of tongs
- Analytical balance

Procedure

The muffle furnace is heated to 550°C and the crucible is placed in there for 30 minutes. The crucible is from then on only moved by use of a pair of tongs to ensure that not even a tiny amount of grease is left on the crucible. It is left in a desiccator to cool down and then weighed on the analytical balance.

Approximately 2.5 g dry SSA is weighed on the analytical balance and poured into the crucible which is then placed in the heated muffle furnace for 1 hour. Afterwards the crucible is left to cool down in the desiccator and then weighed on the analytical balance.

Waste Management

The SSA is poured into a container for soil waste.

A.5 Water Solubility

Apparatus and Materials

- 1 L plastic bottle with lid
- Funnel
- Filter paper
- Measuring cylinder
- Technical scale
- Oven heated to 105°C

Procedure

 $100.00~{\rm g}$ SSA is weighed and poured into the plastic bottle. $500~{\rm mL}$ of destilled water is added, the bottle is shaken thoroughly and is then left to settle.

A filter paper is weighed and then placed in a funnel in a measuring cylinder.

The liquid from the bottle is poured into the filter paper. 500 mL of destilled water is added again, the bottle is shaken and then left to settle.

The liquid is again poured into the filter paper, 500 mL of destilled water is added, the bottle is shaken and then the entire content of the bottle is poured into the filter paper. The filter paper is left overnight to drain and then put into the oven for the night. The following day the combined weight of the ash and the filter paper is noted down.

Waste Management

The SSA is either saved for tests with washed ash or poured into a container for soil waste.

A.6 Preparation for ICP Analysis

Apparatus and Materials

- PYREX glass with screw top
- Volumetric pipette
- Autoclave
- Suction filtration apparatus with $0.45\nu{\rm m}$ filter
- Volumetric flask
- Plastic vial

Procedure

1.00 g dry SSA is weighed and poured into a PYREX glass. 20 mL 1:1 HNO_3 is added by use of a volumetric pipette. The PYREX glass is closed tightly ensuring the HNO_3 will not evaporate. The PYREX glass is placed in an autoclave at 200 kPa (120°C) for 30 minutes. The sample is filtered with suction through a 0.45ν m filter. The PYREX glass is rinsed with distilled water three times. The filter must dry out in between each rinse.

The filtrate is poured into a 100 mL volumetric flask and destilled water is added until 100 mL total is reached. The liquid is poured into a 20 mL plastic vial and placed in the fridge until ICP analysis.

Waste Management

The samples are poured into a container marked X 4.41 (heavy metals). The SSA and the filterpaper is poured into a container for soil waste. The nitric acid is poured into a container marked X 4.18.

A.7 Preparation for Ionchromatography

Apparatus and Materials

- Plastic bottle
- Plastic vial
- Syringe filter
- Agitating table
- Technical scale

Procedure

 $10.00~{\rm g}$ dry SSA is weighed and poured into a $100~{\rm mL}$ plastik bottle. $50.00~{\rm mL}$ of destilled water is added. The sample is placed on an agitating table over night.

The sample is filtered with a syringe filter into a plastic vial and placed in the fridge until ionchromatography.

Waste Management

The syringe filter is thrown in a container for soil waste.

A.8 Grain Size

Apparatus and Materials

- Mastersizer 2000 laser diffractor
- Plastic vials
- 0.005M sodium polyphosphate
- Pipettes

Procedure

Half a teaspoon of SSA is poured into a plastic vial. The vial is then filled almost completely with sodium polyphosphate and it is shaken. The apparatus is rinsed three times and the instructions on the screen is then followed. The sample solution is added by use of a pipette immediately after a thorough shake.

Waste Management

The plastic vials with the samples are thrown into a container for soil waste.

A.9 Titration with Silver Nitrate

Apparatus and Materials

- Titrator 716 DMS Titrino
- 5 mL burette unit
- $AgNO_3$ electrode (6.0430.100)
- 50 mL measuring cylinder
- Beaker 100-150 mL
- Magnetic stirring bar
- $0.1N AgNO_3$
- 1M HNO_3

Procedure

Turn both the titrator and the printer on. Place the electrode and the burette hose in the stand by the magnetic stirrer. Place a waste glass under the electrode and the burette hose. Press DOS to empty the burette and tap carefully on the hose while doing so to let out air bubbles and press FILL to fill the burette. Repeat two times.

Prepare three beakers for calibration each with 80 mL destilled water, 1 mL 0.1N NaCl and 1 mL 1M HNO_3 . Put the magnetic stirring bar in the first beaker and place it on the magnetic stirrer. Adjust the stand with the electrode and the burette. Press START. The result is printed automatically when the titration is completed. Repeat with the other two beakers.

To measure a dissolved sample: Prepare a beaker with 80 mL destilled water, 1 mL 1M HNO_3 and 1-5 mL sample. Follow the same procedure as for the calibration.

To measure a sample of extracted chloride from hardened mortar: The beaker with 150 mL sample is used without any further preparation. The same procedure is followed as for the calibration.

Waste Management

All liquid containing silver nitrate is deposited in a container for silver nitrate. All materials that have been in contact with the silver nitrate is rinsed thoroughly.

A.10 Leaching

Apparatus and Materials

- Plastic bottle
- Plastic vial
- pH meter
- Agitating table
- Technical scale
- Suction filtration apparatus with $0.45\nu\mathrm{m}$ filter
- Volumetric pipette

Procedure

10.00 g SSA or finely crushed dry mortar is weighed and poured into a 50 mL plastic bottle. 20 mL of destilled water is added by use of a volumetric pipette. The sample is placed on an agitating table for exactly 23 hours and then left to settle for 15 minutes. The pH value is measured.

The sample is filtered with suction through a $0.45\nu{\rm m}$ filter into a 20 mL plastic vial and then analysed with ICP.

Waste Management

The filter with SSA is poured into a container for soil waste.

A.11 Extraction of Chloride from Hardened Mortar

Apparatus and Materials

- Conical flask
- Filter
- Beaker
- Technical scale

Procedure

5.00 g of SSA is weighed and poured into a conical flask. Approximately 50 mL of hot destilled water is added and mixed with the SSA. 10 mL of concentrated HNO_3 is slowly added, it is mixed well and then left to cool off.

1 mL of HNO_3 is added to check if all the SSA is dissolved which is identified by no air bubbles forming. If there are still air bubbles HNO_3 is added until the SSA is completely dissolved. The dissolution is filtered into a beaker and the filter is rinsed with 1% HNO_3 . Distilled water is added until a total volume of approximately 100 mL is reached.

Waste Management

The filter with the SSA is poured into a container for soil waste.

A.12 Density and Porosity

Apparatus and Materials

- Oven heated to 105°C
- Autoclave
- Technical scale
- Technical scale that can weigh the specimens whilst underwater
- Vacuum pump
- Cloths

Procedure

The samples are dried in the oven at 105° C until reaching weight consistency. The samples are afterwards cooled of in an autoclave at normal room temperature. When cooled off the samples are weighed (m_{105}).

The dried samples are evacuated continuously dry in an autoclave for at least three hours.

Distilled water with normal room temperature is lead into the autoclave. The samples must be covered completely with water and are left for one hour without pumping. Air is lead into the autoclave and the samples are left overnight.

The water saturated samples are weighed whilst in water (m_{uw}) . After wiping the surfaces of the samples with a moist cloth they are weighed normally (m_{ssd}) .

The samples are finally dried in the oven at 105° C until reaching weight consistency. The samples are once again weighed as a control of whether any leaching of the samples happened during the test.

B All Test Results

B.1 Characterisation of LSA-depot

$\mathbf{p}\mathbf{H}$

		А	В	С	Mean	Standard deviation
m _{LSA-depot}	[g]	5.00	5.00	5.00	-	-
Suspension	[-]	1M KCl	1M KCl	1M KCl	-	-
V _{suspension}	[mL]	12.50	12.50	12.50	-	-
рН	[-]	12.58	12.57	12.57	12.57	0.01

The test was made with unmilled LSA-depot.

The samples were placed on the agitating table for 1h10mins.

Conductivity

		D	E	F	Mean	Standard deviation
m _{LSA-depot}	[g]	5.00	5.00	5.00	-	-
m _{H2O}	[mL]	12.50	12.50	12.50	-	-
Conductivity	[mS/cm]	9.16	9.96	10.80	9.97	0.82

The test was made with unmilled LSA-depot.

The samples were placed on the agitating table for 1h10mins.

Water Content

The water content is calculated by use of:

$$\%$$
 water content = $\frac{m_{wet} - m_{dry}}{m_{wet}} \cdot 100$

		А	В	С	Mean		D	E	F	Mean	Standard deviation
m _{beaker}	[g]	108.74	108.45	98.59	-		108.21	105.11	108.46	-	-
m _{beaker + LSA-depot} before oven	[g]	219.21	211.49	204.67	-		187.46	173.54	181.80	-	-
m _{beaker + LSA-depot} after oven	[g]	214.97	207.32	200.53	-		184.33	170.85	178.94	-	-
m _{wet sample}	[g]	110.47	103.04	106.08	-		79.25	68.43	73.34	-	-
m _{dry sample}	[g]	106.23	98.87	101.94	-		76.12	65.74	70.48	-	-
Water content	[%]	3.84	4.05	3.90	3.93]	3.95	3.93	3.90	3.93	0.07

Sample A, B and C are made with unmilled LSA-depot. Sample D, E and F are made with milled LSA-depot.

Loss on Ignition

The loss on ignition is calculated by use of:

% loss on ignition = $\frac{m_{LSA-depot,before} - m_{LSA-depot,after}}{m_{LSA-depot,before}} \cdot 100$

		А	В	С	Mean	Standard deviation
m _{crucible} before oven	[g]	16.1084	15.5631	13.5575	-	-
m _{LSA-depot} before oven	[g]	2.5014	2.5006	2.5006	-	-
m _{LSA-depot + crucible} before oven	[g]	18.6098	18.0637	16.0581	-	-
m _{LSA-depot + crucible} after oven	[g]	18.5278	17.9815	15.9755	-	-
Loss on ignition	[%]	0.4406	0.4551	0.5144	0.4700	0.04

The tests were made with unmilled LSA-depot

		А	В	С	Mean	Standard deviation
m _{LSA-depot} , before filtration	[g]	100.00	100.00	100.00	-	-
Water content, LSA-depot	[%]	3.93	0.00	0.00	-	-
Water content, LSA-depot	[g]	3.93	0.00	0.00	-	-
$m_{LSA-depot}$, before filtration, adjusted	[g]	96.07	100.00	100.00	-	-
m _{filter}	[g]	10.55	10.68	10.06	-	-
m _{petridish}	[g]	96.57	102.50	108.82	-	-
mLSA-depot + filter + petridish, before oven	[g]	322.35	325.59	284.54	-	-
m _{LSA-depot + filter + petridish} , after oven	[g]	196.79	208.35	213.21	-	-
m _{LSA-depot} , after oven	[g]	89.67	95.17	94.33	-	-
Water solubility	[%]	6.40	4.83	5.67	5.6	0.79

The tests were made with unmilled LSA-depot.

Content of Micro and Macro Elements

		А	В	С	Mean	Standard deviation
Calcium (Ca)	[mg/kg]	247234.0	248626.0	239553.0	245137.7	4886.3
Phosphorus (P)	[mg/kg]	69214.0	69035.3	66611.2	68286.8	1453.9
lron (Fe)	[mg/kg]	37056.3	36103.1	34999.5	36053.0	1029.3
Magnesium (Mg)	[mg/kg]	14541.6	14326.7	13737.3	14201.9	416.4
Aluminium (Al)	[mg/kg]	13562.8	13591.0	12786.2	13313.3	456.7
Potassium (K)	[mg/kg]	9142.2	8892.6	9038.6	9024.5	125.4
Sodium (Na)	[mg/kg]	3895.5	3778.2	3782.4	3818.7	66.5
Zinc (Zn)	[mg/kg]	2308.1	2302.1	2187.8	2266.0	67.7
Copper (Cu)	[mg/kg]	515.52	500.70	505.37	507.20	7.58
Barium (Ba)	[mg/kg]	488.65	478.28	484.56	483.83	5.22
Manganese (Mn)	[mg/kg]	382.94	372.90	376.82	377.55	5.06
Lead (Pb)	[mg/kg]	104.95	94.69	96.68	98.77	5.44
Vanadium (V)	[mg/kg]	51.30	49.39	50.08	50.26	0.97
Chromium (Cr)	[mg/kg]	45.34	44.57	44.75	44.89	0.40
Nickel (Ni)	[mg/kg]	37.93	37.70	37.68	37.77	0.14
Arsen (As)	[mg/kg]	4.287	2.984	3.361	3.544	0.670
Cadmium (Cd)	[mg/kg]	2.077	2.323	2.205	2.202	0.123

Amount	Element	Conversion	Element	Amount
[%]	[-]	[-]	[-]	[%]
24.5	Calcium (Ca)	1.39919	Calcium oxide (CaO)	34.3
6.83	Phosphorus (P)	2.29137	Phosphorus pentoxide (P ₂ O ₅)	15.6
1.42	Magnesium (Mg)	1.65807	Magnesium oxide (MgO)	2.35
1.33	Aluminium (Al)	1.88946	Aluminium oxide (Al ₂ O ₃)	2.52
0.90	Potassium (K)	1.20459	Potassium oxide (K ₂ O)	1.09
0.38	Sodium (Na)	1.34798	Sodium oxide (Na ₂ O)	0.51
0.04	Manganese (Mn)	1.29122	Manganese oxide (MnO)	0.05

Conversion of Elements According to [Ottosen et al., 2013b]

Water Soluble Anions

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		А	В	С	Mean	Standard deviation
Chloride ions, Cl ⁻	[mg/kg]	1795.7840	1805.9496	1804.8092	1802.18	5.57
Nitrate ions, NO ₃	[mg/kg]	16.3696	16.8171	16.6213	16.60	0.22
Sulphate ions, SO ₄ ²⁻	[mg/kg]	8135.212	8264.2653	8342.7703	8247.42	104.80

Grain size

Grain size	Milled 1	Cumulated	Milled 2	Cumulated	Unmilled 1	Cumulated	Unmilled 2	Cumulated
[µm]	[%]	[%]	[%]	[%]	[%}	[%]	[%]	[%]
0.020000								
0.022440	0	0	0	0	0	0	0	0
0.025179	0	0	0	0	0	0	0	0
0.028251	0	0	0	0	0	0	0	0
0.031698	0	0	0	0	0	0	0	0
0.035566	0	0	0	0	0	0	0	0
0.039905	0	0	0	0	0	0	0	0
0.044774	0	0	0	0	0	0	0	0
0.050238	0	0	0	0	0	0	0	0
0.056368	0	0	0	0	0	0	0	0
0.063246	0	0	0	0	0	0	0	0
0.070963	0	0	0	0	0	0	0	0
0.079621	0	0	0	0	0	0	0	0
0.089337	0	0	0	0	0	0	0	0
0.100237	0	0	0	0	0	0	0	0
0.112468	0	0	0	0	0	0	0	0
0.126191	0	0	0	0	0	0	0	0
0.141589	0	0	0	0	0	0	0	0
0.158866	0	0	0	0	0	0	0	0
0.178250	0	0	0	0	0	0	0	0
0.200000	0	0	0	0	0	0	0	0
0.224404	0	0	0	0	0	0	0	0
0.251785	0	0	0	0	0	0	0	0
0.282508	0	0	0	0	0	0	0	0
0.316979	0	0	0	0	0	0	0	0
0.355656	0	0	0	0	0	0	0	0
0.399052	0	0	0	0	0	0	0	0
0.447744	0	0	0	0	0	0	0	0
0.502377	0	0	0	0	0	0	0	0
0.563677	0	0	0	0	0	0	0	0
0.632456	0	0	0	0	0	0	0	0
0.709627	0	0	0	0	0	0	0	0
0.796214	0	0	0	0	0	0	0	0
0.893367	0	0	0	0	0	0	0	0
1.002374	0	0	0	0	0	0	0	0
1.124683	0	0	0	0	0	0	0	0
1.261915	0	0	0	0	0	0	0	0
1.415892	0	0	0	0	0	0	0	0
1.588656	0.056177	0.05618	0.15100	0.15100	0	0	0	0
1.782502	0.22320	0.27938	0.46515	0.61615	0	0	0	0
2.000000	0.539337	0.81872	0.836673	1.45282	0	0	0	0

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Grain size	Milled 1	Cumulated	Milled 2	Cumulated	Unmilled 1	Cumulated	Unmilled 2	Cumulated
[µm]	[%]	[%]	[%]	[%]	[%}	[%]	[%]	[%]
2.244037	0.852564	1.67128	1.233192	2.68601	0.01392	0.01392	0.01020	0.01020
2.517851	1.21862	2.88990	1.63320	4.31921	0.13442	0.14834	0.09849	0.10869
2.825075	1.580531	4.47043	2.007826	6.32704	0.20057	0.34890	0.14597	0.25466
3.169786	1.926663	6.39710	2.345377	8.67241	0.28403	0.63294	0.20547	0.46013
3.556559	2.236457	8.63355	2.628511	11.3009	0.34373	0.97667	0.24800	0.70813
3.990525	2.513705	11.1473	2.865811	14.1667	0.41024	1.38691	0.29599	1.00412
4.477442	2.749365	13.8966	3.053163	17.2199	0.47507	1.86197	0.34375	1.34787
5.023773	2.954687	16.8513	3.203675	20.4236	0.54708	2.40905	0.39783	1.74570
5.636766	3.127973	19.9793	3.31905	23.7426	0.63001	3.03905	0.46085	2.20655
6.324555	3.272874	23.2522	3.404728	27.1474	0.72803	3.76709	0.53557	2.74211
7.096268	3.392162	26.6443	3.465318	30.6127	0.84643	4.61351	0.62531	3.36742
7.962143	3.48840	30.1327	3.506589	34.1193	0.98754	5.60106	0.73074	4.09817
8.933672	3.568121	33.7008	3.535328	37.6546	1.16018	6.76124	0.85679	4.95496
10.02375	3.631657	37.3325	3.555059	41.2096	1.36111	8.12235	0.99951	5.95447
11.24683	3.683893	41.0164	3.569819	44.7795	1.59962	9.72197	1.16398	7.11845
12.61915	3.72429	44.7407	3.58027	48.3597	1.87012	11.5921	1.34469	8.46314
14.15892	3.75322	48.4939	3.585353	51.9451	2.17535	13.7674	1.54227	10.0054
15.88657	3.769029	52.2629	3.58231	55.5274	2.51045	16.2779	1.75276	11.7582
17.82502	3.769976	56.0329	3.567741	59.0951	2.86709	19.1450	1.97100	13.7292
20.00000	3.753392	59.7863	3.53665	62.6318	3.24339	22.3884	2.19688	15.9260
22.44037	3.717287	63.5036	3.48560	66.1174	3.61707	26.0054	2.41968	18.3457
25.17851	3.657475	67.1611	3.40859	69.5260	3.98807	29.9935	2.64360	20.9893
28.25075	3.573445	70.7345	3.305377	72.8314	4.32801	34.3215	2.85738	23.8467
31.69786	3.46080	74.1953	3.171773	76.0031	4.63078	38.9523	3.06509	26.9118
35.56559	3.31940	77.5147	3.01000	79.0131	4.87449	43.8268	3.26198	30.1738
39.90525	3.148329	80.6630	2.82200	81.8351	5.04654	48.8733	3.44890	33.6227
44.77442	2.946214	83.6092	2.609879	84.4450	5.13473	54.0080	3.62725	37.2499
50.23773	2.719073	86.3283	2.383234	86.8282	5.12980	59.1378	3.79362	41.0435
56.36766	2.462817	88.7911	2.140922	88.9692	5.02661	64.1644	3.95212	44.9957
63.24555	2.19289	90.9840	1.898886	90.8680	4.83153	68.9960	4.09380	49.0895
70.96268	1.907426	92.8915	1.655336	92.5234	4.54615	73.5421	4.21881	53.3083
79.62143	1.623508	94.5150	1.423568	93.9469	4.19047	77.7326	4.31596	57.6242
89.33672	1.347231	95.8622	1.205782	95.1527	3.77840	81.5110	4.37706	62.0013
100.2374	1.089375	96.9516	1.007478	96.1602	3.33188	84.8429	4.39017	66.3915
112.4683	0.859484	97.8111	0.832858	96.9931	2.87697	87.7198	4.34542	70.7369
126.1915	0.658389	98.4694	0.679792	97.6729	2.42601	90.1458	4.23229	74.9692
141.5892	0.493928	98.9634	0.552355	98.2252	2.01178	92.1576	4.05023	79.0194
158.8656	0.360018	99.3234	0.444577	98.6698	1.63428	93.7919	3.79334	82.8127
178.2502	0.25773	99.5811	0.358132	99.0279	1.31582	95.1077	3.47607	86.2888
200.0000	0.179216	99.7603	0.287088	99.3150	1.05002	96.1577	3.10126	89.3901
224.4037	0.123632	99.8840	0.22820	99.5432	0.83833	96.9961	2.68858	92.0787

Grain size	Milled 1	Cumulated	Milled 2	Cumulated	Unmilled 1	Cumulated	Unmilled 2	Cumulated
[µm]	[%]	[%]	[%]	[%]	[%}	[%]	[%]	[%]
251.7851	0.06767	99.9516	0.180583	99.7238	0.67220	97.6683	2.25570	94.3344
282.5075	0.038908	99.9905	0.134364	99.8581	0.54188	98.2102	1.81802	96.1524
316.9786	0.009459	100.000	0.100311	99.9585	0.44089	98.6510	1.40533	97.5577
355.6559	0	100.000	0.041542	100.000	0.35788	99.0089	1.02406	98.5818
399.0525	0	100.000	0	100.000	0.29037	99.2993	0.69620	99.2780
447.7442	0	100.000	0	100.000	0.23120	99.5305	0.43673	99.7147
502.3773	0	100.000	0	100.000	0.18051	99.7110	0.23275	99.9474
563.6766	0	100.000	0	100.000	0.13801	99.8490	0.04500	99.9924
632.4555	0	100.000	0	100.000	0.09631	99.9453	0.00756	100.000
709.6268	0	100.000	0	100.000	0.05467	100.000	0	100.000
796.2143	0	100.000	0	100.000	0	100.000	0	100.000
893.3672	0	100.000	0	100.000	0	100.000	0	100.000
1002.374	0	100.000	0	100.000	0	100.000	0	100.000
1124.683	0	100.000	0	100.000	0	100.000	0	100.000
1261.915	0	100.000	0	100.000	0	100.000	0	100.000
1415.892	0	100.000	0	100.000	0	100.000	0	100.000
1588.656	0	100.000	0	100.000	0	100.000	0	100.000
1782.502	0	100.000	0	100.000	0	100.000	0	100.000
2000.000	0	100.000	0	100.000	0	100.000	0	100.000

B.2 Material Testing

B.3 Casting of Mortar Specimens

Task	Start	Finish	July	August	September	October	November	December	January	Feb
Chloride Diffusion	01-07-2013	17-02-2014								
Casting	01-07-2013									-
Curing	01-07-2013	17-09-2013								
Epoxy paint	17-09-2013	23-09-2013								
Saltwater, specimen A	23-09-2013	11-11-2013								
Saltwater, specimen B	23-09-2013	30-12-2013								
Saltwater, specimen C	23-09-2013	17-02-2014								
Material Testing	03-09-2013	20-11-2013								
Casting	03-09-2013									
Curing	03-09-2013	20-11-2013								
Compression strength test	20-11-2013									
Rate of Corrosion with Destilled Water	17-09-2013	17-02-2014								
Casting	17-09-2013									
Curing	17-09-2013	16-10-2013								
Epoxy paint	16-10-2013	21-10-2013								
Saltwater/air cycle, electrical potential specimens	21-10-2013	17-02-2014								
Saltwater/air cycle, visual test, specimen A	21-10-2013	02-12-2013								
Saltwater/air cycle, visual test, specimen B	21-10-2013	17-02-2014								
Saltwater/air cycle, visual test, specimen C	21-10-2013	17-02-2014								
Rate of Corrosion with Saltwater	30-09-2013	17-02-2014								
Casting	30-09-2013						_			
Curing	30-09-2013	29-10-2013								
Epoxy paint	29-10-2013	04-11-2013								
Saltwater/air cycle, electrical potential specimens	04-11-2013	17-02-2014								
Saltwater/air cycle, visual test, specimen A	04-11-2013	16-12-2013								
Saltwater/air cycle, visual test, specimen B	04-11-2013	17-02-2014								
Saltwater/air cycle, visual test, specimen C	04-11-2013	17-02-2014								

В

No	Sample type	Reinforced	Cement	LSA-depot	Sand	Water	Extra water	Cast	Demoulded	Note
	bampie type	nemoreeu	[g]	[g]	[g]	[g]	[g]	[hh:mm]	[hh:mm]	
3	5% sand	No	450.00	67.50	1282.50	225.00	6.02	-	-	The exact measurements are unknown.
5	10% cement	No	405.00	45.00	1350.00	225.00	6.00	-	-	The exact measurements are unknown.
6	Reference	No	450.00	0.00	1350.00	225.00	0.00	-	-	The exact measurements are unknown.
8	Reference	No	450.02	0.00	1350.02	225.01	0.00	09:03	08:57	
9	10% cement	No	405.03	45.02	1350.04	225.01	0.00	09:37	09:32	
10	5% sand	No	450.04	67.52	1282.54	225.06	8.94	09:58	09:56	
11	Reference	Yes	450.02	0.00	1350.02	225.01	0.00	13:25	13:16	A little more dry than the reference
12	10% cement	Yes	405.00	45.07	1350.00	225.09	0.00	13:59	13:52	A little more dry than the reference
13	Reference	Yes	450.01	0.00	1350.02	225.01	0.00	13:41	13:35	Fine consistency.
14	10% cement	Yes	405.07	45.04	1350.04	225.03	0.00	14:18	14:14	Seemed fine during the mixing but a little dry during the vibration
15	Reference	Yes	450.05	0.00	1350.01	225.08	0.00	09:18	09:15	Cast with salt water. The consistency seemed fine.
16	10% cement	Yes	405.02	45.07	1350.07	225.07	0.00	09:37	09:34	Cast with salt water. The consistency seemed fine.
17	Reference	Yes	450.00	0.00	1350.09	225.07	0.00	09:51	09:45	Cast with salt water. The consistency seemed fine.
18	10% cement	Yes	405.02	45.03	1350.00	225.02	0.00	10:07	10:04	Cast with salt water. The consistency seemed fine.
19	10% cement	No	405.03	45.02	1350.02	225.00	0.00	13:35	13:25	Seemed a little more wet than usually.
20	10% cement	No	405.01	45.00	1350.01	225.02	6.02	09:47	09:47	Cast with washed LSA-depot. Fine consistency after addition of extra water.

Compressive Strength

The compressive strength is determined according to DS/EN 196-1:2005.

Specimen type		No.	Weight [g]	Height [mm]	Width [mm]	Length [mm]	Load 1 [kN]	Load 2 [kN]	Area [mm²]	Compressive strength 1 [MPa]	Compressive strength 2 [MPa]	1st mean comp. strength [MPa]	+10% [MPa]	-10% [MPa]	2nd mean comp. strength [MPa]	+10% [MPa]	-10% [MPa]	COMPRESSIVE STRENGTH [MPa]	Standard deviation [MPa]
		А	590.71	40.0	40.8	160.5	99	108	1631	60.7	66.2								
Reference, 78d	8	В	585.31	40.0	40.5	160.5	105	105	1620	64.8	64.8	65.4	72.0	58.9				65.4	2.73
		С	590.87	40.1	40.6	161.0	111	110	1625	68.3	67.7		/ 2.10						
		Α	585.68	40.1	40.5	160.5	94	101	1620	58.0	62.3								
10% cement, 78d	9	В	582.70	40.0	40.6	160.5	95	83	1624	58.5	51.1	58.8	64.7	52.9	60.3	66.3	54.3	60.3	2.03
		С	580.91	40.0	40.5	160.5	98	101	1622	60.4	62.3								
		Α	582.23	40.1	40.5	161.0	110	116	1618	68.0	71.7								
5% sand, 78d	10	В	577.15	40.1	40.6	160.5	100	110	1622	61.6	67.8	65.5	72.1	2.1 59.0				65.5	4.48
		С	584.63	40.0	40.7	160.5	97	105	1627	59.6	64.5								

The height and width of the specimens are found as the mean value of three measurements spread out over the entire specimen.

S Chloride Content in Mortar

The chloride content in the mortar is determined by:

$$C_{Cl}[\%] = \frac{V_{AgNO3} \cdot C_{AgNO3} \cdot M_{Cl}}{m_{sample}} \cdot \frac{1}{10}$$

where

 V_{AgNO3} is the amount of silver nitrate used to reach equilibrium [mL]

 C_{AgNO3} is the silver nitrate concentration which is found by calibration [mol/L]

 M_{Cl} is the molar mass of chloride which is 35.45 g/mol

 m_{sample} is the mass of sample tested [g]

Calibration of $AgNO_3$							
Cal A [mL]	1.0095						
Cal B [mL]	1.0096						
Cal C [mL]	1.0101						
Mean [mL]	1.0097						
•							
C _{AgNO3} [mol/L]	0.0990						

Spacimon type			m _{mortar}	m _{cementitious}	M _{CI}	C _{AgNO3}	m _{AgNO3}	Chloride content	Mean	
specimen type	NO.		[g]	[g]	[g/mol]	[mol/L]	[mL]	[%]	[%]	
Poforonco	0	-	5.00	1.43	35.45	0.0990	0.6021	0.148	0.154	
Reference	0	П	5.00	1.43	35.45	0.0990	0.6531	0.160	0.154	
10% comont	0	Ι	5.00	1.43	35.45	0.0990	0.6540	0.161	0.150	
10% cement	9	II	5.00	1.43	35.45	0.0990	0.6389	0.157	0.159	

Leaching

LEACHING OF PURE LSA-DEPOT

		А	В	С	Mean	Standard deviation
Calcium (Ca)	[mg/kg]	2210	2090	2040	2110	87.4
Potassium (K)	[mg/kg]	2320	2140	2190	2220	92.9
Sodium (Na)	[mg/kg]	274	328	255	286	38.1
Chromium (Cr)	[mg/kg]	1.51	1.59	1.49	1.53	0.05
Phosphorus (P)	[mg/kg]	0.76	1.08	1.15	0.99	0.21
Aluminium (Al)	[mg/kg]	0.07	0.10	0.12	0.10	0.02
Copper (Cu)	[mg/kg]	0.05	0.08	0.04	0.06	0.02
Lead (Pb)	[mg/kg]	0.08	0.03	0.06	0.06	0.03
Magnesium (Mg)	[mg/kg]	0.02	0.01	0.01	0.01	0.00
Zinc (Zn)	[mg/kg]	0.02	0.01	0.00	0.01	0.01
Iron (Fe)	[mg/kg]	0.00	0.00	0.00	0.00	0.00
Nickel (Ni)	[mg/kg]	0.00	0.00	0.01	0.00	0.01
Arsen (As)	[mg/kg]	0.00	0.00	0.00	0.00	0.00
Cadmium (Cd)	[mg/kg]	0.00	0.00	0.00	0.00	0.00

Leaching	OF	MORTAR	$\rm WITH$	5%	SAND	REPLACEMENT
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		А	В	С	Mean	Standard deviation
Calcium (Ca)	[mg/kg]	1120	1230	1210	1190	58.6
Potassium (K)	[mg/kg]	280	250	270	270	15.3
Sodium (Na)	[mg/kg]	182	162	170	171	10.0
Phosphorus (P)	[mg/kg]	0.77	1.25	0.75	0.92	0.28
Aluminium (Al)	[mg/kg]	0.15	0.17	0.17	0.17	0.01
Chromium (Cr)	[mg/kg]	0.07	0.08	0.07	0.07	0.00
Lead (Pb)	[mg/kg]	0.04	0.07	0.05	0.05	0.01
lron (Fe)	[mg/kg]	0.02	0.05	0.05	0.04	0.02
Copper (Cu)	[mg/kg]	0.02	0.02	0.03	0.02	0.00
Nickel (Ni)	[mg/kg]	0.01	0.01	0.02	0.02	0.01
Magnesium (Mg)	[mg/kg]	0.00	0.01	0.01	0.01	0.00
Zinc (Zn)	[mg/kg]	0.00	0.00	0.00	0.00	0.00
Arsen (As)	[mg/kg]	0.00	0.00	0.00	0.00	0.00
Cadmium (Cd)	[mg/kg]	0.00	0.00	0.00	0.00	0.00

		А	В	С	Mean	Standard deviation
Calcium (Ca)	[mg/kg]	1190	1270	1250	1240	41.6
Potassium (K)	[mg/kg]	220	240	270	240	25.2
Sodium (Na)	[mg/kg]	139	159	161	153	12.0
Phosphorus (P)	[mg/kg]	0.60	0.55	1.01	0.72	0.25
Aluminium (Al)	[mg/kg]	0.14	0.17	0.18	0.16	0.02
Chromium (Cr)	[mg/kg]	0.09	0.08	0.09	0.09	0.00
lron (Fe)	[mg/kg]	0.02	0.11	0.07	0.07	0.04
Lead (Pb)	[mg/kg]	0.03	0.05	0.05	0.04	0.01
Copper (Cu)	[mg/kg]	0.02	0.03	0.03	0.02	0.00
Magnesium (Mg)	[mg/kg]	0.00	0.02	0.01	0.01	0.01
Arsen (As)	[mg/kg]	0.00	0.00	0.04	0.01	0.03
Zinc (Zn)	[mg/kg]	0.00	0.00	0.00	0.00	0.00
Nickel (Ni)	[mg/kg]	0.01	0.00	0.00	0.00	0.01
Cadmium (Cd)	[mg/kg]	0.00	0.00	0.00	0.00	0.00

Leaching of mortar with 10% cement replacement

LEACHING OF REFERENCE MORTAR

		А	В	С	Mean	Standard deviation
Calcium (Ca)	[mg/kg]	1110	1260	1180	1180	75.1
Potassium (K)	[mg/kg]	220	210	220	220	5.8
Sodium (Na)	[mg/kg]	163	158	176	166	9.2
Phosphorus (P)	[mg/kg]	0.53	0.89	0.53	0.65	0.21
Aluminium (Al)	[mg/kg]	0.13	0.17	0.16	0.15	0.02
Chromium (Cr)	[mg/kg]	0.10	0.09	0.08	0.09	0.01
lron (Fe)	[mg/kg]	0.03	0.06	0.03	0.04	0.02
Lead (Pb)	[mg/kg]	0.00	0.06	0.04	0.03	0.03
Copper (Cu)	[mg/kg]	0.01	0.02	0.02	0.02	0.00
Nickel (Ni)	[mg/kg]	0.02	0.02	0.00	0.01	0.01
Magnesium (Mg)	[mg/kg]	0.00	0.00	0.00	0.00	0.00
Zinc (Zn)	[mg/kg]	0.00	0.00	0.00	0.00	0.00
Arsen (As)	[mg/kg]	0.00	0.00	0.00	0.00	0.00
Cadmium (Cd)	[mg/kg]	0.00	0.00	0.00	0.00	0.00
B.4 Chloride Diffusion

The numbering of the mortar slices can be seen in figure 49.

5mm 5mm 5mm 5mm 10mm	10mm	20mm	20mm	20mm	20mm	10mm	10mm 5mm 5mm 5mm
+++++++ +++++++++++++++++++++++++++++	• • •			•	•	→ → ·	↔↔↔↔
<pre> I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I I</pre>		VII	VIII	XVI	XV	XIV	XIII IXIIIXIIXIIX 1 1 1 1 1
			160) mm			

Figure 49: Numbering of mortar slices for chloride diffusion tests

The chloride content in the mortar is determined by:

$$C_{Cl}[\%] = \frac{V_{AgNO3} \cdot C_{AgNO3} \cdot M_{Cl}}{m_{sample}} \cdot \frac{1}{10}$$

where

 V_{AgNO3} is the amount of silver nitrate used to reach equilibrium [mL] C_{AgNO3} is the silver nitrate concentration which is found by calibration [mol/L] M_{Cl} is the molar mass of chloride which is 35.45 g/mol m_{sample} is the mass of sample tested [g]

Calibration of $AgNO_3$

For 7 week specimens:									
A B C Mean									
m _{NaCl}	[mL]	1.000	1.000	1.000	1.000				
m _{AgNO3}	[mL]	0.9951	0.9965	1.0006	0.9974				
Konc. $AgNO_3$	[mol/L]				0.1003				

For 14 week specimens:										
	A B C Mean									
m _{NaCl}	[mL]	1.000	1.000	1.000	1.000					
m _{AgNO3}	[mL]	1.0095	1.0096	1.0101	1.0097					
Konc. $AgNO_3$	[mol/L]				0.0990					

	For 21 week specimens:									
A B C Mean										
m _{NaCl}	[mL]	1.000	1.000	1.000	1.000					
m _{AgNO3}	[mL]	0.9492	0.9616	0.9662	0.9590					
Konc. AgNO ₃	[mol/L]				0.1043					

Chloride Content

Specimen	Slice	Sample	Test	Pos. 1	Pos. 2	Centre	V _{AqNO3}	C _{AaNO3}	m _{sample}	M _{CI}	Chloride
			period	[cm]	[cm]	[cm]	[mL]	[mol/L]	[g]	[g/mol]	content
											[%]
	Ι			0.00	0.50	0.25	5.8902		5.00		0.42
	II			0.50	1.00	0.75	3.6290		5.00		0.26
	III			1.00	1.50	1.25	1.8505		5.00		0.13
	IV			1.50	2.00	1.75	0.8593		5.00		0.06
	V			2.00	3.00	2.50	0.5720		5.00		0.04
	VI			3.00	4.00	3.50	0.5907		5.00		0.04
	VII	Sample 2% sand 10% cement		4.00	6.00	5.00	0.6136		5.00		0.04
30	VIII	san	7 wooks	6.00	8.00	7.00	0.5518	0 1003	5.00	25 / 5	0.04
27	IX	%	/ WEEKS	0.00	0.50	0.25	5.3526	0.1005	5.00	55.45	0.38
	Х	ы		0.50	1.00	0.75	3.4722		5.00		0.25
	XI			1.00	1.50	1.25	1.7385		5.00		0.12
	XII			1.50	2.00	1.75	0.9236		5.00		0.07
	XIII			2.00	3.00	2.50	0.5125		5.00		0.04
	XIV			3.00	4.00	3.50	0.4225		5.00		0.03
	XV			4.00	6.00	5.00	0.4996	-	5.00		0.04
	XVI			6.00	8.00	7.00	0.5118		5.00		0.04
	Ι			0.00	0.50	0.25	5.6622		5.00		0.40
	II			0.50	1.00	0.75	4.0215		5.00		0.29
	III			1.00	1.50	1.25	2.1838		5.00		0.16
	IV			1.50	2.00	1.75	1.3400		5.00		0.10
	V			2.00	3.00	2.50	0.5855		5.00		0.04
	VI			3.00	4.00	3.50	0.4216		5.00		0.03
	VII	ent		4.00	6.00	5.00	0.3550		5.00		0.03
54	VIII	em	7 wooks	6.00	8.00	7.00	0.4784	0 1003	5.00	35 / 5	0.03
57	IX	%	/ WEEKS	0.00	0.50	0.25	5.3463	0.1005	5.00	55.45	0.38
	Х	100		0.50	1.00	0.75	3.8394		5.00		0.27
	XI			1.00	1.50	1.25	2.3936		5.00		0.17
	XII			1.50	2.00	1.75	1.1342		5.00		0.08
	XIII			2.00	3.00	2.50	0.4868		5.00		0.03
	XIV			3.00	4.00	3.50	0.4025		5.00		0.03
	XV			4.00	6.00	5.00	0.3820		5.00		0.03
	XVI			6.00	8.00	7.00	0.4132		5.00		0.03

Specimen	Slice	Sample	Test	Pos. 1	Pos. 2	Centre	V _{AgNO3}	C _{AgNO3}	m _{sample}	M _{CI}	Chloride
			period	[cm]	[cm]	[cm]	[mL]	[mol/L]	[g]	[g/mol]	content
											[70]
	Ι			0.00	0.50	0.25	5.3052		5.00		0.38
	II			0.50	1.00	0.75	3.7042		5.00		0.26
	III			1.00	1.50	1.25	2.1791		5.00		0.15
	IV			1.50	2.00	1.75	0.8740		5.00		0.06
	V			2.00	3.00	2.50	0.3702		$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	0.03	
	VI			3.00	4.00	3.50	0.3703		5.00	35.45	0.03
	VII	e		4.00	6.00	5.00	0.3364		5.00 5.00 5.45	0.02	
64	VIII	ene	7 wooks	6.00	8.00	7.00	0.3810 0.1003 5.00 35.45	0.03			
UA	IX	efer	7 WEEKS	0.00	0.50	0.25	5.8838	0.1005	5.00	35.45	0.42
	Х	Å		0.50	1.00	0.75	3.4837		5.00		0.25
	XI			1.00	1.50	1.25	1.8440		5.00		0.13
	XII			1.50	2.00	1.75	0.8432		5.00		0.06
	XIII			2.00	3.00	2.50	0.3590		5.00		0.03
	XIV			3.00	4.00	3.50	0.4395		5.00		0.03
	XV			4.00	6.00	5.00	0.3713		5.00		0.03
	XVI			6.00	8.00	7.00	0.3756		5.00		0.03
	Ι			0.00	0.50	0.25	6.2490		5.00		0.44
	II			0.50	1.00	0.75	5.0635		5.00		0.36
	III			1.00	1.50	1.25	3.9612		5.00		0.28
	IV			1.50	2.00	1.75	2.3040		5.00		0.16
	V			2.00	3.00	2.50	0.8439		5.00		0.06
	VI			3.00	4.00	3.50	0.7721		5.00		0.05
	VII	σ		4.00	6.00	5.00	0.5288		5.00		0.04
20	VIII	san	14 wooks	6.00	8.00	7.00	0.4910	0.0000	5.00	25 45	0.03
JD	IX	%	14 weeks	0.00	0.50	0.25	5.9401	0.0990	5.00	55.45	0.42
	Х	Ň		0.50	1.00	0.75	4.3728		5.00		0.31
	XI			1.00	1.50	1.25	3.1500		5.00		0.22
	XII			1.50	2.00	1.75	1.9485		5.00		0.14
	XIII			2.00	3.00	2.50	0.7480		5.00		0.05
	XIV]		3.00	4.00	3.50	0.5818		5.00		0.04
	XV			4.00	6.00	5.00	0.5284		5.00	-	0.04
	XVI			6.00	8.00	7.00	0.5293		5.00		0.04

Specimen	Slice	Sample	Test	Pos. 1	Pos. 2	Centre	V _{AgNO3}	C _{AgNO3}	m _{sample}	M _{CI}	Chloride
			period	[cm]	[cm]	[cm]	[mL]	[mol/L]	[g]	[g/mol]	content
											[70]
	Ι			0.00	0.50	0.25	5.8444		5.00		0.41
	II			0.50	1.00	0.75	4.8229		5.00		0.34
	III			1.00	1.50	1.25	3.5347		5.00		0.25
	IV			1.50	2.00	1.75	2.3403		5.00		0.16
	V			2.00	3.00	2.50	0.9363		5.00 5.00 5.00 5.00 5.00 5.00 5.00 5.00 5.00 5.00 5.00 5.00 5.00 5.00	0.07	
	VI			3.00	4.00	3.50	0.4246		5.00	35.45	0.03
	VII	ent		4.00	6.00	5.00	0.0942		5.00		0.01
58	VIII	em	14 wooks	6.00	8.00	7.00	0.4585	0.0000	5.00	35.45	0.03
70	IX	0 %	14 WEEKS	0.00	0.50	0.25	5.9567	0.0990	5.00	55.45	0.42
	Х	10%		0.50	1.00	0.75	4.5884		5.00		0.32
	XI			1.00	1.50	1.25	3.4275		5.00	-	0.24
	XII			1.50	2.00	1.75	2.3389		5.00		0.16
	XIII			2.00	3.00	2.50	1.0679		5.00		0.07
	XIV			3.00	4.00	3.50	0.3922		5.00		0.03
	XV			4.00	6.00	5.00	0.6134		5.00		0.04
	XVI			6.00	8.00	7.00	0.4889		5.00		0.03
	Ι			0.00	0.50	0.25	6.3634		5.00		0.45
	II			0.50	1.00	0.75	4.3534		5.00		0.31
	III			1.00	1.50	1.25	3.3911		5.00		0.24
	IV			1.50	2.00	1.75	1.9852		5.00		0.14
	V			2.00	3.00	2.50	0.6979		5.00		0.05
	VI			3.00	4.00	3.50	0.6314		5.00		0.04
	VII	e		4.00	6.00	5.00	0.5351		5.00		0.04
(P	VIII	eno	1.4 weeks	6.00	8.00	7.00	0.5370	0.0000	5.00	25.45	0.04
00	IX	efer	14 weeks	0.00	0.50	0.25	6.6599	0.0990	5.00	35.45	0.47
	Х	Å		0.50	1.00	0.75	4.6699		5.00		0.33
	XI			1.00	1.50	1.25	3.1941		5.00		0.22
	XII	1		1.50	2.00	1.75	1.7618		5.00	1	0.12
	XIII	1		2.00	3.00	2.50	0.9779		5.00	1	0.07
	XIV	1		3.00	4.00	3.50	0.4702		5.00	1	0.03
	XV			4.00	6.00	5.00	0.6091		5.00		0.04
	XVI			6.00	8.00	7.00	1.0754		5.00		0.08

Specimen	Slice	Sample	Test	Pos. 1	Pos. 2	Centre	V _{AgNO3}	C _{AgNO3}	m _{sample}	M _{CI}	Chloride
			period	[cm]	[cm]	[cm]	[mL]	[mol/L]	[g]	[g/mol]	content
											[90]
	Ι			0.00	0.50	0.25	6.2312		5.00		0.46
	II			0.50	1.00	0.75	5.4089		5.00		0.40
	III			1.00	1.50	1.25	4.1972		5.00		0.31
	IV			1.50	2.00	1.75	2.7059		5.00		0.20
	V			2.00	3.00	2.50	1.4428		nol/L] [g] [g/mol] co 5.00 0 0 5.00 0 0 5.00 0 0 5.00 0 0 5.00 0 0 5.00 0 0 5.00 5.00 0 5.00 5.00 0 5.00 5.00 0 5.00 5.00 0 5.00 0 0 5.00 0 0 5.00 0 0 5.00 0 0 5.00 0 0 5.00 0 0 5.00 0 0 5.00 0 0 5.00 0 0 5.00 0 0 5.00 0 0 5.00 5.00 0 5.00 5.00 0	0.11	
	VI			3.00	4.00	3.50	0.6054		5.00	M _{CI} [g/mol]	0.04
	VII	g		4.00	6.00	5.00	0.7495		5.00		
30	VIII	21 weeks 0.00 0.50 0.25 6.0178 0.1043 5.00 35.4	25.45	0.04							
50	IX	%	ZI WEEKS	0.00	0.50	0.25	6.0178	0.1045	5.00	35.45	0.44
	Х	ъ		0.50	1.00	0.75	4.9849		5.00		0.37
	XI			1.00	1.50	1.25	3.7012		5.00		0.27
	XII			1.50	2.00	1.75	2.7825		5.00		0.21
	XIII			2.00	3.00	2.50	1.4003		5.00		0.10
	XIV			3.00	4.00	3.50	0.6293		5.00		0.05
	XV			4.00	6.00	5.00	0.4397		5.00		0.03
	XVI			6.00	8.00	7.00	0.5255		5.00		0.04
	Ι			0.00	0.50	0.25	6.3497		5.00		0.47
	II			0.50	1.00	0.75	5.1149		5.00		0.38
	III			1.00	1.50	1.25	3.7794		5.00		0.28
	IV			1.50	2.00	1.75	3.0719		5.00		0.23
	V			2.00	3.00	2.50	2.0864		5.00		0.15
	VI			3.00	4.00	3.50	0.7654		5.00		0.06
	VII	ent		4.00	6.00	5.00	0.5175		5.00		0.04
FC	VIII	em	21 wooks	6.00	8.00	7.00	0.9093	0 1042	5.00	25 45	0.07
50	IX	0 %	ZI WEEKS	0.00	0.50	0.25	5.9191	0.1045	5.00	55.45	0.44
	Х	10%		0.50	1.00	0.75	5.0603		5.00		0.37
	XI			1.00	1.50	1.25	3.7648		5.00		0.28
	XII			1.50	2.00	1.75	2.7982		5.00		0.21
	XIII	1		2.00	3.00	2.50	1.6291		5.00		0.12
	XIV	1		3.00	4.00	3.50	0.4949		5.00		0.04
	XV			4.00	6.00	5.00	0.4546		5.00	-	0.03
	XVI			6.00	8.00	7.00	0.4973		5.00		0.04

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Specimen	Slice	Sample	Test period	Pos. 1 [cm]	Pos. 2 [cm]	Centre [cm]	V _{AgNO3} [mL]	C _{AgNO3} [mol/L]	m _{sample} [g]	M _{CI} [g/mol]	Chloride content [%]
	Ι			0.00	0.50	0.25	7.3331		5.00		0.54
	II			0.50	1.00	0.75	5.0149		5.00		0.37
	III			1.00	1.50	1.25	3.7341		5.00		0.28
	IV			1.50	2.00	1.75	2.1068		5.00		0.16
	V			2.00	3.00	2.50	0.8473		5.00		0.06
	VI			3.00	4.00	3.50	0.5658		5.00	35.45	0.04
	VII	e		4.00	6.00	5.00	0.4285	0.1042	5.00		0.03
60	VIII	en	21 wooks	6.00	8.00	7.00	0.4286		5.00		0.03
00	IX	efer	ZI WEEKS	0.00	0.50	0.25	6.1008	0.1045	5.00		0.45
	Х	Ř		0.50	1.00	0.75	5.3523		5.00		0.40
	XI			1.00	1.50	1.25	4.2083		5.00		0.31
	XII			1.50	2.00	1.75	2.6509		5.00		0.20
	XIII			2.00	3.00	2.50	1.4655		5.00		0.11
	XIV			3.00	4.00	3.50	0.8184		5.00	-	0.06
	XV			4.00	6.00	5.00	0.3972		5.00		0.03
	XVI			6.00	8.00	7.00	0.3488		5.00		0.03

				7 WEEKS TEST F	PERIOD						
	Distance from surface [cm]	Chloride content [%]		Distance from surface [cm]	Chloride content [%]		Distance from surface [cm]	Chloride content [%]			
	0.25	0.40		0.25	0.39		0.25	0.40			
	0.75	0.25		0.75	0.28		0.75	0.26			
24	1.25	0.13	5A	1.25	0.16	C A	1.25	0.14			
3A	1.75	0.06		1.75	0.09	bА	1.75	0.06			
F 0(a a a b	2.50	0.04	10%	2.50	0.04	Def	2.50	0.03			
5% sand	3.50	0.04	cement	3.50	0.03	кет	3.50	0.03			
	5.00	0.04		5.00	0.03		5.00	0.03			
	7.00	0.04		7.00	0.03		7.00	0.03			
	14 WEEKS TEST PERIOD										
	Distance from surface [cm]	Chloride content [%]		Distance from surface [cm]	Chloride content [%]		Distance from surface [cm]	Chloride content [%]			
	0.25	0.43	58	0.25	0.41		0.25	0.46			
	0.75	0.33		0.75	0.33		0.75	0.32			
20	1.25	0.25	5B	1.25	5 0.33 0.75 5 0.24 6B	0.23					
3B	1.75	0.15		1.75	0.16	6B	1.75	0.13			
F0/ cond	2.50	0.06	10%	2.50	0.07	Def	2.50	0.06			
5% Sano	3.50	0.05	cement	3.50	0.03	Kei	3.50	0.04			
	5.00	0.04		5.00	0.02		5.00	0.04			
	7.00	0.04		7.00	0.03		7.00	0.06			
				21 WEEKS TEST	PERIOD						
	Distance from surface [cm]	Chloride content [%]		Distance from surface [cm]	Chloride content [%]		Distance from surface [cm]	Chloride content [%]			
	0.25	0.45		0.25	0.45		0.25	0.50			
	0.75	0.38		0.75	0.38		0.75	0.38			
20	1.25	0.29	5C	1.25	0.28		1.25	0.29			
50	1.75	0.20		1.75	0.22		1.75	0.18			
F0/ cond	2.50	0.11	10%	2.50	0.14	Def	2.50	0.09			
2% 29110	3.50	0.05	cement	3.50	0.05	Kei	3.50	0.05			
	5.00	0.04		5.00	0.04		5.00	0.03			
	7.00	0.04		7.00	0.05		7.00	0.03			

Since the chloride content is measured with duplicate determination in each distance from the surface the mean values are found and presented in the table on the following page.



Figure 50: Comparison of the chloride diffusion after 14 weeks in salt water



Figure 51: Comparison of the test periods for chloride diffusion into 5% sand replacement samples (left) and reference samples (right)

B.5 Chloride Binding

The amount of bound chloride is determined by:

$$Cl_{bound} = \frac{(Cl_{before} - Cl_{after}) \cdot 0.015L \cdot 1000mg/g}{5.00g}$$

Where Cl_{before} is the calculated amount of chloride ions in the salt water and Cl_{after} is measured by titration with silver nitrate.

$$Cl_{after} = \frac{m_{AgNO3} \cdot C_{AgNO3} \cdot M_{Cl}}{m_{LSA-solution}}$$

where

 m_{AgNO3} is the amount of silver nitrate used to reach equilibrium

 C_{AgNO3} is the silver nitrate concentration which is found by calibration

 M_{Cl} is the molar mass of chloride which is 35.45 g/mol

 $m_{LSA-solution}$ is the amount of sample solution which is used for titration

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Unwashed	I: Screening	1	2	3	Mean	Washed: S	Screening	1	2	3	Mean
m _{NaCl}	[mL]	1.0000	1.0000	1.0000	1.0000	m _{NaCl}	[mL]	1.0000	1.0000	1.0000	1.0000
m _{AgNO3}	[mL]	1.0082	1.0067	1.0076	1.0075	m _{AgNO3}	[mL]	0.9978	0.9994	0.9988	0.9987
C _{AgNO3}	[mol/L]				0.0993	C _{AgNO3}	[mol/L]				0.1001
Unwashed	l: 2 weeks	1	2	3	Mean	Washed: 2	2 weeks	1	2	3	Mean
m _{NaCl}	[mL]	1.0000	1.0000	1.0000	1.0000	m _{NaCl}	[mL]	1.0000	1.0000	1.0000	1.0000
m _{AgNO3}	[mL]	1.0004	0.9996	0.9989	0.9996	m _{AgNO3}	[mL]	1.0028	1.0004	1.0058	1.0030
C _{AgNO3}	[mol/L]				0.1000	C _{AgNO3}	[mol/L]				0.0997
		-			-			-			-
Unwashed	l: 4 weeks	1	2	3	Mean	Washed: 4	1 weeks	1	2	3	Mean
m _{NaCl}	[mL]	1.0000	1.0000	1.0000	1.0000	m _{NaCl}	[mL]	1.0000	1.0000	1.0000	1.0000
m _{AgNO3}	[mL]	0.9949	0.9938	0.9843	0.9910	m _{AgNO3}	[mL]	0.9925	0.9922	0.9912	0.9920
C _{AgNO3}	[mol/L]				0.1009	C _{AgNO3}	[mol/L]				0.1008
Unwashed	l: 6 weeks	1	2	3	Mean	Washed: 6	5 weeks	1	2	3	Mean
m _{NaCl}	[mL]	1.0000	1.0000	1.0000	1.0000	m _{NaCl}	[mL]	1.0000	1.0000	1.0000	1.0000
m _{AgNO3}	[mL]	1.0102	1.0111	1.0093	1.0102	m _{AgNO3}	[mL]	1.0095	1.0096	1.0101	1.0097
C _{AgNO3}	[mol/L]				0.0990	C _{AgNO3}	[mol/L]				0.0990
Unwashed	l: 16 weeks	1	2	3	Mean	Washed: 1	L6 weeks	1	2	3	Mean
m _{NaCl}	[mL]	1.0000	1.0000	1.0000	1.0000	m _{NaCl}	[mL]	1.0000	1.0000	1.0000	1.0000
m _{AgNO3}	[mL]	0.9492	0.9616	0.9662	0.9590	m _{AgNO3}	[mL]	1.0142	1.0116	1.0098	1.0119
C _{AgNO3}	[mol/L]				0.1043	C _{AgNO3}	[mol/L]				0.0988

Screening of Chloride Binding

						Tes	t no.				
		А	В	С	D	E	F	G	Н	I	J
Test period	[weeks]	1	1	1	1	1	1	1	1	1	1
m _{LSA-depot}	[g]	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	0.00
C _{salt water}	[%]	0	1	3	10	20	0	1	3	10	3
C _{CI}	[g/L]	0.00	6.07	18.20	60.66	121.32	0.00	6.07	18.20	60.66	18.20
$V_{salt water}$	[mL]	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00
C _{AgNO3}	[mol/L]			0.0993					0.1001		
m _{LSA-solution}	[mL]	5.00	0.50	0.50	0.25	0.10	2.00	0.50	0.50	0.25	0.5
m _{HNO3}	[mL]	2	2	2	2	2	2	2	2	2	2
m _{AgNO3}	[mL]	2.3804	1.1092	2.8696	4.2555	-	0.2050	0.8437	2.4444	7.6954	2.5896
C _{CI} after	[g/L]	1.68	7.81	20.19	59.89	-	0.36	5.99	17.35	109.27	18.38
C _{CI} before	[g/L]	0.00	6.07	18.20	60.66	121.32	0.00	6.07	18.20	60.66	18.20
	-										
Clound	[g/L]	-1.68	-1.74	-2.00	0.77	-	-0.36	0.08	0.84	-48.61	-0.19
	[mg/g]	-5.03	-5.22	-5.99	2.30	-	-1.09	0.23	2.53	-145.82	-

Chloride Binding into Unwashed LSA-depot

			Test no.														
		A1	A2	A3	A4	A5	A6	A7	A8	A9	A10	A11	A12	A13	A14	A15	A16
Test period	[weeks]	2	2	2	2	4	4	4	4	6	6	6	6	16	16	16	16
m _{LSA-depot}	[g]	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00
C _{salt water}	[%]	0	1	3	5	0	1	3	5	0	1	3	5	0	1	3	5
C _{CI}	[g/L]	0.00	6.07	18.20	30.33	0.00	6.07	18.20	30.33	0.00	6.07	18.20	30.33	0.00	6.07	18.20	30.33
V _{salt water}	[mL]	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00
C _{AgNO3}	[mol/L]		0.1000				0.1	009			0.0	990			0.1	043	
m _{sample}	[mL]	2.00	0.50	0.50	0.50	5.00	0.50	0.50	0.50	5.00	0.50	0.50	0.50	2.00	0.50	0.50	0.50
m _{HNO3}	[mL]	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00
m _{AgNO3}	[mL]	0.3339	0.8763	2.5336	4.2323	0.8153	0.8866	2.5341	4.1705	0.8317	0.8921	2.5718	4.2742	0.3135	0.9247	2.6929	4.2719
C _{ci} after	[g/L]	0.59	6.22	17.97	30.02	0.58	6.34	18.13	29.84	0.58	6.26	18.05	30.00	0.58	6.84	19.91	31.58
C _{CI} before	[g/L]	0.00	6.07	18.20	30.33	0.00	6.07	18.20	30.33	0.00	6.07	18.20	30.33	0.00	6.07	18.20	30.33
Cl bound to ash	[g/L]	-0.59	-0.15	0.23	0.31	-0.58	-0.28	0.07	0.49	-0.58	-0.20	0.15	0.33	-0.58	-0.77	-1.71	-1.25
Cl bound to ash	[mg/g]	-1.78	-0.45	0.69	0.94	-1.75	-0.83	0.20	1.48	-1.75	-0.59	0.44	1.00	-1.74	-2.31	-5.13	-3.76

Chloride Binding into Washed LSA-depot

									Test	t no.							
		C1	C2	C3	C4	C5	C6	C7	C8	C9	C10	C11	C12	C13	C14	C15	C16
Test period	[weeks]	2	2	2	2	4	4	4	4	6	6	6	6	16	16	16	16
m _{LSA-depot}	[g]	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00
C _{salt water}	[%]	0	1	3	5	0	1	3	5	0	1	3	5	0	1	3	5
C _{CI}	[g/L]	0.00	6.07	18.20	30.33	0.00	6.07	18.20	30.33	0.00	6.07	18.20	30.33	0.00	6.07	18.20	30.33
V _{salt water}	[mL]	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00
C _{AgNO3}	[mol/L]		0.0	997			0.1	008			0.0	990			0.0	988	
m _{sample}	[mL]	5.00	0.50	0.50	0.50	3.00	0.50	0.50	0.50	5.00	0.50	0.50	0.50	-	0.50	0.50	0.50
m _{HNO3}	[mL]	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	-	2.00	2.00	2.00
m _{AgNO3}	[mL]	0.0903	0.8237	2.4256	4.1184	0.0658	0.8389	2.5358	4.1373	0.1164	0.938	2.4986	3.8019	-	0.8719	2.5966	4.41
C _{CI} after	[g/L]	0.06	5.82	17.15	29.11	0.08	6.00	18.12	29.57	0.08	6.59	17.54	26.70	-	6.11	18.19	30.90
C _{CI} before	[g/L]	0.00	6.07	18.20	30.33	0.00	6.07	18.20	30.33	0.00	6.07	18.20	30.33	-	6.07	18.20	30.33
						•	•			•		•					
Cl bound by ash	[g/L]	-0.06	0.24	1.05	1.22	-0.08	0.07	0.07	0.76	-0.08	-0.52	0.65	3.63	-	-0.04	0.00	-0.57
CI bound by ash	[mg/g]	-0.19	0.73	3.16	3.65	-0.24	0.21	0.22	2.28	-0.25	-1.56	1.96	10.90	-	-0.13	0.01	-1.71

The 0% sample tested for 16 weeks was dried out and a result could therefore not be found.

										Test no.								
		B1	B2	B3	B4	B5	B6	B7	B8	B8 extra	B9	B10	B11	B12	B13	B14	B15	B16
Test period	[weeks]	2	2	2	2	4	4	4	4	4	6	6	6	6	16	16	16	16
m _{mortar}	[g]	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00
C _{salt water}	[%]	0	1	3	5	0	1	3	5	5	0	1	3	5	0	1	3	5
C _{CI}	[g/L]	0.00	6.07	18.20	30.33	0.00	6.07	18.20	30.33	30.33	0.00	6.07	18.20	30.33	0.00	6.07	18.20	30.33
V _{salt water}	[mL]	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00
C _{AgNO3}	[mol/L]		0.1	000			0.1	009		0.1010		0.0	990			0.1	043	
m _{sample}	[mL]	4.00	1.00	0.50	0.50	5.00	1.00	0.50	0.50	0.50	5.00	0.50	0.50	0.50	6.00	0.50	0.50	0.50
m _{HNO3}	[mL]	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00
m _{AgNO3}	[mL]	0.0578	1.5116	2.24	3.8645	0.0565	1.5275	2.3438	4.0562	4.0353	0.0619	0.7624	2.3551	4.0596	0.0000	0.7255	2.3651	4.0387
C _{CI} after	[g/L]	0.05	5.36	15.89	27.41	0.04	5.46	16.77	29.02	28.89	0.04	5.35	16.53	28.49	0.00	5.36	17.49	29.86
C _{CI} before	[g/L]	0.00	6.07	18.20	30.33	0.00	6.07	18.20	30.33	30.33	0.00	6.07	18.20	30.33	0.00	6.07	18.20	30.33
	•	•	•	•	•	•	•	•	•	•		•		•	•	•	•	
Cl bound by mortar	[g/L]	-0.05	0.71	2.31	2.92	-0.04	0.60	1.43	1.31	1.44	-0.04	0.72	1.67	1.84	0.00	0.70	0.71	0.47
	[mg/g]	-0.15	2.12	6.93	8.76	-0.12	1.81	4.29	3.93	4.31	-0.13	2.15	5.01	5.51	0.00	2.11	2.14	1.42

Chloride Binding into Mortar with Unwashed LSA-depot



Figure 52: Comparison of chloride binding to mortar with and without LSA-depot as a function of the test period. Black = With LSA-depot. Red = Reference. The results for 0% with LSA-depot are placed right underneath the 0% reference results

									Test	t no.							
		D1	D2	D3	D4	D5	D6	D7	D8	D9	D10	D11	D12	D13	D14	D15	D16
Test period	[weeks]	2	2	2	2	4	4	4	4	6	6	6	6	16	16	16	16
m _{mortar}	[g]	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00
C _{salt water}	[%]	0	1	3	5	0	1	3	5	0	1	3	5	0	1	3	5
C _{CI}	[g/L]	0.00	6.07	18.20	30.33	0.00	6.07	18.20	30.33	0.00	6.07	18.20	30.33	0.00	6.07	18.20	30.33
V _{salt water}	[mL]	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00
C _{AgNO3}	[mol/L]		0.0	997			0.1	008			0.0	990			0.0	988	
m _{sample}	[mL]	7.00	0.50	0.50	0.50	7.00	0.50	0.50	0.50	7.00	0.50	0.50	0.50	8.00	0.50	0.50	0.50
m _{HNO3}	[mL]	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00
m _{AgNO3}	[mL]	0.0000	0.7567	2.3847	3.7350	0.0000	0.7849	2.4300	4.0471	0.0563	0.7807	2.3998	3.9099	0.0525	0.7635	2.3769	3.8980
	•																
C _{CI} after	[g/L]	0.00	5.35	16.86	26.40	0.00	5.61	17.37	28.93	0.03	5.48	16.85	27.45	0.02	5.35	16.65	27.31
C _{CI} before	[g/L]	0.00	6.07	18.20	30.33	0.00	6.07	18.20	30.33	0.00	6.07	18.20	30.33	0.00	6.07	18.20	30.33
Cl bound by mortar	[g/L]	0.00	0.72	1.34	3.93	0.00	0.46	0.83	1.40	-0.03	0.58	1.35	2.88	-0.02	0.72	1.54	3.02
Cl bound by mortar	[mg/g]	0.00	2.15	4.02	11.78	0.00	1.37	2.49	4.21	-0.08	1.75	4.04	8.63	-0.07	2.15	4.63	9.05

Chloride Binding into Mortar with Washed LSA-depot

Reference Mortar

			Test no.														
		E1	E2	E3	E4	E5	E6	E7	E8	E9	E10	E11	E12	E13	E14	E15	E16
Test period	[weeks]	2	2	2	2	4	4	4	4	6	6	6	6	16	16	16	16
m _{mortar}	[g]	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00
C _{salt water}	[%]	0	1	3	5	0	1	3	5	0	1	3	5	0	1	3	5
C _{CI}	[g/L]	0.00	6.07	18.20	30.33	0.00	6.07	18.20	30.33	0.00	6.07	18.20	30.33	0.00	6.07	18.20	30.33
V _{salt water}	[mL]	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00	15.00
C _{AgNO3}	[mol/L]		0.0	997			0.1	008			0.0	990			0.0	988	
m _{sample}	[mL]	7.00	0.50	0.50	0.50	7.00	0.50	0.50	0.50	7.00	0.50	0.50	0.50	8.00	0.50	0.50	0.50
m _{HNO3}	[mL]	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00
m _{AgNO3}	[mL]	0.0829	0.7511	2.4458	4.015	0.0826	0.7724	2.376	4.0447	0.084	0.7505	2.4318	3.9873	0.1026	0.778	2.4341	4.0794
C _{CI} after	[g/L]	0.04	5.31	17.29	28.38	0.04	5.52	16.98	28.91	0.04	5.27	17.08	28.00	0.04	5.45	17.06	28.58
C _{CI} before	[g/L]	0.00	6.07	18.20	30.33	0.00	6.07	18.20	30.33	0.00	6.07	18.20	30.33	0.00	6.07	18.20	30.33
Cl bound by mortar	[g/L]	-0.04	0.76	0.91	1.95	-0.04	0.55	1.22	1.42	-0.04	0.80	1.12	2.33	-0.04	0.61	1.14	1.75
Cl bound by mortar	[mg/kg]	-0.13	2.27	2.73	5.85	-0.13	1.64	3.65	4.26	-0.13	2.39	3.37	7.00	-0.13	1.84	3.43	5.24

B.6 Electrical Potential Measurements

SCE values are converted to SHE values by adding 242mV according to table 22. Numeric values are shown.

Specimens Cast with Distilled Water

	SCE		1	3 - Referenc	ce	14	- 10% ceme	ent
	SCE		А	В	С	А	В	С
	Measurement 1	[mV]	352	307	353	353	342	356
Day 0	Measurement 2	[mV]	350	305	355	352	340	356
Day U	Measurement 3	[mV]	350	303	354	352	337	355
	Mean	[mV]	351	305	354	352	340	356
Day 7		[mV]	487	490	435	465	461	373
Day 21		[mV]	546	578	495	495	501	305
Day 35		[mV]	488	580	553	494	530	399
Day 49		[mV]	571	569	544	530	572	554
Day 63		[mV]	559	522	550	545	593	565
	CLIE		1	3 - Referend	ce	14	- 10% ceme	ent
	SHL		А	В	С	А	В	С
	Measurement 1	[mV]	A 594	В 549	C 595	A 595	В 584	C 598
	Measurement 1 Measurement 2	[mV] [mV]	A 594 592	B 549 547	C 595 597	A 595 594	B 584 582	C 598 598
Day 0	Measurement 1 Measurement 2 Measurement 3	[mV] [mV] [mV]	A 594 592 592	B 549 547 545	C 595 597 596	A 595 594 594	B 584 582 579	C 598 598 597
Day 0	Measurement 1 Measurement 2 Measurement 3 Mean	[mV] [mV] [mV] [mV]	A 594 592 592 593	B 549 547 545 547	C 595 597 596 596	A 595 594 594 594	B 584 582 579 582	C 598 598 597 598
Day 0 Day 7	Measurement 1 Measurement 2 Measurement 3 Mean	[mV] [mV] [mV] [mV] [mV]	A 594 592 592 593 729	B 549 547 545 547 732	C 595 597 596 596 677	A 595 594 594 594 707	B 584 582 579 582 703	C 598 598 597 598 615
Day 0 Day 7 Day 21	Measurement 1 Measurement 2 Measurement 3 Mean	[mV] [mV] [mV] [mV] [mV] [mV]	A 594 592 592 593 729 788	B 549 547 545 547 732 820	C 595 597 596 596 677 737	A 595 594 594 594 707 737	B 584 582 579 582 703 743	C 598 598 597 598 615 547
Day 0 Day 7 Day 21 Day 35	Measurement 1 Measurement 2 Measurement 3 Mean	[mV] [mV] [mV] [mV] [mV] [mV] [mV]	A 594 592 592 593 729 788 730	B 549 547 545 547 732 820 822	C 595 597 596 596 677 737 795	A 595 594 594 594 707 737 736	B 584 582 579 582 703 743 772	C 598 597 598 615 547 641
Day 0 Day 7 Day 21 Day 35 Day 49	Measurement 1 Measurement 2 Measurement 3 Mean	[mV] [mV] [mV] [mV] [mV] [mV] [mV]	A 594 592 593 729 788 730 813	B 549 547 545 547 732 820 822 811	C 595 597 596 677 737 795 786	A 595 594 594 707 737 736 772	B 584 582 579 582 703 743 772 814	C 598 597 597 598 615 547 641 796

Specimens Cast with 3% Salt Water

	SCE		1	5 - Referen	ce	16	- 10% cem	ent
	SCE		А	В	С	А	В	С
	Measurement 1	[mV]	531	531	521	548	562	547
Day 0	Measurement 2	[mV]	532	531	521	548	563	544
	Mean	[mV]	532	531	521	548	563	546
Day 7		[mV]	543	528	521	513	531	521
Day 21	ay 21 [mV]		496	561	557	571	535	569
Day 35		[mV]	594	543	575	563	574	571
Day 49	Day 49 [mV			497	569	600	591	544
	СПЕ		1	5 - Referen	ce	16	- 10% cem	ent
	SHE		А	В	С	А	В	С
	Measurement 1	[mV]	773	773	763	790	804	789
Day 0	Measurement 2	[mV]	774	773	763	790	805	786
	Mean	[mV]	774	773	763	790	805	788
Day 7 [mV]			785	770	763	755	773	763
Day 21		[mV]	738	803	799	813	777	811
Day 35		[mV]	836	785	817	805	816	813
	Day 49 [mV]							

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C Test Data from Previous Reports

C.1 Results from Carlsen & Petersen, 2013

$\mathbf{p}\mathbf{H}$

		1	2	3	Mean	Standard deviation
рН	[-]	8.30	8.26	8.31	8.3	0.03

Conductivity

		1	2	3	Mean	Standard deviation
Conductivity	[mS/cm]	5.63	5.67	5.47	5.59	0.11

Water Content

		1	2	3	4	Mean	Standard deviation
Water content	[%]	14.49	14.39	14.43	14.28	14.40	0.09

Loss on Ignition

		1	2	3	Mean	Standard deviation
Loss on ignition	[%]	0.16	0.17	0.16	0.16	0.00

Water Solubility

		1	2	Mean
Water solubility	[%]	0.78	0.89	0.84

Content of Micro and Macro Elements

		1	2	3	Mean	Standard deviation
Aluminium, Al	[mg/kg]	18779.3	19999.3	19610.4	19463.0	623.2
Potassium, K	[mg/kg]	13372.6	14106.1	13949.3	13809.3	386.3
Sodium, Na	[mg/kg]	5460.9	5242.0	5282.0	5328.3	116.6
Zinc, Zn	[mg/kg]	2673.8	2887.6	2863.3	2808.2	117.0
Copper, Cu	[mg/kg]	701.07	688.10	694.09	694.42	6.49
Lead, Pb	[mg/kg]	100.61	98.46	99.33	99.47	1.08
Nickel, Ni	[mg/kg]	35.90	34.76	35.52	35.39	0.58
Chromium, Cr	[mg/kg]	28.75	27.91	29.10	28.59	0.61
Cadmium, Cd	[mg/kg]	2.22	2.06	2.20	2.16	0.09

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Water Soluble Anions

		А	В	С	Mean	Standard deviation
Chloride ions, Cl ⁻	[mg/kg]	1368.8840	1320.6943	1306.6733	1332.08	32.63
Nitrate ions, NO_3^-	[mg/kg]	97.3870	98.3194	99.5829	98.43	1.10
Sulphate ions, SO ₄ ²⁻	[mg/kg]	12976.8056	12985.1707	12928.5122	12963.50	30.58

C.2 Results from Reiff & Meldgaard, 2013

 $\mathbf{p}\mathbf{H}$

		1.1	1.2	1.3	2.1	2.2	2.3	Mean	Standard deviation
рН	[-]	12.48	12.46	12.46	12.45	12.43	12.34	12.4	0.05

Conductivity

		1	2	3	Mean	Standard deviation
Conductivity	[mS/cm]	7.82	7.79	7.82	7.81	0.02

Water Content

		1.1	1.2	1.3	2.1	2.2	Mean	Standard deviation
Water content	[%]	0.07	0.07	0.07	0.14	0.10	0.09	0.03

Loss on Ignition

		1	2	3	Mean	Standard deviation
Loss on ignition	[%]	0.20	0.24	0.30	0.25	0.05

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Water Solubility

		1	2	Mean
Water solubility	[%]	6.99	3.24	5.12

Content of Micro and Macro Elements

		D1	D2	D3	Mean	Standard deviation
Phosphorus, P	[mg/kg]	152445.0	156075.0	174837.0	161119.0	12018.0
Iron, Fe	[mg/kg]	41240.9	42494.6	48366.5	44034.0	3804.1
Aluminium, Al	[mg/kg]	19989.3	20549.7	22912.8	21150.6	1551.6
Potassium, K	[mg/kg]	13542.3	13783.1	15722.9	14349.4	1195.5
Sodium, Na	[mg/kg]	4509.4	4554.9	4605.8	4556.7	48.2
Zinc, Zn	[mg/kg]	2900.7	2960.0	3312.0	3057.6	222.3
Copper, Cu	[mg/kg]	705.64	710.67	716.93	711.08	5.65
Lead, Pb	[mg/kg]	99.94	102.80	104.14	102.29	2.15
Nickel, Ni	[mg/kg]	34.85	34.68	36.004	35.18	0.72
Chromium, Cr	[mg/kg]	29.22	29.33	30.41	29.65	0.66
Cadmium, Cd	[mg/kg]	2.1558	2.2313	2.24	2.21	0.05

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Water Soluble Anions

		D1	D2	D3	Mean	Standard deviation
Chloride ions, Cl	[mg/kg]	97.4230	86.0864	106.0834	96.53	10.03
Nitrate ions, NO ₃ ⁻	[mg/kg]	4.2683	3.1873	3.7134	3.72	0.54
Sulphate ions, SO ₄ ²⁻	[mg/kg]	10347.1026	10373.7205	9976.2374	10232.35	222.20

C.3 Results from Rem, 2013

Density and Porosity

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Sample recipe	No.	Specimen	Density	Porosity
			[kg/m ³]	[%]
		А	2140	14.5
		В	2130	15.1
Reference, 28days	20	С	2140	14.5
		Mean	2140	14.7
		Standard deviation	5.77	0.33
		А	2150	14.4
		В	2140	15.4
10%, 28days	21	С	2150	14.3
		Mean	2150	14.7
		Standard deviation	5.77	0.57

The densities and porosities for the individual specimens are compared to illustrate the difference between respectively the B samples and the A and C samples.



Figure 53: Comparison of the individual specimens with regards to both density and porosity

C.4 Results from Andreasen & Jørgensen, 2014

Density and Porosity

Two samples of each mortar recipe was made as samples 7, 8 and 9 were not vibrated properly due to a malfunctioning vibration table. Samples 10, 11 and 12 were afterwards cast and vibrated on the same vibration table as the specimens cast for this project. In the results it is seen that there is a large difference between the samples vibrated poorly and properly. It is therefore chosen to only take samples 10, 11 and 12 into account in this project.



Figure 54: Comparison of the densities of mortar samples with LSA-new and LSA-depot

Chloride Diffusion

10% cement - 53 days

Reference	- 53	days
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	Chloride	Mean chloride
Distance from surface	content	content
[cm]	[%]	[%]
0.5	0.37	0.32
	0.27	
1.5	0.13	0.00
	0.05	0.09
2.5	0.04	0.04
	0.03	0.04
3.5	0.08	0.06
	0.04	0.00
5.0	0.05	0.05
	0.05	0.05
7.0	0.04	0.05
	0.05	0.05

	Chloride	Mean chloride
Distance from surface	content	content
[cm]	[%]	[%]
0.5	0.38	0.35
	0.31	
1.5	0.11	0.08
	0.04	0.08
2.5	0.03	0.02
	0.03	0.05
3.5	0.03	0.02
	0.03	0.05
5.0	0.03	0.03
	0.03	0.05
7.0	0.03	0.04
	0.04	0.04

C.5 Results from Kurt, 2014

Chloride Diffusion

10% cement - 45 days

Reference - 45 days

	Chloride	Mean chloride
Distance from surface	content	content
[cm]	[%]	[%]
1.0	0.19	0.17
	0.14	0.17
3.0	0.02	0.02
	0.01	0.02

	Chloride	Mean chloride
Distance from surface	content	content
[cm]	[%]	[%]
1.0	0.16	0.15
	0.13	0.15
3.0	0.03	0.02
	0.02	0.05

D Poster

Poster made for midway presentation on 18th November 2013.

Use of Sewage Sludge Ash in Reinforced Concrete Risk of Corrosion

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The combination of a drastic ongoing rise in the overall CO2 emission in which cement production accounts for approximately 5% and both the cost and the environmental issues of depositing sewage sludge ash makes it very relevant to investigate the use of SSA as cement replacement in concrete.

Concrete is often reinforced with steel which creates possible problems with corrosion. Chloride ions promote the corrosion of the steel and comprenhensive research on using SSA as cement replacement with a focus on the risk of corrosion caused by chloride is therefore being performed at the moment. Tests are being conducted with LSA-depot - a sewage sludge ash from Lynetten Rensningsanlæg that has been stored in an outdoor depot for at least two years after the incineration.

Characterisation of LSA-depot

The chemical and physical properties of the SSA and mortar with SSA as cement replacement are determined by numerous different tests.



Rate of Corrosion

It is investigated if the replacement of cement by LSAdepot increases the rate of corrosion in mortar. Specimens are cast with a reinforcement bar:

Reference and 10% cement replacement with destilled water for both visual and electrical potential test
Reference and 10% cement replacement with salt water (3% NaCl) for both visual and electrical potential test







Test cycle: 7 days in 3% salt water and 7 days of drying out. Electrical potential test every fortnight. Results only shown for destilled water specimens.

Chloride Binding

It is investigated if either the SSA itself or mortar containing SSA can bind the chloride as the risk of corrosion in mortar can be decreased if the mortar has an ability to bind the chloride.

Tests are made with both pure LSAdepot, mortar with LSA-depot (both unwashed and washed LSA-depot) and reference mortar.



Chloride Binding into SSA and mortar with SSA



The SSA and the finely crushed mortar are placed in 0%, 1%, 3% and 5% salt water concentrations.

Tests are carried out for 2, 4, 6 and 16 weeks.

Chloride Diffusion

It is investigated what influence the SSA has on the speed of the diffusion of chloride ions into mortar.

Test specimens with respectively no LSA-depot, 5% sand replacement and 10% cement replacement are placed in 3% salt water. Chloride penetration can only happen through the ends of the specimens since the sides are painted with epoxy Chloride profiles are made after 7, 14 and 21





More experiments..?

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